Paired soil and rice arsenic and cadmium from northeastern U.S. rice farms

Ruifang Hu | William A. Teasley | Angelia L. Seyfferth

Abstract
Arsenic (As) and cadmium (Cd) are two toxic elements found in rice (Oryza sativa L.) grain that pose threats to human health. While attention has focused on major rice-growing regions, rice has been grown on small-scale farms in the northeastern United States since approximately 2005, but the grain As and Cd levels were not previously reported. Unpolished grain from these farms have mean As of 0.12 mg kg\(^{-1}\) and mean Cd of 0.007 mg kg\(^{-1}\), more than 40% lower than previously reported in U.S. rice. Therefore, rice from these farms poses low risk for consumers.

1 | INTRODUCTION

Arsenic (As) and cadmium (Cd) are trace elements of concern in rice (Oryza sativa L.). Both are toxic to humans (ATSDR, 2002a, 2002b; Nordberg, 2009) and can decrease rice yield (Abedin, Cresser, Meharg, Feldmann, & Cotter-Howells, 2002; Clemens, Aarts, Thomine, & Verbruggen, 2013; Williams et al., 2007b). The availabilities of As and Cd in soil are affected differently by water management (Arao, Kawasaki, Baba, Mori, & Matsumoto, 2009), and the uptake of As can be decreased by increasing soil silicon (Si) (Fleck, Mattusch, & Schenk, 2013; Seyfferth & Fendorf, 2012). To protect human health, it is imperative to evaluate As and Cd levels in rice grown under different soil conditions. Moreover, grain As speciation is critical to assess food-chain risk, as inorganic As (iAs) species are more toxic than some organic As species (Hughes, 2002) and only iAs is used in rice regulations.

While grain As levels from major U.S. farming regions in the Mid-South and California have been reported, little is known about levels in rice from other areas. A network of small-scale rice farms was established in the northeastern United States that uses organic and/or low-input practices, some used for specialized rice products and others for personal use, but there have been no published reports of grain elemental analysis from these farms. Surveys in the United States (Heitkemper, Kubachka, Halpin, Allen, & Shockey, 2009; Williams et al., 2007; Wolnik et al., 1985; Zavala & Duxbury, 2008) took place before most of the northeastern rice farms were established, and these surveys do not typically link grain concentrations with soil-chemical parameters.

We collected paired rice and soil samples from small-scale rice farms in the northeastern United States and evaluated the plant elemental concentrations and correlations with soil-chemical parameters. We hypothesized that this grain would have lower As and Cd concentrations than grain produced in the major rice-growing regions of the United States due to holistic crop residue management and cooler soil temperatures. We found that grain As and Cd from northeastern farms are among the lowest reported in the United States.
2 | MATERIALS AND METHODS

2.1 | Sample preparation

We obtained nine rice grain samples from six small-scale rice farms in the northeastern United States (Table 1). Grain samples were paired with husk, straw, and soil samples from the respective farm for all but one sample. During soil sampling, three to five locations across each field were sampled to a depth of approximately 25 cm and composited into one per farm. Soils were air-dried and sieved (<2-mm). Shoots of three to five plants per cultivar were randomly collected and straw was cut at least 5 cm above the soil surface. For NE3, no straw was obtained. Rice straw was oven-dried at 65 °C. Air-dried rough rice was dehusked to separate unpolished rice from husk. Each plant fraction was ground with stainless-steel grinders.

2.2 | Plant analysis

Established protocols were used for digestion and analysis of As and Cd (Seyfferth et al., 2016; Teasley, Limmer, & Seyfferth, 2017). Samples were digested in concentrated HNO₃ using closed-vessel microwave digestion (MARS 6, CEM Corp.). Undissolved Si-gel residue in husk and straw was separated via centrifugation, digested in 2 M NaOH, and analyzed colorimetrically for Si (Derry, Kurtz, Ziegler, & Chadwick, 2005; Kraska & Breitenbeck, 2010). The acid fraction was diluted to achieve 4% (v/v) acid and analyzed for total As and Cd concentrations with inductively coupled plasma mass spectrometry (ICP–MS; Thermo iCap-TQ). Standard checks, blanks, and duplicates, NIST1568a rice flour, and WEPAL IPE 188 oil palm reference materials were used for quality control (Seyfferth & Fendorf, 2012; Seyfferth, McCurdy, Schaefer, & Fendorf, 2014). Duplicate As concentration results for all plant samples had 11.0 ± 5.0% error, NIST1568a analyses had 107 and 91% recovery for As and Cd respectively, and WEPAL IPE 188 oil palm had 81.0 ± 5.8% of the reported “indicative” Si value.

Unpolished grain samples were extracted in 2% (v/v) HNO₃ at 100 °C, diluted to 1% HNO₃ and filtered (0.22 μm) for As speciation analysis (Maher, Foster, Krikowa, Donner, & Lombi, 2013). Separation was achieved using a Hamilton PRP-x100 column with gradient elution using 50 mM ammonium carbonate and 3% (v/v) methanol (Jackson, 2015), and quantification with ICP–MS (Thermo, iCap-TQ). Blanks and a duplicate certified rice NIST1568b analyses were included for quality control. Recovery in NIST1568b was iAs = 118.0(± 2.5)%; dimethylarsinic acid (DMA) = 105.7(± 0.4)%; and monomethylarsonic acid = 142.9(± 2.3)%.

2.3 | Soil analysis

The <2-mm soils were subjected to 1 h of 0.5 M acetic acid extraction (Korndörfer, Coelho, Snyder, & Mizutani, 1999), soil pH (1:10), HNO₃-digestible As and Cd (EPA 3051A) and soil organic matter (SOM) by loss on ignition. Further sieving to 150-μm was used for acid ammonium oxalate, citrate bicarbonate dithionite, and diethylene-triaminepentaacetic acid (DTPA) extractions to estimate As associated with poorly crystalline iron (Fe) oxides, total free Fe oxides, and plant-available Cd, respectively (Amacher, 1996; Loeppert & Inskeep, 1996). The DTPA extractions and total soil digestions were analyzed with ICP–MS (Agilent 7500); NIST2711 soil had 82% Cd and 82% As recovery. Other extractions were analyzed with inductively coupled plasma optical emission (Thermo DuoView).

3 | RESULTS

3.1 | Plant As, Cd, and Si

Plant As concentrations increased in the order unpolished grain < husk < straw. Grain total As ranged from 0.06 to 0.23 mg kg⁻¹ with a median of 0.11 mg kg⁻¹ (Table 1). The farms reported as flooded for most of the production period (Table 2) tended to have the highest grain As. Grain As was composed of mostly iAs, but grain DMA increased as grain total As increased (Figure 1a). Husk As ranged from 0.08 to 0.38 mg kg⁻¹, and straw As ranged from 0.34 to 1.48 mg kg⁻¹ with one outlier of 11 mg kg⁻¹, which may have been due to soil As contamination of this sample. Straw Si concentrations were low and ranged from 0.7 to 4.1% with median concentration of 2.1%. Husk Si concentrations were higher and ranged from 2.0 to 6.7% with a median concentration of 3.9% (Table 1).

Plant Cd concentrations also increased in the order grain < husk < straw. Grain Cd ranged from
 FIG URE 1  Relations between (a) grain dimethylarsinic acid (DMA) and grain total As, (b) total grain As and soil As, (c) total grain As and the ratio of soil As to acetic acid–extractable Si, and (d) total grain As and ratio of soil As to citrate bicarbonate dithionite (CBD)–extractable Fe. All analyzed with Robust Linear Regression in JMP Pro.

0.001 to 0.032 mg kg\(^{-1}\) with a median of 0.002 mg kg\(^{-1}\), and the highest concentration was found in fields reported as grown under dryland conditions (Table 2). Husk Cd ranged from 0.005 to 0.095 mg kg\(^{-1}\), and straw Cd ranged from 0.009 to 0.367 mg kg\(^{-1}\) (Table 1).

3.2  |  Soil chemical parameters

Soil total As and Cd concentrations were relatively low and ranged from 0.91 to 6.34 mg kg\(^{-1}\) and 0.083 to 0.417 mg kg\(^{-1}\), respectively. Soil organic matter contents were relatively high and ranged from 6 to 35%; higher SOM usually correlated with higher cation exchange capacity (CEC) (Table 2).

3.3  |  Plant–soil relationships

Positive linear relationships were found between grain As and soil As (Figure 1b), the ratio of soil As and plant-available Si (Figure 1c), and the ratio of soil As and citrate bicarbonate dithionite–extractable Fe (Figure 1d). No significant relationships were found between plant Cd and any tested soil parameter.

4  |  DISCUSSION

Results support our hypothesis that rice from small-scale rice farms in the northeastern United States contain lower grain As and Cd concentrations than rice from conventional farms in major U.S. rice-growing regions. We observed lower mean grain Cd (0.007 mg kg\(^{-1}\)) than the 0.012 mg kg\(^{-1}\) previously reported from major rice-growing regions (Wolnik et al., 1985); this may be because the high SOM and CEC (Table 2) played a role in Cd retention (Haghiri, 1974), even though no significant relationship was found between Cd and SOM or CEC across farms (Supplemental Table S1). Soil Cd fell within the northeastern soil Cd range (Page, Chang, & El-Amamy, 1987) except NE1 from Delaware and NE2.
**TABLE 1  Rice As, Cd, and Si concentrations and grain As speciation from small-scale rice farms in the northeastern United States**

| Sample ID | State | Variety    | Grain total As | Grain iAs | Grain DMA | Grain MMA | Husk As | Straw As | Grain total Si | Husk Si | Straw Si |
|-----------|-------|------------|----------------|-----------|-----------|-----------|---------|---------|----------------|---------|---------|
| NE1A      | DE    | long grain | 0.107          | 0.114     | 0.025     | 0.009     | 0.138   | 0.86    | 6.7            | 2.8     | 0.006   |
| NE1B      | DE    | short grain| 0.135          | 0.112     | 0.025     | 0.004     | 0.157   | 1.48    | 6.3            | 3.7     | 0.007   |
| NE2       | NY    | carnaroli  | 0.229          | 0.188     | 0.040     | 0.011     | 0.383   | 11.0*   | 5.4            | 4.1     | 0.002   |
| NE3       | VT    | Hayakuki   | 0.157          | 0.077     | 0.051     | 0.006     | 0.218   | ns      | 2.0            | 0.7     | 0.002   |
| NE4A      | VT    | Matsumae   | 0.115          | 0.081     | 0.017     | 0.004     | 0.086   | 0.456  | 2.0            | 0.7     | 0.002   |
| NE4B      | VT    | Tannemochi | 0.057          | 0.078     | 0.015     | 0.006     | 0.080   | 0.341  | 2.4            | 1.5     | 0.002   |
| NE5       | MA    | Duborskian | 0.072          | 0.078     | 0.013     | 0.004     | 0.092   | 0.403  | 5.2            | 2.1     | 0.009   |
| NE6       | MA    | Duborskian | 0.095          | 0.089     | 0.009     | 0.004     | 0.153   | 0.389  | 3.9            | 2.1     | 0.032   |

**Note.** DMA, dimethylarsinic acid; iAs, inorganic arsenic; MMA, monomethylarsonic acid.

*Outlier potentially due to soil contamination of the sample.*

**TABLE 2  Soil chemical properties of sampled ecological rice farms in the northeastern USA**

| Sample ID | State | Reported water management | pH | SOM LOI | CEC | Nitric acid digestible As | Acetic acid extractable As | Nitric acid digestible Cd | DTPA - extractable Cd | Acetic acid extractable Si | CBD extractable Fe | AAO extractable Fe |
|-----------|-------|---------------------------|----|---------|-----|--------------------------|---------------------------|--------------------------|------------------------|---------------------------|-------------------|-----------------|
| NE1       | DE    | Flooded                   | 7.3| 13.9    | 34.1| 4.32                     | 1.63                      | 0.364                    | 0.161                  | 31.16                     | 9,353             | 3,768           |
| NE2       | NY    | Flooded                   | 7.3| 34.8    | 48.0| 6.34                     | 1.59                      | 0.417                    | ns                     | 18.13                     | 7,195             | 5,528           |
| NE3       | VT    | Wet                       | 6.4| 5.9     | 7.2 | 3.35                     | 1.04                      | 0.132                    | ns                     | 14.81                     | 14,492            | 5,312           |
| NE4       | VT    | Wet                       | 6.4| 8.0     | 10.7| 1.77                     | 1.01                      | 0.134                    | 0.114                  | 11.33                     | 17,981            | 7,258           |
| NE5       | MA    | Dry land                  | 5.9| 7.1     | 4.7 | 0.91                     | 1.02                      | 0.083                    | 0.077                  | 17.2                      | 15,365            | 6,405           |
| NE6       | MA    | Dry land                  | 5.9| 7.1     | 4.7 | 0.91                     | 1.02                      | 0.083                    | 0.077                  | 17.2                      | 15,365            | 6,405           |

**Note.** AAO, acid ammonium oxalate; CBD, citrate bicarbonate dithionite; CEC, cation exchange capacity; DTPA, diethylenetriaminepentaacetic acid; LOI, loss on ignition; SOM, soil organic matter.

*ns = not sampled.*
from New York City, which were higher but still within the range of U.S. agriculture soil Cd (Holmgren, Meyer, Chaney, & Daniels, 1993). The highest grain As and Cd were found in flooded and dryland fields, respectively, reflecting the opposite soil redox impacts on soil As and Cd availabilities (Arao et al., 2009). The mean grain As concentration (0.12±0.03 mg kg$^{-1}$) was 43% lower than previously reported in unpolished grain from major U.S. rice-growing regions (0.21±0.19 mg kg$^{-1}$) (Heitikemper et al., 2009). Moreover, our unpolished grain As concentrations were 29 to ~60% lower than the polished grain from the U.S. market survey by Williams, Raab, Feldmann, and Meharg (2007a). The grain iAs concentration in our unpolished grain samples lies at the lower end of iAs concentrations in polished rice previously reported in the United States (0.07-0.17 mg kg$^{-1}$) (Chen et al., 2016; Jitaru et al., 2016; Liang et al., 2010; Ma, Wang, Jia, & Yang, 2016; Rahman, Rahman, Reichman, Lim, & Naidu, 2014; Zavala, Gerads, Gürleyük, & Duxbury, 2008) and other countries (0.02-0.47 mg kg$^{-1}$) (Batista, Souza, De Souza, & Barbosa, 2011). Considering that our samples were unpolished “brown” rice, the actual As risk of rice from the sampled northeastern farms is even lower because polishing removes the As-rich bran (Sun et al., 2008). The low grain As in northeastern rice may be due to relatively low (<6 mg kg$^{-1}$) soil As concentrations (Smith et al., 2005). In addition, the relatively cooler climate in the northeastern United States could contribute to less As uptake because of lower As mobility in cooler soils (Muehe, Wang, Kerl, Planer-Friedrich, & Fendorf, 2019; Neumann, Seyfferth, Teshera-Levy, & Ellingson, 2017); temperature could also have played a role in limiting Cd uptake.

The positive correlation between grain and soil As (Figure 1B) and between grain DMA and grain total As (Figure 1A) were expected at the low soil As levels in the sampled farms (Lu et al., 2009; Zavala et al., 2008). The positive linear relationship between grain As and the ratio of soil total As and plant-available Si (Figure 1C) reflects the competition between soil As and Si for uptake by rice (Ma et al., 2008), where higher soil As relative to plant-available Si results in more As uptake. The positive linear relationship between grain As and the ratio of soil total As and total free Fe (Figure 1D) reflects the As retention function of free iron oxide in soil (Takahashi et al., 2004).

## 5 | CONCLUSIONS

Our data show that small-scale rice farms in the northeastern United States have low iAs and Cd in grain. While the few rice farms that exist in the area limited our statistical power, our data suggest that rice produced in small-scale rice farms in the northeastern United States poses low human health risk.

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## AUTHOR CONTRIBUTIONS

R. Hu: Investigation, formal analysis, writing-original draft. W.A. Teasley: Formal analysis, writing-reviewing and editing. A.L. Seyfferth: Conceptualization, project administration, supervision, writing-original draft, writing-reviewing & editing.

## CONFLICT OF INTEREST

The authors report no conflicts of interest.

## ORCID

Angelia L. Seyfferth [https://orcid.org/0000-0003-3589-6815](https://orcid.org/0000-0003-3589-6815)

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