Ecofriendly sustainable synthesized nano-composite for removal of heavy metals from aquatic environment

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Abstract

The toxicity of heavy metals in the aquatic environment is a serious challenge to the global community. Even at low concentrations, heavy metals have a cumulatively destructive effect on living organisms in the aquatic environment. Biomass wastes have been investigated for heavy metals removal in the published literature; however, the low performance and capacity of the biomass represents a drawback towards effective application. Therefore, in this study, biomass waste such as corn leaves that have low cost is investigated as a precursor for eco-friendly sustainable nanostructured composite. In this research, several experiments have been conducted focusing on upgrading the capacity of the bioresource for removing heavy metals from the aquatic environment. In addition to low-cost biomass material, nanomaterials such as zinc oxide represents an attractive combination for effective removing heavy metals such as iron and nickel ions. Characterization of the synthesized composite material was conducted using XRD–FESEM-mapping and EDX–HRTEM and SAED–Zeta size and Zeta potential. Moreover, studying the efficiency of synthesized nano-composite for heavy metals ions adsorption of iron and nickel ions shows an outstanding increase of performance. The results suggest that adding nanomaterial to biomass matter and obtaining a composite at nanosize, enables the increase of the adsorption efficiency of heavy metals.

Keywords Bioresource · Heavy metals · Adsorption · Biomass · Nano-composite

Introduction

Biomass wastes represent attractive low-cost materials for solving major environmental problems such as heavy metals in the aquatic environment. With the growth of serious problems for the environment due to the rising concentration of toxic contaminants, there is an imperative need for developing healthy, low cost and eco-friendly materials to detox and purify water sources and to protect the environment

health (Babel and Kurniawan 2003). Pollution of water resources with heavy metals leads to contamination of the food chain as it represents a major threat to the ecosystem of the planet (Gupta 2009). Accordingly, there is a serious gap of knowledge and need for more research that can contribute to solving this crisis related to water purification, improving its quality, and the possibility of reusing wastewater in agriculture, for example, instead of wasting it. Many treatment techniques are available with varying degrees of success to control water contamination (Gupta et al. 2009). However, the limitations of most of these techniques are high functioning and preservation costs and the complex process of the treatment. Comparatively, the adsorption process is considered a good alternative for water treatment because of its ease of operation (Ahmaruzzaman and Gupta 2011). Biomass sorbents are considered worldwide adsorbents for wastes are generally used for the removal of different contaminants from water. Many adsorbents of low-cost have been studied for their ability to remove several types of contaminants from wastewater (Mohan et al. 2014). Comparatively, the adsorption process is considered a good alternative for water treatment because of its

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ease of operation (Lankathilaka et al. 2021; Tansir Ahamad et al. 2020). In this study, bio-adsorbents efficiency will be enhanced by adding a nanomaterial to produce a synthetic nano-composite. In this work, corn leaves, ZnO and synthesized nanocomposite will be examined for removing iron and nickel ions from the water environment. Recent studies have shown that at least some of the NPs can be absorbed in water treatment. The newly emerging field of nanotechnology offers a potential offer for water purification with low cost, high work efficiency in removing contaminants and the ability to be reused again. Shortly, the study of nanoparticles in water purification is considered to evaluate positive stand-points (Fernando et al. 2020; Xiao-long et al. 2020). Several treatment techniques are available with varying degrees of success to control water contamination (Zhaojin et al. 2021; Yaowei et al. 2021). However, the limitations of these techniques are high functioning and preservation costs (Aruna et al. 2021; Ahmed et al. 2021). Many low-cost adsorbents have been studied for their ability to remove several types of contaminants from wastewater (Jingjing et al. 2021; Aya et al. 2021). In this work, corn leaves, ZnO and synthesized nanocomposite will be examined for removing iron and nickel ions from the water environment.

Materials and methods

Biomass precursor and synthetic metal solution

A 500 mg corn leaves (CL) (Egyptian origin planted near Cairo) and 250 mg of nanoparticles (ZnO) (synthesized by our lab) (Aya et al. 2021) were used in each experiment, nanocomposite material consisting of 250 mg of (waste corn leaves and ZnO in a 1:1 ratio). Iron sulphate (Fe_2SO_4) supplied by Merck was used to prepare synthetic water with a concentration of 38.33 mg/L. Nickel sulphate (NiSO_4) supplied by Merck was used to prepare synthetic water with a concentration of 10 mg/L. The pH of the water samples was adjusted during experimental work using (0.1 M) HCl to reduce the pH or (0.1 M) NaOH to raise the pH when required. In all measurements, glass containers were employed to prevent metal ions from being absorbed into the walls of the containers housing.

Biomass preparation

A biomass waste of (corn leaves) was prepared after carefully choosing samples of organic matter (corn leaves) and ensuring that there were no contaminants, bacteria, or other dangerous substances adhered to the corn leaves. The samples were thoroughly washed and dried in a drying oven that is specifically used for drying purposes for 48 h at a temperature of 50 °C. After complete drying of the sample,

it is subjected to grinding multiple times to confirm that the samples were within the desired size range. The powder resulted from the grinding process is sieved with different mesh sizes until it reaches the desired size of less than 0.5 mm. Glass containers were used to store the samples, which were maintained dry and free from moisture.

Preparation of nanocomposite

Zinc oxide (ZnO) used in the experimental work of this study was kept in an inert atmosphere using deoxygenated distilled water. The following chemical process was used for the preparation of the nanocomposite (Medha et al. 2021):

1. Biomass (1000 mg) was disseminated in (200 ml) distilled water 120 min.
2. ZnO (1000 mg) was homogenized in 100 ml distilled water for 30 min.
3. The two mixes were combined of nanoparticles ZnO and corn leaves to make nanocomposite and held in the ultrasonic probe in an iced water bath for 30 min to provide heating or drying of the samples.

All samples were collected by centrifugation and dried for 48 h at 50 °C.

Characterization of materials

X-ray diffraction (XRD) was examined by (202964 PAN analytical Empyrean) using CuK α radiation ($\lambda = 1.54060 \text{ \AA}$) under the operating conditions of 30 mA and 40 kV. The scanning range was from 5° to 80° with a step size of 0.04° and a time per step of 0.5 s. Adsorbent's morphology was determined by the scanning electron microscope (ZEISS Gemini Scanning Electron Microscope). The morphology was depicted using Quanta FEG 250 (Czechoslovakia) field emission scanning electron microscopy (FESEM). The microstructure was examined using high-resolution transmission electron microscopy (HRTEM) model JEOL-JEM 2100 (Japan) with an acceleration voltage of 200 kV. The zeta potential was measured by Zetasizer Nano-Zs90 (Malvern, UK). The measurements were performed by withdrawing 10 μl of (corn leaves, ZnO and nanocomposite) suspension and followed by dilution in 1 ml of distilled water.

Adsorption experiment

Five hundred milligrams of solid biomass waste powder (corn leaves) was added to 50 ml of the solution prepared heavy metals (iron 40 ppm and nickel 16 ppm) at different pH. The beakers were held on an electric shaker, the sample fluid was collected from the mixture every 15 min to record a reading of heavy metal concentration using atomic

absorption spectroscopy and to compute the removal efficiency. To modify the acidity and/or alkalinity, a high-precision solution (HCl) and/or (NaOH) was added with a high-tech pipette to control the addition. The pH was determined using pH Meter equipment (Quimis; model Q400AS) (with a range of pH 3–6). The adsorption effectiveness of waste biomass (corn leaves) for heavy metal ion removal was investigated (iron and nickel). In all experiments, the adsorbent dosage was (500 mg biomass, 250 mg ZnO nanoparticles and 250 mg nanocomposite).

Results and discussion

X-ray diffraction (XRD)

XRD diffraction patterns of the three synthesized samples are illustrated in Fig. 1a–c. The patterns look very impressive because of the different crystalline phases depicted. The corn leaves were found to be amorphous where one broad hump was seen at $2\theta \approx 21$. The ZnO nanoparticles pattern was compared and indexed with the JCPDS file no. (04-016-6646). As it was observed, the nanoparticles ZnO were crystallized in hexagonal symmetry belonging to the space group P6₃mc(186) with two molecules per unit cell. The peaks are representative of nine reflections and many planes of the standard ZnO (Ferin et al. 2021; Rajesh et al. 2020) were of low intensities when compared to the bulk counterpart. These findings are characteristic of nanoscale dimensions. The reflections are depicted, indexed and reported in Table 1. The microstrain was found to have larger values and depends strongly on the diffraction angle. This is correlated to the hexagonal symmetry and the preferential. This is correlated

Table 1 Structural parameters of ZnO nanoparticles

2θ (°)	d value (Å)	I/I_0	Microstrain	(hkl)
31.8132	2.81292	57.26	0.404925	100
34.5356	2.59716	56.06	0.371260	002
36.2723	2.47670	100.00	0.244802	101
47.6460	1.90867	26.89	0.456261	102
56.6734	1.62422	37.29	0.309894	110
62.9257	1.47705	35.65	0.272966	103
68.0341	1.37805	25.23	0.196178	112
69.1876	1.35787	14.50	0.291751	201
77.1309	1.23665	3.66	0.337868	202

to the hexagonal symmetry and the preferential c -axis in the crystal. The crystalline size was computed using Debye Scherrer's formula and found to be 30 nm which is relatively small in hexagonal crystals. The nanocomposite pattern in XRD looks to be as crystalline nanoparticles embedded on an amorphous layer of the corn leaves. The pattern was distinguished by four main peaks of very small intensities and broad width. This is mainly due to the existence of 50% weight of corn leaves in the nanocomposite under investigation. The peaks are also shifted due to the above-mentioned reasons. For the nanocomposites, and from the main diffraction peak, the crystallite was calculated to be 8 nm. Which will be a key factor in the removal of heavy metals.

Field emission scanning electron microscope (FESEM)

The surface morphology of the studied samples, whether corn leaves, ZnO, or the formed nanocomposite, was

Fig. 1 a–c XRD of corn leaves, ZnO and nanocomposite

investigated using field emission scanning electron microscope (FESEM). From the modest magnifications up to 300,000 $\times$, it displays structural and elemental data. Computer software was used to make the micrographs at various magnifications. The original morphology of the corn leaf is illustrated in Fig. 2a–d, which provides information on the imaging and surface arrangement of the sample. The primary structure of CL is a rough surface. The literature indicates high cellulose content, low hemicellulose content, and low lignin content, which has been employed for various heavy metal absorption abilities (Jingjing et al. 2021). The leaves were found to have curls of their ends and rolled sheets. Even at lesser magnifications, pores and micro-cracks may be detected in the samples. The ZnO nanoparticles looked to have clear hexagonal plated with different sizes around 30–40 nm on average the slight wale scene is due to the absence of the surfactant as clear in Fig. 3a–d. Figure 3a, b clarified the successful formation of ZnO decorated on the surface of the corn leaves. The nanocomposite was found to be a wrinkled sheet decorated with ultrafine nanoparticles at higher magnifications as shown in Fig. 4a–d.

Mapping and EDX

The mapping of the nanocomposite (ZnO and corn leaves) sample is presented in Fig. 5; the elements of C, O, Na, Cl, K and Zn were detected. The weight percentage of the major elements was analyzed on the spectrum surface by the EDX. The electron image from Fig. 5a, shows that the nanocomposite contains many crystalline grains; this is a result of the formation of white colour minerals by the pores

present in the material's structure. Figure 6 shows EDX of the corn leaves and nanocomposite. The elements detected by EDX on the sample surface were 61.45 % of C, 23.28 % of O, 13.85 % of Zn, 0.6% of Cl and 0.83 % for K. Table 2 shows the mapping of nanocomposite (corn leaves and ZnO) (Table 3).

HRTEM and selected SAED

High-resolution transmission electron microscopy was used to study the microstructure of the produced samples in greater detail (HRTEM). Micrographs of corn leaves at various magnifications are shown in Fig. 7a–c, using a micrograph's scale bar, the morphology of the corn leaves particle size was assessed. Furthermore, the shells of the corn leaves were made up of nanoparticles with a cubic form and an average diameter of (nm), resulting in a corn-leaves-like structure. The SAED illustrate in Fig. 7d is a large spot identifying the amorphous nature of the corn leaves which is in good agreement with the pattern of XRD Fig. 1a. Figure 8a–c also depicts the morphology microstructure of ZnO at various magnifications. The particles are hexagonal with an average particle size of (50 nm). In Fig. 8d, the SAED shows preferred orientation as diffraction rings are pointing to an excellent crystallinity. This preferred orientation is certainly correlated to the hexagonal symmetry of the ZnO crystal. In addition, Fig. 9a–c depicts micrographs of corn leaves-ZnO nanocomposites at various magnifications. The ZnO particles and corn leaves were not chemically reacted. The SAED here for the nanocomposite Fig. 9d is interesting as the poor crystallinity predominates. Two diffraction rings

Fig. 2 FESEM of corn leaves, **a** 40 μm , **b** 20 μm , **c** 10 μm , **d** 4 μm

Fig. 3 FESEM of nano-ZnO, **a** 1 μm , **b** 500 nm, **c** 300 nm, **d** 200 nm

Fig. 4 FESEM of Nanocomposite, **a** 10 μm , **b** 5 μm , **c** 2 μm , **d** 400 nm

Fig. 5 Mapping of nanocomposite, **a–g** (corn leaves and ZnO)

Fig. 6 EDX of the corn leaves and nanocomposite, kV: 20, mag: 100, takeoff: 45.4, live time (s): 60, amp time (µs): 3.84, resolution: (eV) 129.5

Table 2 Mapping of the nanocomposite (corn leaves and ZnO)

Smart quant results								
Element	Weight %	Atomic %	Net int	Error %	K ratio	Z	A	F
C K	61.45	75.01	3490.44	6.77	0.2732	1.0474	0.4245	1.0000
O K	23.28	21.33	889.65	9.67	0.0403	1.0025	0.1727	1.0000
ZnL	13.85	3.11	734.09	3.33	0.0823	0.7619	0.7803	0.9996
ClK	0.60	0.25	92.66	2.72	0.0050	0.8498	0.9563	1.0148
K K	0.83	0.31	108.55	2.25	0.0072	0.8459	0.9958	1.0288

are clear and other planes seemed disappeared. This is the same result of XRD patterns where only broad reflections exist.

Table 3 Langmuir isotherm fitting parameter

Langmuir isotherm		
	Iron	Nickel
Corn leaves	$R^2 = 0.9867$	$R^2 = 0.9911$
ZnO	$R^2 = 0.9994$	$R^2 = 0.9976$
Nanocomposite (Corn leaves and ZnO)	$R^2 = 0.9983$	$R^2 = 0.9961$

Adsorption assessment

The adsorption efficiency was determined using the following formula shown in Eq. 1 (Rajesh et al. 2020; Saravanan 2021):

$$\text{Adsorption efficiency} = ((C_i - C_e) / C_i) \times 100, \quad (1)$$

where C_i and C_e are the initial and equilibrium concentrations (mg/L) of metal ion solution, respectively. The process of adsorption shows the efficiency of using the nanocomposite (corn leaves and ZnO) in the best record time to removal of heavy metals ion (iron/nickel).

Fig. 7 HRTEM of corn leaves, a 0.5 μm , b 500 nm, c 200 nm, d SAED

Fig. 8 HRTEM of ZnO, **a** 1 μm , **b** 500 nm, **c** 200 nm, **d** SAED

Effect of pH value and saturation time on the removal efficiency of iron ions

The process of removing iron ions from the aquatic environment using corn leaves, ZnO and nanocomposite are described here. The adsorption efficiency was plotted versus pH; corn leaves revealed the highest adsorption efficiency of 85.83% at pH 7 and the lowest efficiency of 59.4% at pH 3. ZnO showed an excellent adsorption efficiency of 97.68% at pH 7 and the lowest efficiency was 91% at pH 3. The nanocomposite had the highest adsorption efficiency of 96.4% at pH 7 and the lowest efficiency of 84.56% at pH 3. Figure 10 shows the relationship between removal efficiency of Iron ions and pH at contact time 270 min. From the results, the addition of ZnO to the nanocomposite leads to the enhancement of (Fe) removal percentage using corn leaves from 85.8 to 96.4% at the same conditions.

Figure 11 shows the best adsorption and fastest saturation time at pH 7 after 180 min for corn leaves, and the lowest saturation time was at pH 3 after 240 min. While the fastest saturation time of adsorption was at pH 7 after 180 min

and the slowest saturation time was at pH 3 after 240 min for (ZnO), the fastest saturation time of adsorption was at pH 7 after 180 min and the slowest saturation time was at pH 3 after 225 min for the nanocomposite. The value of saturation time is plotted versus the pH in Fig. 11 for the three investigated samples for the Fe ion removal process at room temperature. It was found that the saturation time is strongly dependent on the pH value of the solution. It is noted from the results that at pH 5, the saturation time of the nano composite is higher than the saturation time of the other two precursors of corn-leaves and zinc oxide.

Effect of pH value and time on the Ni ion removal efficiency

It is shown on Fig. 12, the process of Ni ion removal at room temperature for various pH values (3–8). The best adsorption efficiency of corn leaves was 88.52% at pH 7 and the weakest efficiency was 75.16% at pH 3. The highest adsorption efficiency of ZnO was 99.07% at pH 7 and the weakest efficiency was 91% at pH 3. The nanocomposite

Fig. 9 HRTEM of nanocomposite, **a** 500 nm, **b** 200 nm, **c** 1 μ m, **d** SAED

had the highest adsorption efficiency of 97.64% at pH 7 and the lowest efficiency of 83.18% at pH 3. It was discovered that the highest removal efficiency of nickel ion using

nanocomposite as improved performance for corn leaves at contact time 270 min.

Fig. 10 The relationship between removal efficiency of Iron ions and pH

Fig. 11 The relationship between the saturation time and pH

Fig. 12 The relationship between removal efficiency of nickel ions and pH

From the results, the optimum pH value is 7 for the 3 investigated samples at a stable time. The best efficiency order is 99% for ZnO > 97.85% for the nanocomposite and finally 88.52% for corn leaves. These results highlighted the role of ZnO nanoparticles in emphasizing the adsorption process of heavy elements using biomass.

Fig. 13 The relationship between the saturation time and pH to the removal of nickel ions

Figure 13 shows the analysis of the saturation time to remove nickel ions from water vs pH values (3–8) for nickel ions using corn leaves, nanoparticles (ZnO), and nanocomposite at room temperature. After 165 min, pH 8 had the fastest absorption saturation time, whereas pH 3 and pH 4 had the slowest absorption saturation time after 225 min. The fastest saturation time of absorption was at pH 6, pH 7 and pH 8 after 180 min, and the slowest saturation time was at pH 3 after 225 min for (ZnO) nanoparticles. After 165 min, the fastest saturation time of absorption for nanocomposite was at pH 7 and pH 8, while the slowest saturation time was at pH 4 after 225 min. However, at pH 4, the performance of the nano-composite is the best which propose that the composite perform better in the acidic medium.

Adsorption validation models

Langmuir isotherm analysis

$$Q_e(\text{mg/g}) = (C_i - C_e) \times (V/W)$$

Fig. 14 a–c Langmuir isotherm analysis of iron ions removal

$$q_{\max} = 1/\text{Intercept},$$

$$K_L = 1/(\text{slope} \times q_{\max}),$$

$$R_L = 1/(1 + C_i \times K_L),$$

where C_e is the equilibrium concentration (mg/l), V volume of the heavy metal solution in a litre, W weight of the material in the experiment in g, C_i is the initial concentration (mg/l). K_L (l/mg) and $q_{\max}$ (mg/g) are the Langmuir isotherm parameters (Sunil et al. 2021; Gyanendra and Mukesh 2021). From the results, the values of R^2 for the nano-composite is comparable with the nano zinc oxide which suggests that the

synthesized nano composite has good applicability because of its low cost compared to the nano zinc oxide. However, the performance of the nano-composite for the removal of iron ions is slightly better than nickel ions (Figs. 14, 15).

Freundlich isotherm analysis

$$\log q_e = \log K_F + (1/n) \log C_e,$$

where K_F (mg ((l/mg)— $1/n$)) and n are the Freundlich isotherm parameters (Lina et al. 2021). The results suggests that the adsorption behavior of the nano composite is examined

Fig. 15 a–c Langmuir isotherm analysis efficiency removal of nickel ions

Table 4 Freundlich isotherm fitting parameter

Freundlich isotherm		
	Iron	Nickel
Corn leaves	$R^2 = 0.9555$	$R^2 = 0.94$
ZnO	$R^2 = 0.9483$	$R^2 = 0.7743$
Nanocomposite (corn leaves and ZnO)	$R^2 = 0.9362$	$R^2 = 0.8338$

better using Langmuir isotherm than Freundlich isotherm. However, even after examination of the experimental results using Freundlich isotherm, the performance of the nanocomposite for the removal of iron ions is still better than nickel ions (Table 4, Figs. 16, 17).

Zeta size and zeta potential

Zeta potential (ZP) in millivolt (mV) value of corn leaves, ZnO, and nanocomposite material (corn leaves, ZnO) was

Fig. 16 a–c Freundlich fitting for the removal of iron

measured by a Zetasizer Malvern instrument. The results of the ZP analysis is demonstrated in Fig. 18a–f. The hydrodynamic diameter of the biomass particles (corn leaves) was found to be around (464.2) nm, which agrees well with previous characterization approaches. Those particles are negatively charged and have a potential value (− 7.91 mV), indicating that they are stable. In the case of ZnO nanoparticles, the hydrodynamic diameter here ensured particle aggregation, resulting in a size of about (13.46 nm). The presence of a negative charge on the surface of ZnO and its great stability have been discovered (− 91.3 mV). The

nanocomposite exhibited a peak around (382.6 nm). Furthermore, with a large value of (− 17.1 mV). The zeta potential remained negative. These results are profitable because of the high elimination efficiency in a short period. We could deduce that the positively charged metal cations are electrostatically attracted to the negatively charged particles on the surface-active sites of the three samples under examination which agrees with many of the published research (Xuefeng et al. 2021; Saad et al. 2021; Roman 2014).

Fig. 17 a–c Freundlich fitting for removal of nickel ions

Conclusion

The results suggest that nanoparticles of ZnO possessing nanoparticle size (30 nm) helped to improve the capability of corn leaves through the synthesis of the nanocomposite. It acts as an excellent bio-adsorbent for heavy metals such as Fe and Ni at optimum conditions of pH and adsorption time. The adsorption assessment and validation results showed that the nanocomposite material is competitive compared to

the nano zinc oxide. However, the integration of the biomass precursor and the nano zinc oxide resulted in low-cost applicable adsorbent for removal of heavy metals from aquatic environment. However, this approach may help to tackle two problems of agriculture solid waste such as corn leaves and heavy metals removal from the aquatic environment. The results suggest testing the proposed nanocomposite for industrial application.

Fig. 18 Zeta size **a** corn leaves, **b** ZnO and **c** nanocomposite respectively

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Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest. All the authors do not have any SOURCE OF FINANCIAL SUPPORT related to this publication.

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