Efficient removal of Cd (II) from contaminated water and soils using nanoparticles from nitrogen fertilizer industry waste

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Abstract

Background Cadmium (Cd) is used extensively in many industries and can cause environmental pollution and severe damage to human health. As millions of tons of lime-based solid by-product from nitrogen fertilizer industry (NFIB) are produced each year, the main purpose of this study was to develop a novel, efficient and cheap nanoscale sorbent from NFIB for remediation of Cd (II) contaminated soil and water to protect and preserve public and ecosystem health.

Methods A novel nanoscale adsorbent was developed from the nitrogen fertilizer industry byproduct (NFIB) and was characterized using X-ray diffraction (XRD) and scanning electron microscope (SEM). Batch sorption equilibrium and kinetic experiments were conducted to evaluate the efficiency of nano- NFIB (nNFIB) in sequestering Cd(II) in contaminated soil and water.

Results The results of adsorption equilibrium and kinetics experiments revealed that Langmuir and power function models best described Cd adsorption on bulk NFIB and nNFIB as evidenced by high R^2 (determination coefficient) and low SE (standard error of estimates) values. The Langmuir maximum adsorption capacity (q_{max}) of nNFIB for Cd(II) was 100 mg g^{-1} which is twenty times higher than that of Bulk NFIB. The distinguishing features of NFIB nanoparticles involve efficient removal of Cd(II) from contaminated water (>90%) and enhancement of Cd (II) immobilization (146%) in contaminated soil. Fourier Transmission Infrared (FTIR) spectra of Cd(II) contaminated water and soil before and after nNFIB application revealed the important role of calcite nanoparticles in Cd(II) sequestration.

Conclusions The accessibility, low cost, and Cd sequestration efficiency of nNFIB nominate it to be an economic and a promised adsorbent for environmental remediation.

Keywords Nanoparticles · Mechano – synthesis · Kinetics · Cd immobilization

Introduction

Cadmium (Cd) is largely used in various industries such as electroplating, nickel-cadmium batteries, pigments, plastics, pesticides, dyes and textile operations [1] (Wang et al. 2009) and can cause severe environmental pollution. Accumulation of Cd in humans has been reported to cause severe damage to the kidney, liver, brain functions and central nervous system [2] (Satarug et al. 2003). Therefore, low cost, yet efficient and environment-friendly technique is required for the remediation of Cd contaminated soil and water to protect and preserve public and ecosystem health.

A common practice frequently used to remediate Cd contaminated soils and water is in situ immobilization technique [3]. In this technique, different amendments like zero valent iron, liming materials, aluminum and iron oxides, and/or hydroxides have been used to reduce the potential toxicity of

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heavy metals to humans and the environment. Nowadays, biological wastes like oyster shells and eggshells [4] as well as industrial by-products like water treatment residuals [5–7] and fly ash [8] have been suggested as alternative cost effective sorbents for environmental remediation.

Nanotechnology is a branch of interdisciplinary research engaged with the creation of nanoscale structures for advanced applications [9]. The nanoparticles have enormous importance due to their exceptionally small size and big surface to volume ratio, and they exhibits new peculiar properties contrast to the large particles of bulk material [10]. Since millions of tons of lime-based solid by-product from nitrogen fertilizer industry (NFIB) are produced each year, the NFIB in nanoscale (nNFIB) can be used as cost-effective substitute of traditional high cost amendments for remediation of heavy metals contaminated soils and water. Therefore, the main purpose of this study was to develop a low cost and efficient nanoscale sorbent from by-products of nitrogen fertilizer industry for remediation of Cd (II) contaminated soil and water. The specific objectives were to synthesize NFIB nanoparticles for the first time and to evaluate the efficiency of nNFIB in sequestering Cd(II) in contaminated soil and water.

Materials and methods

Preparation and characterization of nanoscale nitrogen fertilizer industry byproduct (nNFIB)

The nitrogen industry byproduct (NFIB) was obtained from nitrogen fertilizer company, Alexandria, Egypt. The NFIB samples were collected, air -dried, ground and passed through two different sieves having pore diameters of 2 mm (mNFIB) and 51 μm (μNFIB). Nanoparticles of nNFIB were produced using subsamples of μNFIB (<51 μm) and Planetary Mono Mill according to the method of Elkhatib et al. [6]. The crystalline nature of nNFB was determined using Bruker AXS D8 Advance X-ray Diffractometer. The particles size, morphology and elemental composition of nNFIB were investigated using scanning electron microscope equipped with energy dispersive spectroscopy (SEM-EDS) (INCAx-Sight, Oxford Instruments, UK). Surface area of nNFIB was determined using autosorbiQ surface area analyzer (Quanta chrome, USA).

Sorption isotherms

Cadmium (II) sorption equilibrium studies were performed on mNFIB and nNFIB at normal pH (7.2) using 0.01 M KNO_3 and Cd(II) concentrations ranging

from 10–500 mg/L. The NFIB–Cd mixtures (in replicate) were equilibrated on a shaker for 24 h, centrifuged for 10 min at 4000 rpm, filtered through a membrane filter (0.45 μm) and the filtrate was analyzed for Cd by Atomic absorption spectroscopy (AAS). Seven sorption isotherm models were assessed for their ability to fit the sorption data [6]. The sorbed Cd by nNFIB was examined via SEM equipped with an EDS (INCAx-Sight model 6587, Oxford Instruments, UK).

Sorption kinetics

Batch sorption kinetic experiments were conducted with Cd at room temperature (25 ± 2 °C). A known dose of nNFIB (150 mg) was mixed with 20 mL of Cd (II) solutions with initial concentration of 500 mg/L in 50 mL centrifuge tubes. The mixtures (in replicate) were shaken for different time intervals (5 min–24 h) using an end-over-end shaker at 3 different pH levels (pH 5, 7 and 9). The pH was kept constant by automatic titration with HCl or NaOH. The Cd - nNFIB suspensions were centrifuged and then filtered using 0.45 μm Millipore filter. Atomic absorption spectrometry (AAS, Perkin Elmer AAnalyst 300) was used to analyze Cd (II) concentrations in the supernatant solutions. Stock standard Cd (NO_3)₂ solution (1000 mgL^{-1}) was used to prepare cadmium(II) solutions. The kinetics of Cd sorption on the nNFIB samples were investigated by fitting the sorption data to power function, parabolic diffusion, first order, and Elovich kinetic models [11].

Spiking biosolids and incubation experiment

Biosolids samples originally contained 2.38 mg kg^{-1} Cd were collected and spiked with Cd nitrate at a rate of 135 mg Cd kg^{-1} (the soil Cd concentration limit recommended by the USDA [12]). The Cd-spiked biosolids samples were incubated under aerobic conditions at room temperature (25 ± 2 °C) and 70% water holding capacity for 30 days. At the end of incubation period, the Cd-spiked biosolids were mixed with 2 kg of sandy soil. Four different rates of nNFIB (0, 0.25, 0.5, and 1%) were added to soil -biosolids mixtures, placed in plastic bags, and incubated for 20 days at room temperature (25 ± 2 °C). During the incubation period, moisture content of the mixtures was maintained at 70% of water holding capacity (WHC).

Cadmium fractionation

The procedure of Tessier et al. [13] was used to fractionate Cd in soil-biosolids mixtures before and after application of nNFIB. The used procedure fractionates

Cd into five fractions: Exchangeable(Exch), carbonates (Carb), Fe–Mn oxides (FeMnO), organic matter (OM) and Residual(Res). Cadmium in the fractions was determined using AAS(Perkin Elmer AAnalyst 300).

Statistical analysis

Microsoft Excel and the linear regression COSTAT programs were used to analyze the data. Separation of differences among the treatment means was performed by using Fisher's least significant difference at level of significance $P \leq 0.01$ [14] (SAS2000).

Results and discussion

Characterization of nanoscale nitrogen fertilizer industry by-product

The XRD patterns of bulk NFIB and nanoscale NFIB (Fig. 1) demonstrate a strong characteristic peak at $2\theta = 30^\circ$ indicating that both samples are mainly containing high percentage (93%) of calcite (CaCO_3). The SEM and EDS analyses of both samples confirmed XRD results and ascertained that the main component of bulk and nanoscale NFIB is calcite (Fig. 2a, b). SEM and EDS analyses of NFIB nanoparticles(nNFIB) before and after Cd saturation are shown in Fig. 2b, c. The SEM image of nNFIB sample before Cd saturation revealed the spherical shape of nanoparticles and the representative single particle sizes are less than 100 nm in diameter and the EDS analysis revealed that calcium percentage was decreased from 93.3 to 87.7% of the total elements in nNFIB, in addition to magnesium, silicon, iron, zinc and copper (Fig. 2a). The SEM image of Cd saturated nNFIB revealed that adsorbed Cd has formed a coating layer on nNFIB surface as a result of saturating nNFIB with Cd (Fig. 2c). The presence of Cd peak (4.2%) is also confirmed by EDS spectrum of Cd saturated nNFIB (Fig. 2c).

The specific surface area of nNFIB ($225.4 \text{ m}^2 \text{ g}^{-1}$) is much higher than that of the bulk NFIB sample ($8.8 \text{ m}^2 \text{ g}^{-1}$). Figure 3a has shown a continuous increase of Cd sorbed by nNFIB and mNFIB with increasing Cd concentration from 5 to 500 mg L^{-1} . However the amounts of Cd sorbed by nNFIB was much higher than that sorbed by mNFIB. Reliable prediction of Cd adsorption parameters including maximum sorption capacity was further analyzed using seven isotherm models (Freundlich, Langmuir, Elovich, Temkin, Fowler–Guggenheim (FG), Kiselev, and Hill-de Boer) [10]. The high capability of Langmuir and Fowler–Guggenheim models to describe Cd sorption data in the order Langmuir > Fowler–Guggenheim is evidenced by the high values of determination coefficient (R^2) and low values of standard error of estimate (SE) of these two models (Table 1). The superiority of

Langmuir model to describe Cd(II) adsorption on nNFIB and mNFIB (Fig. 3b) indicate the involvement of monolayer adsorption in the Cd(II) adsorption process by nNFIB and mNFIB [15]. The Langmuir maximum adsorption capacity (q_{max}) of nNFIB for Cd(II) is 100 mg g^{-1} which is twenty times higher than that of Bulk NFIB. This is not surprising since the BET specific surface area of nNFIB is 25 times higher than BET specific surface area of the Bulk NFIB. High surface area of nanoparticles greatly enhance the adsorption capacity and surface reactivity of nNFIB [16]. Therefore, modifying bulk NFIB to nanoparticles greatly enhanced its capability for Cd removal from contaminated water.

Effect of adsorbent-sorbate contact time

Cadmium adsorption data by the nNFIB material as a function of time are presented in Fig. 3c. Apparently, Cd(II) adsorbed onto the nNFIB was initially rapid as more than 90% of Cd(II) was removed from solution in the first 10 min, and then Cd(II) adsorption advanced slowly until equilibrium was reached in about 24 h. The fast Cd removal is attributed to the high proportion of calcium carbonate (more 93%) in nNFIB and with time the availability of adsorption active sites rendered non available. Song et al. [17] and Ma et al. [18] reported that the abundance of accessible sorption sites on CaCO_3 surface could be the cause of the initial rapid adsorption rate. Cadmium sorption kinetics studies performed on nNFIB at different pH values (5, 7 and 9) clearly show that the efficiency of nNFIB to remove Cd(II) ions from solution is affected by the initial pH values of solutions i.e. Cd(II) adsorption on nNFIB increased with increasing initial pH of the solutions (Fig. 3c). At high pH values ($\text{pH} > 7$) the surface charge of nNFIB became more negative and could lead to high Cd(II) sorption.

The selected kinetic models were employed to fit the experimental data. R^2 and SE values of the kinetic models tested indicate that Cd(II) adsorption data were in best agreement ($p < 0.01$) with the power function model (Table 1 and Fig. 3d). The adsorption rate () of the power function model increased from 3.22×10^4 to $5.86 \times 10^4 \text{ min}^{-1}$ with the increase in the system pH from 5 to 9 (Table 1) which indicates that Cd sorption is preferably at high pH values. At low pH ($\text{pH} = 5$), 60% of Cd(II) was removed from aqueous solution by nNFIB in the first 10 min, and the percentage of Cd(II) removed was increased to reach 89% at equilibrium time(24 h). Meanwhile, it was observed that the solution pH changed rapidly to around circumneutral value (7.2) when the nNFIB was placed into a solution with initial pH 5. These results are also supported by Wang et al. [1]. The high Cd(II) removal efficiency of nNFIB at low pH value($\text{pH} = 5$) could be attributed to the increase of solution pH as a result of calcitr dissolution of as

Fig. 1 The X-ray diffraction (XRD) analyses of **a** bulk NFIB and **b** nNFIB

follows [17]:

With increasing pH values ($\text{pH} > 7$), the surface charges of nNFIB became more negative which may accelerate ion-exchange reaction between $\text{Cd}(\text{II})$ and $\text{Ca}(\text{II})$ and cause greater sequestration of $\text{Cd}(\text{II})$ at the nNFIB surface [19].

Fig. 2 Scanning electron microscopy (SEM) image and energy dispersive X-ray (EDX) elemental distribution of **a** mNFIB, **b** nNFIB and **c** the Cd-loaded nNFIB

FT-IR analyses was performed on nNFIB and Cd(II)-loaded nNFIB samples to elucidate Cd(II) removal mechanism (Fig. 4). The intensities of the bands corresponded to amorphous calcium carbonate at 1085, 875 and 712 cm^{-1} in the Cd(II)-loaded nNFIB samples clearly decreased while the symmetric stretch at 1398 cm^{-1} broadens, and shifts to 1436 cm^{-1} . However, intensities of other adsorption bands did not remarkably change (Fig. 4). These results indicate that important role of CaCO_3 in Cd(II) adsorption by nNFIB through ion exchange, and precipitation mechanisms [20, 21].

Cadmium removal in single and multielement system by nNFIB

To evaluate Cd adsorption by nNFIB in single and multielement system, batch adsorption experiments were performed in the absence and presence of two competing cations (Cu and Pb) at concentrations equal to Cd concentration. The percentage of Cd removed from the single system by nNFIB was little higher than the percentage of Cd removed from the multielement (Cd, Cu and Pb) system (Fig. 5). The percentage of Cd removed by nNFIB from single system was higher than Cd

Fig. 3 **a** Cadmium adsorption isotherms for two different particle sizes of NFIB, **b** Langmuir isotherms of Cd(II) adsorption onto mNFIB and nNFIB materials, **c** Kinetics of Cd(II) removal by nNFIB at three

different pH values, **d** power function model for Cd(II) adsorption by nNFIB at different pH values, **e** Fourier transmission infrared (FTIR) spectra of nNFIB and Cd-loaded (II) nNFIB

removed from multi-system by 0.75, 1.65, 4.48, 4.75, and 5.38% at Cd concentration of 5, 20, 40, 80 and 160 mg L⁻¹ respectively. These results seem to concur with the findings of Appel et al. [22], they reported higher removal efficiency of limestone in single system than do in mixed systems.

Effect of nNFIB application on Cd (II) sequestration in contaminated soil

The effect of nNFIB application on distribution of Cd fractions in contaminated biosolids amended soil is presented in Fig. 6a. The percentages of cadmium fractions in nNFIB un-amended soil followed the order: OM ~ Carb > Res > Exch > FeMnO. Application of nNFIB to the contaminated biosolids amended soil at rates of 0.25, 0.50 and 1.0% greatly reduced the Exch-Cd and OM-Cd fractions and simultaneously increased carbonate fractions. In the soil amended with 1.0%

nNFIB, ~74% of Cd (II) was associated with the carbonate fraction, whereas organic (13.66%), oxides (2.48%), and exchangeable (1.99%) fractions represent the minor association. Cadmium bound to carbonate increased by 116%, 131% and 146% for the 0.25%, 0.50% and 1.0% nNFIB treatment, respectively; such increase indicated that nNFIB application significantly increased Cd association with carbonate fraction and consequently enhanced Cd (II) immobilization in the soil studied. These results are in accord with the findings of Rajaie et al. [23], who reported that most of the native Cd in a calcium carbonate rich soil was bound to carbonate and residual fraction and rendered unavailable. Chen et al. [24] reported significant transformation of exchangeable (mobile) form of Cd to unavailable form after amendment with calcium carbonate. Therefore, it is suggested that the use of nNFIB for Cd sequestration could provide long-term geochemical stability for heavy metals in contaminated soils.

Table 1 Equilibrium and kinetics model constants and statistical parameters for cadmium adsorption by mNFIB and nNFIB

Equilibrium models					
		Parameter	m NFIB	n NFIB	
Langmuir	$q_e = q_{\max}(K_L C_e/1 + K_L C_e)$	$q_{\max}$ ($\mu\text{g g}^{-1}$)	0.50×10^4	10.0×10^4	
		K_L (L mg^{-1})	0.50	−0.03	
		R^2	0.995	0.995	
		SE	4.30×10^{-5}	4.32×10^{-5}	
Fowler–Guggenheim(FG)	$K_{FG}C_e = \theta/1 - \theta \exp(2 \theta w/RT)$	$W(\text{kJmol}^{-1})$	−2.38	2.04	
		$K_{FG}(\text{L mg}^{-1})$	1.61	0.84	
		R^2	0.99	0.83	
		SE	0.06	0.15	
Kinetics models					
Parameter		n NFIB			
First-order	Parameter	pH 5	pH 7	pH 9	
	$k_a \text{ min}^{-1}$	0.007	0.003	0.002	
	$a \mu\text{g g}^{-1}$	10.03	6.91	8.41	
	$\ln (q_0\text{-}q) = a - k_a t$	R^2	0.96	0.85	0.88
		SE	0.312	0.206	0.131
	$k_a \text{ min}^{-1}$	3.22×10^4	5.64×10^4	$5.86 \times 10^4 \times 10^4 \times 10^4 \times 10^4$	
Power function	$1/m$	0.100	0.007	0.013	
	R^2	0.94	0.86	0.91	
	$q = k_a C_o t^{1/m}$	SE	0.036	0.003	0.004

q_e (mg g^{-1}) is Cd adsorbed per gram of adsorbent, C_e (mg L^{-1}) is equilibrium Cd concentration in solution, $q_{\max}$ (mg g^{-1}) is the maximum adsorption capacity of the adsorbent, K_L (L mg^{-1}) is Langmuir constant related to the free energy of adsorption, θ is fractional coverage, R is the universal gas constant ($\text{kJ mol}^{-1} \text{K}^{-1}$), T is the temperature (K). q is Cd adsorbed (mg kg^{-1}) at time (min), q_0 is Cd adsorbed (mg kg^{-1}) at equilibrium, k_a is apparent sorption rate coefficient, C_0 is initial Cd concentration (mg L^{-1}), k_a is sorption rate coefficient (min^{-1}), and $1/m$ is constant. R^2 is determination coefficient and SE is standard error of estimate

To further investigate Cd sequestration mechanism onto nNFIB treated soil, FTIR spectroscopy for biosolids amended sandy soil before and after nNFIB application were carried out. The FTIR analyses of the biosolids amended soil (Fig. 6b) exhibits remarkable bands attributed to OH vibrations of molecular water, $(\text{CO}_3)^{2-}$ stretching mode vibration, and Si-O

Fig. 4 Fourier transmission infrared (FTIR) spectra of nNFIB and Cd-loaded (II) nNFIB

stretching vibration at 3409, 1435, 876 and 1033 cm^{-1} respectively [25, 26]. Application of nNFIB to Cd-loaded biosolids amended soil caused strong interaction of Cd ions with OH groups as indicated by a shift of OH bonded water band at

Fig. 5 Cadmium (II) removal in single and multielement system by nNFIB

Fig. 6 **a** percentage of Cd fractions in sandy soil with biosolids (3%,w/w) and amended with nNFIB at different rates, **b** Fourier transmission infrared (FTIR) spectra of Cd-loaded sandy soil amended biosolids before and after nNFIB application

3409 cm^{-1} to higher wave numbers and by a reduction in its intensity. Similarly, the interaction of Cd^{2+} ions with Si-O of sandy soil caused a decrease in the intensity of Si-O stretching vibration band at 1033 cm^{-1} . In addition increasing CO_3^{2-} content in the biosolids amended sandy soil as a result of nNFIB application increased the intensity of the bands corresponded to calcite at 1435 and 876 cm^{-1} (Fig. 6b). The FTIR results clearly showed the important role of calcite in Cd(II) adsorption and suggested precipitation transformation mechanism for Cd adsorption on nNFIB materials [27, 28].

Conclusions

Effective sequestration of Cd(II) in contaminated soil and water has been successfully developed using the nanostructured NFIB. The capability of NFIB nanoparticles to remove Cd(II) greatly enhanced and reached nearly 20 times higher than that of bulk NFIB. Such powerful capability of the low cost nNFIB in sequestration of Cd(II) has demonstrated the great potential of these nanoparticles in water and soil remediation.

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Compliance with ethical standards

Declaration Not applicable.

Ethics approval and consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare that they have no competing interests.

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