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Study on application of green-synthesized ZnO and Si nanoparticles in enhancing aquaculture sediment quality

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Abstract

Background Fishpond sediments (FPS) are rich in organic carbon and nutrients, making them valuable as fertilizers and soil conditioners. Stabilizing heavy metals like chromium (Cr), copper (Cu), and zinc (Zn) is essential to reduce their bioavailability and risks. This study evaluates zinc oxide (ZnO) and silicon (Si) nanoparticles synthesized from *Azolla pinnata* and *Equisetum arvense* for heavy metal immobilization and nutrient enhancement in FPS from San Jiang (SJ) and Tan Niu (TN), China.

Methods Nanoparticles were synthesized using Azolla pinnata and Equisetum arvense. Fishpond sediments from San Jiang (SJ) and Tan Niu (TN) were treated with ZnO and Si nanoparticles. Heavy metals and nutrients were analyzed via ICP-OES and soil analysis, while sequential extraction assessed metal distribution in geochemical fractions.

Results The application of these nanoparticles, especially the green-synthesized zinc oxide nanoparticles (GSZnONPs), was found to significantly reduce the concentrations of chromium (Cr), copper (Cu), and zinc (Zn) in both the overlying and pore water of the FPS. This reduction not only minimizes the leachability of these heavy metals, but also substantially decreases their bioavailability. The study recorded a notable shift in the acid-soluble metal fraction, resulting in an average reduction of Cr concentrations by 31–28%, Cu by 18–21%, and Zn by 32–23% in the sediments from San Jiang (SJ) and Tan Niu (TN). Moreover, the application of these nanoparticles also improved the nutrient profile of the sediments, potentially enhancing their utility as fertilizers.

Conclusion Zinc oxide and silicon nanoparticles synthesized from *Azolla pinnata* and *Equisetum arvense* are effective in immobilizing heavy metals in fishpond sediments, significantly reducing their bioavailability and potential environmental risks. The use of these green-synthesized nanoparticles not only mitigates heavy metal contamination, but also enhances the nutrient content of the sediments, making them more suitable for use as soil conditioners and fertilizers. This dual benefit highlights the potential of these nanoparticles as a sustainable solution for managing contaminated fishpond sediments while contributing to agricultural productivity.

Keywords Zinc oxide nanoparticles, Silicon nanoparticles, Fishpond sediments, Nanoparticle–sediment interactions, Heavy metal detoxification

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Introduction

The elevated levels of heavy metals (HMs) and metalloids in aquatic systems, stemming from industrial processes, present a significant threat to human health and the environment, and causing adverse impacts [1]. The progression of industrialization has led to a notable increase in HM(s) and metalloids levels in riverine systems near mining and smelting areas, posing severe hazards to ecosystems and human health [2-5]. Fishpond sediments are vulnerable to contamination by various pollutants, including heavy metals, pesticides, and organic compounds, which may pose environmental and health hazards. When they enter in subsequent sediments, HM(s) are often sorbed onto solid particles, resulting in higher concentrations in sediments compared to the water phase [6]. Fishpond sediments can pose a biohazard due to potential contamination with (HMs) and metalloids and other pollutants. To prevent this, proper treatment methods such as stabilization, immobilization, or remediation should be implemented to mitigate environmental and health risks because sorbed HM(s) on sediment surfaces generally pose a low toxicity risk [7]. However, alterations in sediment pH, specific surface area, and nutrient content can impact the retention capacity and sorption of HM(s) [8]. As fishpond sediments, rich in nutrients and organic matter, present a valuable fertilizer for crop production [9]. While nutrient accumulation in sediments is well-documented [10–14] the persistence of HM(s) on sediment surfaces hinders direct application [11, 15]. For safe utilization as agricultural fertilizers, it is crucial to stabilize heavy metals (HMs) present in sediments and convert them into environmentally friendly amendments [16].

Chromium (Cr), copper (Cu), and zinc (Zn) were selected for this study because they are among the most prevalent heavy metals found in fishpond sediments due to aquaculture activities, industrial runoff, and the use of metal-based feed additives [17]. These metals pose significant environmental and ecological risks due to their potential bioaccumulation and toxicity [18, 19]. These metals were chosen to assess the effectiveness of greensynthesized nanoparticles in immobilizing a range of heavy metals with diverse chemical properties and environmental impacts.

The fractionation process of HMs in sediments involves categorizing them into acid-soluble, reducible, oxidizable, and residual fractions, with a gradient indicating decreasing mobility and bioavailability from acid-soluble to residual fractions. Soil/sediment remediation aims to shift HM(s) from mobile to immobile/residual forms, reducing their bioavailability [16]. Immobilization techniques, as effective remediation methods, can mitigate the mobility of HM(s) [2]. To address this issue, the remediation of fishpond sediments with green-synthesized nanoparticles offers a cost-effective and environmentally friendly solution, enhancing nutrient status and promoting plant growth. This approach proves quicker than traditional methods like biochar, providing an efficient strategy for pollutant removal [20]. Nanotechnology has emerged as a pivotal advancement in various fields of science and technology [21]. In agriculture, which serves as the foundation for industrial raw materials, the integration of nanotechnology is crucial for achieving high yields, enhancing crop production and protection, all while ensuring cost-effectiveness, eco-friendliness, and sustainability [22].

Biosynthesized nanomaterials offer a promising alternative to conventional approaches, utilizing natural resources such as plant extracts, Laurencia papillosa seaweed [23], bacteria [24], fungi [25], and algae [26]. In green synthesis processes, toxic chemicals are not required, resulting in the production of biocompatible, cost-effective [27], non-toxic [28], and often biodegradable, relying on biomolecules derived from plants and microorganisms [29]. These properties make them suitable for applications in agriculture and environmental remediation [30]. Green-synthesized nanoparticles have been explored for enhancing soil fertility, immobilizing heavy metals, and improving plant growth, while their environmental applications include pollutant removal, water purification, and serving as eco-friendly alternatives to synthetic chemicals. Among plant-based sources, Azolla pinnata has gained attention for heavy metal sequestration and its role in nitrogen fixation in rice crops. Nanoparticles derived from Azolla pinnata have demonstrated versatility in pollutant removal, weed suppression, and as supplements in animal diets [31].

Silicon nanoparticles (SiNPs) have gained significant attention in agriculture and environmental applications due to their versatility as nano-fertilizers, nanopesticides, and nano-zeolites [32]. These nanoparticles are particularly valued for their ability to enhance plant growth, improve stress tolerance, and facilitate the sustainable management of resources. Derived from horsetail (*Equisetum arvense*), a silica-rich plant, SiNPs offer a green synthesis route leveraging the abundant silica and silicic acids found in its hollow, jointed stems. This makes horsetail a cost-effective and sustainable source for SiNP production. In addition to improving plant productivity, SiNPs play a crucial role in environmental remediation, particularly in heavy metal removal through adsorption. Horsetail's global availability, rapid growth rate, and excellent surface coverage capacity further support its suitability for SiNP synthesis and environmental applications. Previous studies have explored the synthesis of SiNPs from horsetail, highlighting its potential in agricultural and environmental settings [33], but its specific application in removing heavy metals from fishpond sediments remains underexplored.

To the best of our knowledge no study introduces a novel, eco-friendly method for synthesizing zinc oxide and silicon nanoparticles, emphasizing sustainable practices over traditional chemical methods. The research is unique in demonstrating the dual functionality of these nanoparticles, which not only effectively remove heavy metals from fishpond sediments, but also enhance nutrient levels. By providing new insights into the mechanisms of nanoparticle-sediment interactions and exploring optimal application techniques, this study offers practical and sustainable solutions for sediment management. The objective of this study is to explore the potential of green-synthesized nanoparticles as an innovative solution for enhancing the quality of fishpond sediments. Specifically, the study focuses on: (i) synthesizing zinc oxide and silicon nanoparticles through green synthesis using plant extracts from Azolla pinnata and Equisetum arvense as eco-friendly and sustainable methods; (ii) evaluating the effectiveness of these nanoparticles in reducing the concentration of heavy metals in fishpond sediments; (iii) analyzing changes in the nutrient composition of fishpond sediments following nanoparticle application; (iv) investigating the mechanisms by which the nanoparticles mitigate heavy metal contamination and improve nutrient levels in sediments.

Materials and methods

Materials, chemicals, and reagents

All chemicals and reagents utilized in this study were of analytical grade and sourced from Fuchen Chemical Reagents, China. Raw materials of *Azolla pinnata* and horsetail (*Equisetum arvense*) plants were collected in Haikou City and Shaoguan City, China, respectively. These materials were employed for the green synthesis of ZnO and Si nanoparticles, respectively, based on their effectiveness against heavy metals as observed in preliminary trials [34, 35]. Anhydrous zinc sulfate (ZnSO₄) was procured from Xilong Science Co., Ltd. (China), while hydrochloric acid (HCl) and sodium hydroxide (NaOH) were obtained from Shanghai McLean Biochemical Technology Co., Ltd. (China). Potassium dichromate (K₂Cr₂O₇) was supplied by Guangzhou Huada Chemical Reagent Co., Ltd. Ultra-pure water (18.2 M Ω cm⁻¹ resistivity; Milli-Q, Germany) was used throughout the experiment. The methodology described by Sharma et al. [36] was followed for preparing the plant extract, with minor adjustments.

Nanoparticle synthesis

The synthesis of ZnO nanoparticles (ZnONPs) from *Azolla pinnata* leaf extract followed the method outlined by Jin et al. [37] with slight modifications. Specifically, 20 mL of *Azolla pinnata* leaf extract was combined with 20 mL of ZnSO4 solution (1:1, 1 M) and stirred at 70 °C for 6 h. The pH was adjusted to 12 using a 2 M NaOH solution. The successful fabrication of ZnONPs was indicated by a color change from light yellow to white. The chemical process for the plant-mediated synthesis of ZnONPs using *Azolla pinnata* leaf extract can be represented by the following simplified equations.

Azolla pinnata leave extract + $ZnSO_4 \rightarrow Zn - complex$

Zn-complex + NaOH \rightarrow $Zn(OH)_2$ \rightarrow $ZnO(nanoparticles) + H_2O$

The white precipitate was dried in an oven (Shanghai Yiheng-BPG-9240A, China) at 80 °C for further analysis.

Horsetail plants served as a green Si precursor, and the synthesis of silicon nanoparticles (SiNPs) followed the procedures outlined by Mohd et al. [38] with slight modifications for Si extraction from horsetail plants. The plant extract was subjected to reflux with sodium hydroxide (NaOH; 1.0 N) for one hour. The rationalized reaction after refluxing the plant extract with NaOH is as follows:

Si(horsetail plant extract)+ $NaOH \rightarrow Na_2SiO_3 + byproduts$

The separation of silicon nanoparticles (SiNPs) from sodium silicate (Na_2SiO_3) was achieved by gradually adding hydrochloric acid (HCl; 0.1 M) until the solution's pH reached 6.0, following the method described by Chapa-Gonzalez et al. [39]. The addition of HCl to Na_2SiO_3 led to a reaction forming NaCl and SiO₂, which can be rationalized as:

 $Na_2SiO_3 + 2HCl \rightarrow 2NaCl + SiO_2 + H_2O$

Subsequently, the precipitate obtained was subjected to multiple washes with ethanol and water to eliminate the formed NaCl. The collected precipitate was then separated through centrifugation at 9000 G for 10 min. The obtained product underwent further drying in an oven at 50 °C for 24 h. Following the drying process, the green-synthesized silicon nanoparticles (GS-SiNPs) were transferred to a vial for subsequent characterization and analysis.

Characterization of green-synthesized nanoparticles

The analysis of GS-ZnO- and Si-NPs included the determination of pore size distribution and BET surface area using a TriStar II 3020 Version 3.02 Serial # 2154 equipment from Micromeritics Instrument Corporation, China. Conditions for the analysis were as follows: adsorptive-N2, analysis bath temperature-195.8 °C, equilibration interval-5 s, and sample density-1.00 g cm-³. Micromorphological structures were characterized by a Gemini300 thermal field emission scanning electron microscope with energy spectrum (Oxford X-MAX) and Electron Backscatter Diffraction (Oxford SYMMETRYS), as well as a transmission electron microscope (TEM) model FEI Talos F200X. The iS10 FTIR spectrometer from Nicolli (USA) was used for surface functional group detection. Crystallinity of GS-ZnO- and Si-NPs was obtained using XRD wide-angle diffraction (D8 ADVANCE X-ray diffractometer from Bruker, Germany). X-ray photoelectron spectroscopy (250Xi from Thermo Fisher Scientific, USA) examined the composition and valence state, and zeta potential was measured using a Nano Brook Zeta PALS Potential Analyzer (Brookhaven Instruments, Holtsville, NY, USA).

Study area and sediment collection and characterization

The extraction of sediment samples took place in July 2023, involving the use of a core sampler at five parallel points for each site (Figure S1; supplementary information). The resulting sediment subsamples were combined to create composite samples, underwent air-drying at approximately 25 °C, and were subsequently sieved to achieve complete homogeneity. All sediment specimens were securely stored in air-tight plastic bags until subsequent analyses. Basic attributes of collected sediments, collect plant extract and prepared green nanoparticles are presented in Table S1.

Sediment treatment with nanoparticles

Each sediment sample (100 g dry weight) was blended with 120 mL of ultrapure water (18.2 M Ω cm⁻¹) in triplicate and left to stand for 24 h in a 250-mL screw-mouth bottle. Subsequently, 1 g of both GSNPs and GSSiNPs was added to the bottles in triplicate for TN and SJ sediments. A control treatment (CK) comprised bottles containing only sediment without nanoparticles. Following sealing, the bottles underwent vigorous shaking to ensure comprehensive mixing of the nanoparticles and sediments. The mixture was then placed in an incubator for 90 days at a constant temperature of 20±1 °C, shielded from light to facilitate natural interaction between the contents.

Metal extraction and analyses Metal in overlying water and pore water

Following the incubation period, the overlying water was completely withdrawn and filtered through a 0.45-µm filter before analysis. Subsequently, the remaining sediment-NPs mixture underwent centrifugation at 4000 rpm for 15 min to facilitate water separation from the pores. The extracted pore water was also filtered through a 0.45-µm filter. This methodology aligns with techniques employed and documented in Tou et al. [40]. Concentrations of Cr, Cu, and Zn in both the overlying water (Cow) and pore water (Pow) were determined using inductively coupled plasma optical emission spectrometry (ICP-OES; Optima 8000).

Metal fractionation in sediments

The geochemical fractions of Cr, Cu, and Zn in the TN and SJ sediments were sequentially extracted using the methodology outlined by the Commission of the European Communities Bureau of Reference (BCR; [41]). This sequential extraction technique, also employed by [42–44], involves fractionating metals into four operationally defined fractions: F1 (acid-soluble), F2 (reducible), F3 (oxidizable), and F4 (residual), the detailed information of this procedure is provided in Table S2. The extracted metal fractions were subsequently analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES; Optima 8000).

Toxicity characteristic leaching procedure (TCLP)

The leaching potential of heavy metals (HMs) in the TN and SJ sediments was assessed using the Toxicity Characteristic Leaching Procedure (TCLP) method 1311, as developed by the U.S. Environmental Protection Agency. An extracting solution was prepared with glacial acetic acid (CH₃COOH; pH= 4.93 ± 0.05 ; [45]). 40 ml of the extracting solution were mixed with 2 g of sediment, stirred at 190 rpm for 18 h (25 °C), and then centrifuged at 4000 rpm for 15 min. The resulting supernatant was filtered through a 0.45-µm filter, and the concentrations of Cr, Cu, and Zn were analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES; Optima 8000).

Characterization and nutrients content in the sediments

The finely ground sediments underwent characterization using standard methods outlined in [46]. The pH was measured potentiometrically with the pH meter HJ 962-2018. Electrical conductivity (EC) of the sediments was determined using the electrode method (HJ 802-2016). Total nitrogen was assessed through the wet oxidation and micro-Kjeldahl method (LY/T 1228-2015). Available phosphorus and exchangeable potassium were determined via extraction with sodium bicarbonate (NY/T 1121.7-2014) and ammonium acetate (LY/T 1246-1999), respectively. Total calcium, magnesium, and sodium in both sediments were determined following the "NY/T 296-1995" method, and the contents of calcium and magnesium were analyzed using atomic absorption spectrophotometry, while sodium content was measured with a flame photometer. Carbon content was detected using a general method for the chemical characteristics of domestic waste (CJ/T 96-2013) and analyzed through the element analyzer method. Sediment particle distribution was determined using the hydrometer method (LY/T 1225-1999). The total content of copper (Cu), zinc (Zn), and chromium (Cr) in both sediments was extracted and measured by inductively coupled plasma optical emission spectrometry (ICP-OES).

Data quality assurance and statistical analysis

All treatments and experiments were performed in triplicate to ensure robustness and reliability of the results. To maintain accuracy in metal concentration measurements, quality control measures were implemented, including the use of certified reference samples. The recovery percentages for chromium (Cr), zinc (Zn), and copper (Cu) were found to be 99.6%, 96.6%, and 103.7%, respectively. Blank and triplicate measurements were conducted for analyses, with standard solutions from Merck utilized to validate the precision of the results. A maximum relative standard deviation (RSD) of 10% was accepted between replicates to ensure consistency. Statistical analyses were performed using IBM SPSS Statistics 23, employing oneway ANOVA with Duncan's multiple range tests at a significance level of p<0.05. Graphs were created using OriginPro 9.1 b215 (OriginLab Corporation, Northampton, USA).

Results and discussion

Characterization of nanoparticles

Figure 1a illustrates the microstructures and morphology of green-synthesized zinc oxide nanoparticles (GSZnONPs) and green-synthesized silicon nanoparticles (GSSiNPs). SEM micrographs reveal that GSZnONPs exhibit uniform sizes and multifaceted structures, consistent with the findings of Wenjie et al. [47] who utilized ZnONPs from plant extract as a Cr adsorbent in water. Conversely, the SEM micrographs of GSSiNPs depict compactness, unevenness, and uniform size, aligning with observations by Mehmood et al. [35].

Transmission electron microscopy (TEM) analysis was employed to examine the particle morphology of



Fig. 1 a Scanning electron microscope (SEM) images, b transmission electron microscopy (TEM) images, c Fourier transform infrared (FTIR) spectra, and d X-ray diffraction (XRD) spectra of horsetail plant (HP), green-synthesized silicon nanoparticles (GSSiNPs), *Azolla pinnata* (AP) and green-synthesized zinc oxide nanoparticles (GSZnONPs)

GSZnONPs and GSSiNPs. TEM images of GS-SiNPs (Fig. 1b) reveal a layered, porous structure characterized by unique, bulky, multi-layered aggregations and monodispersed nanoparticles (NPs). The BJH adsorption average pore diameter (4 V/A) is measured at 15.01 nm. This increased porous structure enhances the accessible area, resulting in a greater adsorption capacity with practical implications for real environments [36]. Similarly, TEM images of GSZnONPs (Fig. 1b) exhibit a clear adsorbent shape, size, and microstructure, with an average pore diameter of 15.01 nm in the multi-layered porous structure, suggesting their suitability for adoption in real environments without compromising adsorption capacity. Additionally, Fig. 1d confirms hexagonal symmetry for GSZnONPs as determined by XRD [47].

The surface functional groups of GSZnONPs and GSSiNPs were analyzed using Fourier-transform infrared spectroscopy (FTIR). The FTIR spectra, recorded by drop-casting the nanoparticles onto KBr pellets (Fig. 1c), revealed distinctive peaks for GSZnONPs at 916.66 (C–O–C), 1509.06 (C=C aromatic), 1632.86 (C=C alkene), and 3442.61 (N–H stretch) [48]. Additionally, there were CO₂ peaks in the remaining spectrum [47]. In the case of GSSiNPs, the data indicated prominent Si–O–Si symmetric bond stretching at 474 cm⁻¹, and the peak at 1069 cm⁻¹ was attributed to the asymmetric stretching of the Si–O–Si bond, characteristic of SiNPs [36]. Bands at 1634 and 3442 cm⁻¹ were associated with N–H stretching and bending, respectively [49]. These findings underscore the distinct functional groups playing a role in the reduction and stabilization of the nanoparticles [36].

The X-ray diffraction (XRD) analysis was conducted following the procedure outlined in Sect. 2.4 to assess the crystallinity, size, and phases of GSZnONPs and GSSiNPs. For GSZnONPs, the XRD spectrum revealed sharp peaks corresponding to polycrystalline wurtzite structure with indices (100), (002), (101), (102), (110), (103), (200), (112), (201), and (004) at angles 31.67° , 34.31° , 36.14° , 47.40° , 56.52° , 62.73° , 66.28° , 67.91° , 69.03° , and 72.48° , indicating the crystallization of the material (Fig. 1d). Similarly, the XRD spectrum for GSSiNPs exhibited well-defined peaks at positions 28.40 < 111 >, 47.20 < 220 >, 56.05 < 311 >, 69.10 < 400 >, and 76.30 < 331 >, indexed with silicon phase (Si-NPs). The sharpening of the peaks suggests the nanoparticles' regime, and the absence of impurity-related peaks confirms the high purity and crystalline nature of the processed material [32].

Figure S2 presents elemental mapping images of GSZnONPs and GSSiNPs. The weight proportion of elements, determined by EDS, indicates a composition of 46.11% C, 39.67% O, 13.06% Zn, 0.67% P, 0.36% N, and 0.13% Si for GSZnONPs. The concentration of Zn is affirmed by the elemental distribution, contributing to enhanced adsorption capacity for water pollutants in the presence of oxygen and carbon. For GSSiNPs, the weight proportion comprises 65.6% C, 26.4% O, and 8.0% Si. The elemental distribution confirms the presence of Si, while the coexistence of O and C enhances the adsorption capacity of GSSiNPs for water pollutants [50, 51].

Brunauer–Emmett–Teller (BET) analysis revealed that GSZnONPs and GSSiNPs have average pore diameters of 15.99 nm and 9.76291 nm, and particle sizes of 315.98 nm and 437.7283 nm, respectively. The larger surface area of these nanoparticles, with a BET value of 18.98 m² g⁻¹ for GSZnONPs and 13.7071 m² g⁻¹ for GSSiNPs, suggests their potential to bind heavy metals [52]. GSZnONPs exhibited a single-point adsorption total pore volume of 0.075945 cm³ g⁻¹, while GSSiNPs had a value of 0.033455 cm³ g⁻¹. Additionally, t-Plot micropore volume for GSZnONPs was 0.001006 cm³ g⁻¹, while GSSiNPs showed -0.000062 cm³ g⁻¹. These characteristics indicate the potential of both green-synthesized nanoparticles as effective adsorbents for water pollution mitigation [53].

Effect of nanoparticles on sediment properties and nutrients content

In the evaluation of the impact of green-synthesized zinc oxide (GS-ZnO) and silicon (GSSi) nanoparticles (NPs) on the properties and nutrient content of fishpond sediments (SJ and TN), the initial chemical and nutrient analyses revealed elevated nutrient levels, suggesting their suitability as fertilizers for sustainable crop production. However, the unamended sediments exhibited significantly different mean pH values (SJ pH=8.61; TN pH=6.59). The addition of GSZnONPs and GSSiNPs led to a significant increase in pH, with higher values observed in GSZnONP-treated sediments (SJ pH=9.36; TN pH=7.16) (Table S3). The elevated pH, particularly in SJ, may potentially limit crop growth. Nonetheless,

TN pH remained within the acceptable soil pH range (5.0-7.0). The rise in pH enhances the sediments net negative charge, facilitating heavy metals (HMs) binding and reducing their solubility and bioavailability. Additionally, GSZnONPs increased the electrical conductivity (EC) of both sediments, further influencing their physicochemical properties. In general, the presence of both the nanoparticles resulted in an increase in pH, electrical conductivity (EC), and organic carbon (OC) content. In line with our results many studies reported the same trend of results in soil with the application of various nanoparticles for example for silver nanoparticles in soil for 60 days [54]; TiO₂, ZnO and CuO in soil for 90 days [55-57]. The introduction of nanoparticles (NPs) significantly enhanced the plant nutritional value of both sediments by contributing nutrients and adsorbing pollutants. This improvement can be attributed to the chemical reactivity of NPs, as documented in previous studies [24, 58–60]. Incorporating GSZnONPs into the SJ sediment led to significant increases in total nitrogen (N) by 8.77%, available phosphorus (P) by 4.39%, and exchangeable potassium (K) by 9.42%, compared to the control (Table S3). Similarly, GSZnONPs addition to the TN sediment resulted in elevated total N by 28.70%, available P by 13.75%, and exchangeable K by 17.98% (Table S3). This trend extended to calcium (Ca) and magnesium (Mg), with increases of 6.58% (Ca) and 5.48% (Mg) in the SJ sediment, and 21.07% (Ca) and 17.37% (Mg) in the TN sediment post-GSZnONPs incorporation. Despite quasiidentical initial plant nutrient content in both sediments, nanoparticle addition significantly enhanced their fertilization potential, with GSZnONPs demonstrating the most pronounced effects, surpassing GSSiNPs.

Effect of nanoparticles on dissolved metal concentrations

The assessment of ecological risks posed by heavy metals (HMs) in water bodies has been extensively investigated, as evidenced by studies [12, 61]. In aquatic ecosystems, the emphasis on measuring dissolved metal concentrations is crucial, given their significance as toxicity predictors [62]. Monitoring these concentrations provides valuable insights into the potential ecological impact of HMs on aquatic environments. In the untreated SJ sediments, the mean concentrations of Cr, Cu, and Zn in the overlying water (Cow, $\mu g L^{-1}$) were 39.81, 3.27, and 19.83, while in the untreated TN sediments, the corresponding concentrations were 24.27, 11.30, and 17.50 (Fig. 2). The addition of nanoparticles resulted in a significant reduction in metal concentrations in the overlying water (Cow) (Fig. 2). When compared with the unamended sediments (control), GSZnONPs exhibited higher effectiveness in reducing the average concentrations ($\mu g L^{-1}$) of Cr (by 110.93% in SJ and 85.02% in TN), Cu (by 73.31% in SJ and

4.5

3.6

Си (µg L⁻¹)

a

A) 45.5

36.4

27.3

18.2

 $Cr (\mu g L^{-1})$





Fig. 2 Impact of green-synthesized nanoparticles on the concentrations (μ g L.⁻¹) of Cu, Cr, and Zn in the overlying water layer (Cow) of the San Jiang (SJ) fishpond sediments (**a**) and fishpond Tan Niu (TN) sediments (**b**). Results are the mean values ± standard deviation (n = 3). Error bars indicate standard deviations. Different small letters on the bars indicate significant differences among treatments at P < 0.05

83.30% in TN), and Zn (by 117.58% in SJ and 78.91% in TN) in the Cow of both sediments. Similarly, GSSiNPs showed effectiveness, reducing concentrations by 37.72–44.58% for Cr, 19.31–22.28% for Cu, and 32.64–46.66% for Zn (Fig. 2).

In the untreated SJ sediment, concentrations of metals in the pore water (Cpw) were observed 6.14 µg L⁻¹ for Cr, 0.91 µg L⁻¹ for Cu, and 7.89 µg L⁻¹ for Zn, while in the untreated TN sediment, corresponding values were 6.57 µg L⁻¹ for Cr, 3.56 µg L⁻¹ for Cu, and 5.72 µg L⁻¹ for Zn (Fig. 3). The addition of nanoparticles significantly mitigated metal concentrations in the Cpw (Fig. 3), with GSZnONPs proving more effective than GSSiNPs. Specifically, GSZnONPs reduced average concentrations (µg L⁻¹) of Cr by 91.56% in SJ and 50.17% in TN, Cu by 58.55% in SJ and 52.85% in TN, and Zn by 12.58% in SJ and 72.02% in TN. GSSiNPs also exhibited effectiveness, reducing concentrations by 33.76–32.89% for Cr,



Fig. 3 Impact of green-synthesized nanoparticles on the concentrations (μ g L.⁻¹) of Cu, Cr, and Zn in the pore water (Cpw) of the San Jiang (SJ) fishpond sediments (**a**) and fishpond Tan Niu (TN) sediments (**b**). Results are the mean values±standard deviation (n=3). Error bars indicate standard deviations. Different small letters on the bars indicate significant differences among treatments at P < 0.05

15.41-21.57% for Cu, and 2.46-35.54% for Zn in both sediments (Fig. 3). This interpretation aligns with findings from recent studies [4, 9, 15, 63]. Metal concentrations in the Cow of all treatments were generally higher than in the Cpw, potentially indicating metal release from sediments to the Cow during the 90-day incubation under flooded conditions. This release may be attributed to reduced redox potential (Eh) during flooding, leading to reductive dissolution of Fe-Mn oxides, microbial decomposition of organic matter, and subsequent metal release. Notably, Cr concentrations in the Cow were higher than those of Cu and Zn, likely due to the higher total content of Cr compared to Cu and Zn. Additionally, the potential precipitation of Cu and Zn, particularly Cu, with sulfide under flooded reducing conditions, may contribute to their lower release compared to Cr. The results





Fig. 4 Impact of green-synthesized nanoparticles on the TCLP-extracted concentrations (mg kg.⁻¹) of Cu, Cr, and Zn in San Jiang (SJ) fishpond sediment (**a**) and fishpond Tan Niu (TN) sediment (**b**). Results are the mean values \pm standard deviation (n = 3). Error bars indicate standard deviations. Different small letters on the bars indicate significant differences among treatments at P < 0.05

underscore a significant reduction in metal concentrations in both the overlying water (Cow) and pore water with nanoparticle addition, highlighting the superior efficacy of GSZnONPs attributed to its higher surface-active functional groups, greater crystallinity, and higher BET surface area.

Effect of nanoparticles on metals leachability

The average concentrations of TCLP-extracted Cr, Cu, and Zn (μ g L⁻¹) were determined to be 39.96, 3.45, and 23.52 in the untreated SJ sediment, and 30.79, 12.47, and 19.36 in the untreated TN sediment, respectively (Fig. 4). The addition of nanoparticles led to a significant reduction in TCLP-extracted Cr, Cu, and Zn compared to the unamended sediments (Fig. 4). Specifically, GSZnONPs demonstrated greater efficacy than the control in reducing the average concentrations (μ g L⁻¹) of Cr (by 132% in SJ and 118% in TN), Cu (by 546% in SJ and 74% in TN), and Zn (by 212% in SJ and 115% in TN), followed by GSSiNPs (63–71% for Cr, 139–30% for Cu, and 94–60% for Zn) (Fig. 4). This finding underscores the effectiveness of both applied nanoparticles in mitigating the solubility and leachability of Cr, Cu, and Zn in the studied sediments. Notably, GSZnONPs exhibited a higher ability than GSSiNPs in reducing the leachability of the three metals (Fig. 4). These findings suggest that environmentally relevant heavy metal (HM) fractions, such as pore water (Cpw) and overlying water (Cow), may respond differently to anthropogenic activities and non-point sources of HMs [64].

Figure 5 illustrates potential mechanisms for the immobilization of HMs. The O/C molar ratio is indicative of oxygen-containing groups in nanoparticles, while the BET surface area reflects pore characteristics [65]. The superior performance of GSZnONPs compared to GSSiNPs is attributed to its high BET surface area and oxygen-containing groups, both known to significantly influence the adsorption capacity of nanoparticles [66, 67]. However, the analysis of NP mineral components (Fig. 1c) reveals that the presence of oxygen reactive groups on NP surfaces enhances the binding capacity of NPs to HMs [68, 69].

Effect of nanoparticles on metals fractions

In the untreated SJ sediment, the concentration $(mg kg^{-1})$ of the acid-soluble fraction accounted for 26.08 (49.5% of the total content) for Cr, 3.91 (38.09%) for Cu, and 19.76 (47.04%) for Zn, while in the untreated TN sediment, the corresponding values were 19.31 (47.75%) for Cr, 9.93 (45.04%) for Cu, and 12.61 (48.02%) for Zn (Fig. 6). Notably, the acid-soluble fraction dominated the total content, surpassing the reducible, oxidizable, and residual fractions (Fig. 6). The prevalence of the acid-soluble fraction and the limited presence of the residual fraction indicate the high solubility and potential mobility of these metals in both sediments, suggesting a heightened potential risk. To ensure the reliability of the fractionation results, the sum of fractions for each metal was compared with the total metal content determined through acid digestion. The control results demonstrated a satisfactory recovery percentage within the range of 90-112%. These findings affirm the effectiveness of the sequential extraction procedure, indicating that it facilitated an acceptable quantitative recovery of total metals in our study. The acid-soluble fraction, considered the most bioavailable among all fractions, exhibited a significant reduction in Cr, Cu, and Zn concentrations in sediments treated with different nanoparticles (NPs) compared to unamended sediments (Fig. 6). In comparison to the unamended control, GSZnONPs demonstrated greater efficacy in decreasing the average concentrations of acid-soluble Cr (by 35.05% in SJ and 42.29% in TN), Cu (by 47.74%





Fig. 5 Possible mechanisms of green-synthesized nanoparticles action in heavy metals (HMs) immobilization in the fishpond sediments



Fig. 6 Impact of green-synthesized nanoparticles on the geochemical fractions of Cu, Zn, and Cr (% of total content) in the in San Jiang (SJ) fishpond sediments (**a**) and fishpond Tan Niu (TN) sediments (**b**)

in SJ and 45.05% in TN), and Zn (by 35.10% in SJ and 53.27% in TN), followed by GSSiNPs treatment (reductions of 20.79-35.54% for Cr, 35.54-42.09% for Cu, and 42.07-51.37% for Zn) (Fig. 6). The reducible fraction, primarily composed of HMs bound onto Fe and Mn oxides or hydroxides, also decreased with the addition of NPs for Cr, Cu, and Zn in both sediments, although this effect was not as pronounced as observed for the acid-soluble fraction (Fig. 6). The oxidizable metal fractions in both SJ and TN were relatively smaller compared to the other three fractions and showed a slight increase upon NPs addition. This phenomenon could be attributed to the potential formation of complexes between HM ions and reactive groups on the surface of NPs [70]. Subsequently, the residual fractions of Cr, Cu, and Zn in both sediments increased to varying degrees following NPs treatment. As residual HMs are virtually non-bioavailable, our findings suggest that NPs effectively enhanced the immobilization of HMs and facilitated their conversion into more stable pools, with the extent of conversion closely tied to the specific type of NPs chosen (Fig. 6). The observed reduction in heavy metal (HM) concentrations can be attributed to the establishment of chemical interactions between HMs and active sites on the nanoparticles, resulting in irreversible bonding. Additionally, the alkaline nature of the nanoparticles, with a pH greater than 8, promotes the precipitation of metal hydroxides [71]. This precipitation process likely explains the high levels of residual metal fractions observed, as heavy metals tend

to form low-solubility hydroxides under alkaline conditions, thereby becoming immobilized in the sediment matrix [72]. Moreover, the interaction of nanoparticles (NPs) with natural organic matter in sediments forms stabilizing hard and soft layers, significantly influencing their environmental behavior and preventing ions (Ca²⁺ and Mg²⁺)-induced aggregation [73]. Furthermore, cations released from nanoparticles may form complexes or precipitates with HMs. The substantial presence of nutrients on the nanoparticle surfaces enhances the formation of low-solubility precipitates when interacting with HMs [74]. This mechanism contributes to the immobilization of HMs in fishpond sediments, rendering them suitable as amendments/fertilizers for sustainable crop production. Nanoparticles, with their small size and innovative surface coatings, have the ability to permeate through tiny soil pores. Additionally, their large surface area relative to their size makes them advantageous for applications involving water and gas interactions. These advancements include the adsorption of water and gas vapors, as well as photocatalytic methods for the degradation of pollutants [75].

Conclusions and future implications

In conclusion, this study highlights the potential of green-synthesized zinc oxide (ZnO) and silicon (Si) nanoparticles, derived from the aqueous plants Azolla pinnata and Equisetum arvense, for immobilizing heavy metals (HMs) and enhancing nutrient content in fishpond sediments (FPS). The application of these nanoparticles, particularly GSZnONPs, significantly reduces the concentrations of chromium (Cr), copper (Cu), and zinc (Zn) in both overlying and pore water, thereby decreasing the leachability of these metals from FPS. This reduction in metal bioavailability is accompanied by significant changes in the acid-soluble metal fraction, leading to lower average concentrations of Cr, Cu, and Zn in sediments from San Jiang (SJ) and Tan Niu (TN), China. These findings underscore the potential of GSNPs in mitigating heavy metal contamination and improving the reutilization of FPS as fertilizers and soil conditioners.

Future research should aim to optimize the concentrations of green-synthesized nanoparticles (GSNPs) to maximize their efficacy while minimizing environmental impacts. Long-term studies are required to evaluate the persistence and stability of GSNPs in sediments and their sustained effects on soil health and plant growth. Furthermore, investigating their effectiveness under diverse environmental conditions and assessing their impact on plant health will provide a comprehensive understanding of their practical applications. Conducting economic feasibility analyses will also be essential for facilitating large-scale implementation.

Supplementary Information

The online version contains supplementary material available at https://doi. org/10.1186/s40538-024-00716-4.

Additional file 1: Section S1. Preparation of plant extract. Figure S1. Fishpond sediments sites in San Jiang and Tan Niu in eastern Hainan Province, China. Figure S2: Elemental mapping of Green synthesized silicon nanoparticles (GSSINPs) and green-synthesized zinc oxide nanoparticles (GSZnONPs). Table S1. Basic attributes of green-synthesized nanoparticles, *Azolla pinnata* (AP) and horsetail plant (HP). Table S2: The BCR sequential extraction procedure. Table S3. Basic attributes of sediments from San Jiang (SJ) and Tan Niu (TN) before and after addition of green-synthesized (GS) zinc oxide (ZnO) and silicon (Si) nanoparticles.

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Author contributions

Yaoqiang Zhu conducted the experiment, collected and treated the data, created figures, and drafted the manuscript. Weidong Li contributed to experiment performance, investigation, analysis, and writing. Waqas Ahmed provided review, editing, and proofreading support. Mohsin Mahmood conducted data analyses, contributed to writing, and made corrections. Hayssam M. Ali provided review, editing, and proofreading support and fund-ing support. Muhammad Rizwan conducted data analyses, contributed to writing, and proofreading support and fund-ing support. Muhammad Rizwan conducted data analyses, corrected, edited, and proofread the manuscript. Muhammad Akmal ensured the accuracy of data analysis, made corrections, and assisted with editing. Sajid Mehmood contributed to writing, reviewing, correcting, editing, and proofreading the manuscript, as well as corresponding with collaborators.

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Availability of data and materials

No datasets were generated or analysed during the current study.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication

All co-authors have reviewed and consented to the contents of the work, and there are no financial conflicts of interest to disclose.

Competing interests

The authors declare no competing interests.

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