

Conversion of Oyster Shell Waste to Amendment for Immobilising Cadmium and Arsenic in Agricultural Soil

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Abstract

A bulky waste, oyster shell (OS), was calcinated at 400–800°C to produce Ca-rich products ($OS_{400}-OS_{800}$) to reduce the human health risk of soil cadmium (Cd) and arsenic (As). Thermogravimetric analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), and BET method were used to characterize OS and its calcined products. OS and $OS_{400}-OS_{700}$ removed little Cd and As from water, whereas OS_{800} removed 1508 mg Cd or 514 mg As per kg of OS_{800} from solutions of 1032 mg Cd/L or 257 mg As/L via adsorption and precipitation. Adding OS_{800} at a 2% dose to a Cd- and As-contaminated soil lowered its exchangeable Cd from 60% to 27%, and reduced Cd content in the edible part of vegetable Bok Choy from 2.80 to 0.048 mg/kg and As from 1.73 to 0.47 mg/kg. Converting OS to soil amendment has the dual benefits to soil remediation and sustainable oyster aquaculture.

Keywords Oyster shell waste · Soil remediation · Cadmium · Arsenic · Bioavailability

Soil contamination by heavy metal(loid)s in China often occurs in compound forms, of which the combination of cadmium (Cd) and arsenic (As) is a common one. A national soil survey in China revealed that Cd content in 7.0% of sampling sites and As in 2.7% sites exceeded national standards (MEPRC 2014). The non-biodegradability of heavy metal(loid)s contributes to their hidden, irreversible, and accumulative features of soil contamination (Jolly et al. 2013). Soil contamination by Cd and As not only affects agricultural production (Singh et al. 2015) but also poses a threat to animal and human health and hinders social development (Khan et al. 2017).

The remediation of Cd- and As-contaminated soils often follows the mobilization or immobilization approach (Bolan et al. 2014). The former involves the removal of heavy metal(loid)s from soils by synthetic chemicals, such as acids, surfactants, chelating agents, and salts (Beiyuan et al. 2018), or by natural humic substances (Bi et al. 2019). In contrast, the immobilization approach does not reduce the total contents of heavy metal(loid)s in soil but reduces their solubility, reactivity, and bioavailability, thus reducing their uptake by plants. In this regard, a range and variety of materials have been investigated for their adsorptive properties and effectiveness in immobilizing metal(loid)s, including minerals (Yuan et al. 2013), waste or by-products (Wang et al. 2014), humic substance (Meng et al. 2017), and a combination of them (Gu et al. 2019). Despite these advancements, studies on As have been relatively fewer (Chen et al. 2018) and work on simultaneous immobilization of Cd and As, two metal(loid)s with very different chemical behaviors in soil, has been rare (Qiao et al. 2018). It is desirable to remediate lightly contaminated soils for the safe production of cereals and vegetables, to which converting waste to soil amendments would be an appealing option.

China produced 4.57 million tons of oyster in 2015 (Mao et al. 2019), accounting for about 80% of world production. A large amount of oyster shell (OS) has been piled on the coast, producing odor, breeding insects, and polluting water (de Alvarenga et al. 2012). Developing beneficial use of OS waste is a requirement for sustainable oyster aquaculture.

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Oyster shell is a natural nanocomposite, consisting of brick-like layers of calcium carbonate (ca. 96% by weight) (Silva et al. 2019) and thin layers of protein as organic glue. OS has been investigated to remove Cd (Lee et al. 2018) and phosphorus (Liu et al. 2010) from water. We hypothesize that its kinetic ability to remove heavy metal(loid)s would be enhanced by (1) thermally removing its protein layer, thus creating micropores with a large specific surface area (SSA), and (2) converting CaCO₃ to CaO (Ok et al. 2010). This study, therefore, aimed to find a suitable temperature for thermal treatment, determine the capacity of calcined OS to combine Cd and As, and assess its usability for Cd and As immobilization in contaminated soil.

Materials and Methods

The topsoil (0–20 cm) was sampled following a S-route from a vegetable farm along the lower reach of the Xijiang River (West River) in Foshan City, Guangdong Province (22°58'N, 112°52'E). After the removal of foreign debris and animal and plant residues, the composite sample was air-dried, gently crushed, and sieved through a 2-mm nylon mesh for use. Laboratory analyses of the soil sample included particle size distribution by a particle size analyzer (Mastersizer 2000, UK), pH in distilled water (1:5, w/v) by a pH meter (Mettler Toledo Five Easy Plus, Switzerland), Cd and As concentrations by digestion with $HClO_4$ - HNO_3 -HF at a 3:1:1 ratio (v: v: v) for ICP-MS analysis (Inductively Coupled Plasma Mass Spectrometry, PerkinElmer Elan DRC II, USA) (Lu 1999). The recovery rate of a certified reference soil in the continuous digestion process was more than 95%.

Oyster shell was collected from Yantai, Shandong Province (37°28'N, 121°26'E). After cleaning with a brush to remove surface sediment and impurities, the shell was dried in an oven at 105°C, crushed, sieved through a 2-mm nylon mesh (labeled as OS), and placed in crucibles for calcination at 400, 500, 600, 700, and 800°C for 4 h in a muffle furnace and then ground to pass through a 0.25-mm nylon mesh (referred to as OS_{400} , OS_{500} , OS_{600} , OS_{700} , and OS_{800} , respectively). They were observed or analyzed for morphological features by SEM (HITACHI S-4800, Japan), mineral compositions by XRD (Bruker D8 Discover, Germany), thermogravimetric properties by Mettler 5MP (Switzerland), SSA by N₂ adsorption at 77 K using an Autosorb-1 gas analyzer (Quantachrome Autosorb-iQ, USA), and Cd and As concentrations by HClO₄-HNO₃-HF digestion and ICP-MS analysis.

Stock solutions, prepared from $Cd(NO_3)_2 \cdot 4H_2O$ or $Na_3AsO_4 \cdot 12H_2O$, were diluted to get working solutions of 0, 25, 50, 300, 700, 900, 1050, 1500 mg Cd/L or 0, 5, 10, 25, 100, 150, 200, 250 mg As/L, with 1 mM NaNO₃ as the background electrolyte. After the pH was adjusted to 6.0 with 0.5 M HCl, 40 mL of solutions were added into centrifuge tubes with 20 mg OS or OS₄₀₀₋₈₀₀, and the tubes were shaken in an oscillator (25 °C, 300 r/min) for 48 h and then centrifuged at 3000 r/min for 10 min. The supernatant was filtered through a 0.45-µm filter to analyse Cd and As concentrations by ICP-MS. The experiment was conducted in triplicate.

Based on the capacity of OS and its calcined products to remove Cd and As from water, OS_{800} was chosen to immobilize Cd and As in soil, at doses of 0, 0.5, 1.0, 2.0, and 5.0% (w: w). The soil- OS_{800} mixtures were first aged at 70% field capacity for 30 days at room temperature, and then air-dried and sieved through a 2-mm nylon mesh for Cd and As fractionation by Tessier sequential extraction method (Gabarrón et al. 2019), using 1 M MgCl₂ for exchangeable fraction, 1 M NaOAc for carbonate-bound fraction, 0.04 M NH₂OH·HCl for Fe–Mn oxide-bound, 0.02 M HNO₃, 30% H₂O₂, and 3.2 M NH₄OAc for organically bound, and digestion with HClO₄ and HF for the residual fraction. The recovery rates of the certified reference material in the continuous extraction process were more than 95%.

The plant availability of Cd and As in the soil was assessed by carrying out a pot experiment, using a popular vegetable Bok Choi or Pak Choi (*Brassica rapa chinensis*) as an indicator. The treatments included OS_{800} at 0, 0.5, 1.0, 2.0, and 5.0% dosages in triplicates. Briefly, after the germination of seeds three plants were kept for growth in each pot in an artificial climate chamber (25°C, 55% relative humidity) for 30 days, during which soil moisture content was maintained at 70% field capacity. In the end, Bok Choi was harvested, dried, and digested by nitric acid (Havlin and Soltanpour 1980) for the analysis of Cd and As contents by ICP-MS.

Excel 2013, SPSS 16.0, and Origin 9.0 were used for calculation, data analysis, and figure drawing. One-way analysis of variance was performed for statistical significance analysis (Duncan's test, p < 0.05).

Results and Discussion

The soil was slightly acidic, with a silty loam texture (Table 1). Its total Cd concentration exceeded the control value (2.0 mg/kg) in the *Risk Management Standard for*

Table 1Particle sizedistribution, pH and metal(loid)concentrations of the soil

Properties	Sand (%)	Silt (%)	Clay (%)	pН	Total Cd (mg/kg)	Total As (mg/kg)
Soil sample	41.73 ± 0.44	46.25 ± 0.92	12.02 ± 0.63	6.03	5.23 ± 0.37	9.88 ± 0.73



Fig. 1 Thermogravimetric changes of the oyster shell during calcination

Table 2 Properties of oyster shell and its calcined products

Samples	рН	Specific surface area (m ² /g)	Total Cd (mg/ kg)	Total As (mg/kg)
OS	8.97	4.33±0.35d*	0.016 ± 0.003	0.434 ± 0.196
OS ₄₀₀	10.32	$10.24 \pm 0.12a$	0.019 ± 0.004	0.451 ± 0.216
OS ₅₀₀	11.28	7.03 ± 0.19 b	0.016 ± 0.000	0.589 ± 0.184
OS ₆₀₀	11.89	$3.34 \pm 0.21e$	0.025 ± 0.001	0.571 ± 0.401
OS ₇₀₀	12.17	$5.88 \pm 0.41c$	0.023 ± 0.005	0.628 ± 0.251
OS ₈₀₀	12.75	9.85±0.71a	0.026 ± 0.001	0.708 ± 0.323

*Different lower-case letters indicate a significant difference (at p < 0.05) between samples

Agricultural Land for Soil Pollution in China, posing a high risk to food production and safety. The remediation of the soil would be desirable for its current land use for vegetable growth.

During heating, OS gradually decomposed (Fig. 1) and its weight loss occurred at two stages: (1) water loss and organic matter decomposition from room temperature to ca. 600° C resulted in a weight loss of 14%, and (2) the conversion of CaCO₃ to CaO above 600°C (Mohamed et al. 2012) incurred 41% weight loss close to the theoretical value. At 800° C, both stages were completed. Kobatake and Kirihara (2019) reported a similar link between CaO formation and temperature.

During heating, organic matter decomposition at 400°C (Choi et al. 2011) created voids between layers of CaCO₃ and increased SSA of OS (Table 2) by exposing the inner surface. When the temperature was increased to 600° C, the layer structure of CaCO₃ collapsed, resulting in a decrease in SSA. As CaCO₃ started to decompose from 600° C and released CO₂, the condense CaCO₃ became loose CaO, further increasing SSA from OS₆₀₀ to OS₈₀₀. Alidoust et al. (2015) reported similar trends in SSA changes during OS calcination.

Figure 2 clearly show the lamellar structure in OS (left). The observed morphology was similar to Alberts et al. (2015) reported. At 800°C, organic matter disappeared, and lamellar CaCO₃ became micrometer-size particles of CaO with regular rectangular block structure (Fig. 2, right). As particle size decreased, SSA increased.

The mineralogical changes during calcination are shown in Fig. 3. The main peak of OS at $2\theta = 29.3$ was assigned to calcite (CaCO₃, JCPDS No. 86-0174), and the main peak of OS₈₀₀ at $2\theta = 37.4$ was due to calcium oxide (CaO, JCPDS No. 48-1467). The mineralogical changes from calcination agree with pH values in Table 2 and with Ok et al. (2010) that the primary component of calcined OS was CaO.

The abilities of OS and OS_{400} – OS_{800} to remove Cd and As from aqueous solutions are shown in Fig. 4. OS removed little Cd and As. Whereas calcined OS removed a varied amount of Cd and As. Particularly, OS_{800} had the highest ability to combine Cd, which is in agreement with its highest pH value (Table 2) and with the formation of CaO (Fig. 3).

Calcination enhanced the ability of OS to remove Cd for two reasons. First, calcite in OS is barely soluble in water (0.013 g/L at 25°C) with a solubility product (K_{sp}) of 3.3×10^{-9} . In other words, Cd²⁺ sorption to calcite via ion exchange with Ca²⁺ was very limited. Whereas CaO reacts with water to become Ca(OH)₂, the latter has a much larger solubility of 1.73 g/L (20°C) and K_{sp} of 5.5×10^{-6} . Second, the high pH of calcined OS would precipitate Cd²⁺.

OS and OS_{400} - OS_{700} had low capacities to remove As from aqueous solution (Fig. 4b). OS_{800} , however, could

Fig. 2 Scanning electron micrographs of oyster shell (left) and its 800°C calcined product (right)





Fig.3 X-ray diffraction patterns of oyster shell (OS) and its calcined products at different temperatures: (filled diamond) Calcite and (filled circle) Calcium oxide

react with water to form Ca(OH)₂, its ionization produced Ca²⁺ and reacted with AsO₄³⁻ to form Ca₃(AsO₄)₂ at high pH (Hu et al. 2014). Although Ca(OH)₂ is only slightly soluble in water (1.73 g/L at 20°C), its solubility product K_{sp} (5.5×10⁻⁶) was much higher than that of Ca₃(AsO₄)₂·3H₂O (10^{-21.14} at 25°C) (Zhu et al. 2006), resulting in continuous removal of As, as simplified below:

$$CaO + H_2O \rightarrow Ca(OH)_2 \tag{1}$$

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^-$$
(2)

$$3\operatorname{Ca}^{2+} + 2\operatorname{AsO}_4^{3-} \to \operatorname{Ca}_3(\operatorname{AsO}_4)_2 \downarrow$$
 (3)

The bio-availability of heavy metal(loid)s in soil is influenced by their chemical forms. A high content of the exchangeable heavy metal(loid)s led to high toxicity to plants (Pavlovic et al. 2018). With increasing dosage, OS_{800} reduced the exchangeable Cd (bioavailable) in the soil from 60.2 to 9.2% (Fig. 5). OS_{800} was more effective in reducing exchangeable Cd than other materials reported in the literature, such as steel slag (Zhuo et al. 2012) and biochar (Puga et al. 2015). OS_{800} also affected arsenic fractions in the soil, but in an inconsistent trend. As discussed above (K_{sp}), the formation of new compounds with very low solubility complicated the usability of sequential extraction methods for As.

The addition of OS_{800} to the soil at 0.5%-2.0% doses maintained or enhanced Bak Choi growth, as the aboveground biomass (Table 3) indicates. A 5.0% dose dramatically increased soil pH and hindered Bak Choi growth. Besides its impact on biomass, OS_{800} also effectively reduced the Cd and As contents of Bak Choi. At a 2.0% dose, Cd content in the edible part was reduced by 98% and As by 73%, both below the limits in Chinese national food safety standards (Cd: 0.05 mg/kg, As: 0.5 mg/kg). Further, OS_{800} was produced from waste, and its Cd (0.026 mg/kg) and As (0.708 mg/kg) contents were low. Thus, its use for the remediation of Cd- and As-contaminated soil merits field trials in different types of soils for various vegetables.

In summary, calcination of an oyster shell at 800°C produced a CaO-dominant material (OS_{800}) with a high pH, moderate SSA, and low Cd and As contents. It had excellent capacities to remove Cd and As from aqueous solution via adsorption and precipitation. Its use as a soil amendment at a 2.0% dose reduced the exchangeable Cd of the soil by 55% and lowered Cd and As contents in the edible part of a popular vegetable Bak Choi by 98% and 73%, respectively, making the vegetable safe for consumption. OS_{800} is a safe, inexpensive, and effective amendment for the remediation of Cd- and As-contaminated soil for vegetable production. This



Fig. 4 OS and its calcined products for Cd (a) and As (b) removal at various equilibrium concentrations



Table 3 Effects of OS_{800} addition to soil on Cd and As contents (mg/kg) in vegetable Bak Choi

Fig. 5 Fractions of Cd (left) and

As (right) in OS₈₀₀-amended

soil

OS ₈₀₀ dose		CK (0%)	0.5%	1.0%	2.0%	5.0%
Soil pH	at sowing	6.03	7.64	7.81	8.72	10.86
	at harvest	6.05	7.31	7.26	7.33	9.00
Leaf dry weight (g/pot)		$0.46 \pm 0.12b^*$	$0.43 \pm 0.15b$	$0.46 \pm 0.09 b$	$0.76 \pm 0.05a$	0.12 ± 0.01 c
Leaf	Cd	2.80 ± 0.73 a	$2.53 \pm 0.36a$	$0.72 \pm 0.14b$	$0.048 \pm 0.00c$	$0.07 \pm 0.02c$
	As	$1.73 \pm 0.04a$	1.00 ± 0.40 b	$1.15 \pm 0.26b$	$0.47 \pm 0.16c$	$0.51 \pm 0.17c$
Root	Cd	$2.28 \pm 0.42a$	$1.83 \pm 0.52a$	$1.24 \pm 0.26a$	$1.65 \pm 0.06a$	$1.52 \pm 0.84a$
	As	$2.72 \pm 0.01a$	$2.27 \pm 0.13a$	$1.62 \pm 0.59a$	$1.94 \pm 0.03a$	$1.91 \pm 1.53a$

*Different lower-case letters indicate a significant difference (at p < 0.05) between samples

use would also help sustainable aquaculture by alleviating its waste problem, and lessen the environmental damage from limestone mining for lime production. As OS is widely available and simple to activate, it would be particularly sensible in coastal areas to develop its practical applications in the remediation of Cd and As contaminated soil.

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