

Mercury in Biomass Feedstock and Combustion Residuals

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Abstract An exploratory survey of the mercury content of some common California biomass feedstocks shows that the concentrations are well below EPA toxicity levels with representative feedstock concentrations of 20 ppb for rice straw, 28 ppb for wheat straw, and 32 ppb for whole-tree wood chips. The temporal variability for rice straw (17–20 ppb) is near the analytical uncertainty (~2 ppb). Saline-irrigated feedstock does not contain greatly higher mercury contents (17–38 ppb) compared to normally irrigated feedstock. Water leaching has likewise no detectable effects on mercury mobility, despite an up to 30% increase in the Hg concentrations attributable to mass losses during leaching. Combustion at temperatures of at least 575°C results in complete volatilization of mercury leaving solid ash and slag residuals with mercury contents at or near the lower limit of detection (5 ppb). The mercury strongly concentrated in fly ash can reach concentrations up

to 40 times (<1,166 ppb) the corresponding fuel concentrations.

Keywords Mercury · Biomass · Fuel feedstock · Combustion · Leaching · Ash · Slag · Fly ash

1 Introduction

Mercury in biomass and its emission during controlled or accidental combustion are of considerable interest to health risk assessment and environmental regulation and protection efforts (EPA 1997, 1998a). The releases of mercury from biomass during wildfires and from coal combustion for energy conversion constitute the main sources for atmospheric mercury emissions (Kouvo 2002; Friedli et al. 2001, 2003; Ebinghaus et al. 2007; Obrist et al. 2008). This atmospheric mercury either in particulate or gaseous forms may accumulate in lakes and watersheds (Coolbaugh et al. 2002; Slotton et al. 2004; Sanders et al. 2008) and eventually in the biosphere where highly toxic compounds (methylmercury) can build up in animal and human tissues (Slotton et al. 2004; Brookens et al. 2008), causing long-term neurological and developmental damage (NRC 2000). In addition to coal combustion, major contributors of mercury to the environment are from municipal waste incinerators (Conaway et al. 2005; Park et al. 2008; Obrist et al. 2008; Hutson 2008). In California and a number of

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other regions, additional emission sources are naturally occurring volcanic and hydrothermal activity as well as mining-related deposits (Coolbaugh et al. 2002; Slotton et al. 2004).

Combustion facilities utilizing various biomass fuels constitute a potential source of mercury to the atmosphere. Here we present an exploratory evaluation of the mercury concentrations in selected biomass feedstocks and their solid combustion products. The results allow a preliminary evaluation of the potential environmental release of mercury from large-scale biofuel consumption and provide regulatory baseline for the utilization of waste products from combustion. We show that mercury concentrations in common California biomass feedstock is well below toxicity levels. Ash and slag from combustion (bottom ash) likewise contain low mercury concentrations near or below detection limits (~5 ppb) and thus provide no particular concern for waste utilization. Mercury is, however, strongly enriched in fly ash collected from commercial-type boilers utilizing clean feedstock, even without municipal and other enriched source feed contamination. Such fly ash waste-types may pose environmental and health concern and thus require careful removal and storage.

2 Fuel and Ash Selection and Preparation

Three principal feedstocks were selected for the study (Table 1): (1) mixed conifer, whole-tree wood chips with an estimated 20% bark content from Shasta County, California; (2) rice straw (M202 medium grain Japonica variety) from Colusa County, California; and (3) wheat straw from Yolo County, California (Thy et al. 2006). These fuels (respectively prefixed ‘W,’ ‘R,’ or ‘Wh’ in Table 1) were air-dried to 7–10% moisture contents (Table 1) in ambient air for a week and then milled to a maximum 3 mm (1/8”) grain size. Ashes and slag were prepared at temperatures from 525°C to 1,525°C or within the melting intervals (Thy et al. 2006).

In addition to the primary feedstock listed above, leached rice straw (LR) was prepared from rice straw following solid–liquid extraction in water using the procedures of Jenkins et al. (1996). Rice straw from several other sources were also included for comparison (HEL, EER; Table 1) together with a selection of saline-irrigated plants (Blunk et al. 2005). These latter include salt-tolerant trees (*athel* or tamarisk (A) and

eucalyptus (E) species) from agroforestry test plots in the San Joaquin Valley of California grown under saline irrigation to investigate soil remediation and ground-water management alternatives. Perennial grasses *Agropyrum elongata* or Jose tall wheat grass (JTW) and *Leymus triticoides* or creeping wildrye (CWR) from similar saline irrigation experiments in the same region were also investigated. Leaching of these salt-tolerant crops was done as for the rice straw. The ash contents for the various feedstock were determined at 525–600°C and are summarized in Table 1.

Two sets of experimental fly ash were also included in the study. The first set is from a cyclone catch of laboratory scale fluidized bed combustion experiments using wood-dominated blends of the wood and rice straw fuels discussed above and prefixed ‘CycFBC’ in Table 1 (Thy et al. 2004). The other set is from controlled fuel experiments conducted in a commercial biomass power boiler (Jenkins et al. 1999) using blends of leached rice straw and urban wood and agricultural residues (prefixed ‘Wood’ or ‘Straw’ in Table 1).

3 Analytical Methods

The total mercury (Hg) concentrations were determined using atomic absorption methods (LECO AMA254 Mercury Analyzer) that conform to requirements in EPA Method 7343 (EPA 1998b, Mercury in Solids and Solutions) and ASTM D-6722 2006, (Total Mercury in Coal and Combustion Residues). The analytical method requires no pre-treatment or concentration of elemental constituents and thus allows feedstock as well as their solid combustion products to be analyzed. The basic analytical design involves combustion in oxygen in a decomposition tube heated to ~750°C, scrubbing of the evolved gas, amalgamation over gold plated ceramic, and release to ~900°C of mercury from the amalgamator to the detector. The atomic absorption detector uses a mercury-specific lamp and a UV diode detector. All analyses in this study were based on area integration of the resolved peak from the detector and are reported on an air-dried fuel basis as parts per billion on a weight basis (ppb).

Calibration was done using NIST 1633b fly ash (certified value 143±2 ppb; May and Watters 2004) and NIST 1645 river sediment (certified value 1.1 ppm; Uriano 1982). The calibration was done using a third

Table 1 Mercury concentrations (ppb) of California biomass feedstock and their combustion products

ID	Description	Average and standard deviation	Hg ppb	Moisture %	Ash % wet basis	L-O-I (950°C) wt.%	Delta L-O-I wt.%	Corrected ash wt.%	Retained Hg ppb, fuel basis	Hg loss from raw fuel (%)
R	Rice straw raw		20	7.5	20.4					
R-0	Rice straw ash (525°C)		14			10.94	0.0	20.4	2.9	94.6
R-9	Rice straw ash (572°C)		<5			7.95	0.6	20.3	0.8	98.5
R-1	Rice straw ash (621°C)		<5			7.18	1.2	20.2	0.9	98.2
R-8	Rice straw ash (670°C)		5			7.18	1.8	20.0	3.0	94.3
R-2	Rice straw ash (718°C)		6			6.59	2.4	19.9	1.2	97.7
R-7	Rice straw ash (767°C)		6			5.85	3.0	19.8	1.2	97.8
R-3	Rice straw ash (816°C)		6			5.35	3.5	19.7	2.0	96.3
R-6	Rice straw ash (864°C)		5			4.82	4.1	19.6	1.0	98.2
R-4	Rice straw ash (913°C)		5			4.65	4.7	19.4	1.0	98.2
R-5	Rice straw ash (962°C)		<5			3.98	5.3	19.3	0.8	98.4
R-10	Rice straw ash (1,011°C)		<5			2.85	5.9	19.2	0.9	98.3
R-11	Rice straw ash (1,125°C)		6			0.71	7.3	18.9	1.1	97.9
R-12	Rice straw ash (1,225°C)		<5			0.28	8.5	18.7	0.8	98.5
R-13	Rice straw ash (1,325°C)		7			0.59	9.1	18.5	1.3	97.6
Rice straw ash average		6±3 ppb (N=13)								
Wh	Wheat straw raw		28	7.8	9					
Wh-0	Wheat straw ash (525°C)		17			14.91	0.0	9.0	1.5	97.1
Wh-9	Wheat straw ash (572°C)		6			7.26	5.2	8.5	0.5	99.0
Wh-1	Wheat straw ash (621°C)		<5			6.94	8.6	8.2	0.4	99.3
Wh-8	Wheat straw ash (670°C)		7			4.17	10.3	8.1	0.6	98.9
Wh-2	Wheat straw ash (718°C)		9			3.32	10.7	8.0	0.7	98.6
Wh-7	Wheat straw ash (767°C)		6			3.51	10.4	8.1	0.5	99.1
Wh-3	Wheat straw ash (816°C)		<5			4.92	9.7	8.1	0.4	99.3
Wh-6	Wheat straw ash (864°C)		<5			5.25	9.0	8.2	0.3	99.3
Wh-4	Wheat straw ash (913°C)		<5			5.27	8.8	8.2	0.3	99.4
Wh-5	Wheat straw ash (962°C)		5			4.75	9.6	8.1	0.4	99.2
Wheat straw ash average		6±2 ppb (N=9)								
W	Wood raw		32	10.4	1.1					
W-0	Wood ash (525°C)		10			27.59	0.0	1.1	0.11	99.8
W-2	Wood ash (621°C)		7			26.73	4.1	1.1	0.07	99.9

Table 1 (continued)

ID	Description	Average and standard deviation	Hg ppb	Moisture %	Ash % wet basis	L-O-I (950°C) wt.%	Delta L-O-I wt.%	Corrected ash wt.%	Retained Hg ppb, fuel basis	Hg loss from raw fuel (%)
W-3	Wood ash (718°C)		6			22.94	7.8	1.0	0.06	99.9
W-4	Wood ash (816°C)		<5			13.94	11.0	1.0	0.03	99.9
W-5	Wood ash (913°C)		7			13.33	13.8	0.9	0.07	99.9
W-1	Wood ash (1,011°C)		7			13.67	16.1	0.9	0.06	99.9
W-6	Wood ash (1,125°C)		7			11.42	18.3	0.9	0.06	99.9
W-9	Wood ash (1,225°C)		6			8.74	19.7	0.9	0.05	99.9
W-7	Wood ash (1,325°C)		<5			8.24	20.5	0.9	0.03	99.9
W-10	Wood ash (1,425°C)		<5			9.33	20.9	0.9	0.04	99.9
W-8	Wood ash (1,525°C)		<5			6.39	20.9	0.9	0.04	99.9
Wood ash average										
		6±1 ppb (N=10)								
Wb	Wood raw (high bark)		11	8.6	2.7					
Wb-0	Wood ash (high bark, 525°C)		11							
HELA	Rice straw raw (Jenkins et al. 2003)		19		14.9 [#]					
HELA-0	Rice straw ash (Jenkins et al. 2003)		8							
EER	Rice straw raw (Meister et al. 2005)		17		19.5 [#]					
EER-0	Rice straw ash (Meister et al. 2005)		11							
CFM-0	California feed lot cattle manure ash (575°C)		14							
Wood17.40	Power plant fly ash (wood stack baseline, 8/10/98, 17:40) ^a		1,166							
Wood15.52	Power plant fly ash (wood stack baseline, 8/10/98, 15:52) ^a		870							
Straw12.35	Power plant fly ash (straw blend, 8/11/98, 12:35) ^a		479							
Straw14.30	Power plant fly ash (straw blend, 8/11/98, 14:30) ^a		673							
FlyashMarch02	Commercial boiler fly ash collected March 11, 2000		331							
CycFBC6	Cyclone ash March 26, 2003 (FBC-6, leached rice blend) ^b		48							

CycFBC5	Cyclone ash March 15, 2003 (FBC-5, wood) ^b	75		
CycFBC3	Cyclone ash March 2, 2003 (FBC-3, rice blend) ^b	153		
LR	Leached rice straw raw	13	10.7	18.8
LR-0	Leached rice straw ash (525°C)	8		
A	Athel raw (600°C) ^c	23	8.4	4.9
A-0	Athel ash (575°C) ^c	7		
LA	Leached athel raw ^c	25		
LA-0	Leached athel ash (575°C) ^c	<5		
E	Eucalyptus raw (600°C) ^c	17	7.8	1.8
E-0	Eucalyptus ash (575°C) ^c	7		
LE	Leached eucalyptus raw ^c	24		
LE-0	Leached eucalyptus ash (575°C) ^c	8		
CWR	Creeping wild rye raw (600°C) ^c	38	9.4	6.3
CWR-0	Creeping wild rye ash (575°C) ^c	7		
LCWR	Leached creeping wild rye raw ^c	50		
LCWR-0	Leached creeping wild rye ash (575°C) ^c	7		
JTWG	Jose tall wheat grass raw (600°C) ^c	31	6.4	8.2
JTWG-0	Jose tall wheat grass ash (575°C) ^c	6		
LJTWG-0	Leached Jose tall wheat grass ash (575°C) ^c	7		
NBS 1633b	Fly ash standard average			150±8 ppb (N=18)

Hg (ppb) as determined in this study. Ash wt. % on air-dried basis as determined or estimated from sources listed. Temperature in parenthesis is the ashing temperature. Retained Hg (ppb) on a fuel basis is the analyzed Hg content taken into account the corrected ash content. Hg loss in % of the raw fuel content calculated from the Hg content determined in the raw fuel and the calculated content retained on a raw fuel basis.

L-O-I (wt.%) is the loss-on-ignition determined by weight loss at 950°C, *delta L-O-I* is the smoothed changes of loss-on-ignition as a function of temperature, *corrected ash content* (wt. %) corrected for the temperature effect from previous column, *N* is the total numbers of analyses used for calculating average and standard deviation

^aJenkins et al. (1999)

^bThy et al. (2004)

^cBlunk et al. (2005) and Chao Wei Yu personal communication (2008)

[#] on a dry basis

order regression equation with correction for blank and drift when required. Repeated analysis of NIST 1633b ($N=18$) as an unknown yielded 150 ± 8 ppb Hg that closely conforms to listed values (143–155 ppb; Govindaraju 1994; Feng et al. 2004). The standards were not oven dried as recommended by Health Canada (2006). The lower limit of detection as measured on blanks is ~ 0.35 ng total mercury or equivalent to 3–5 ppb Hg for typical sample masses.

4 Results

The concentrations of Hg in the analyzed biomass feedstock, ashes, and other combustion products are listed in Table 1. The analytical results of the unknown samples are mostly within the range defined by the calibration standards ($\leq 1,100$ ppb). Multiple analyses of NIST fly ash standards indicate a relative standard deviation of 5.3% for the mercury determinations.

The raw feedstock contains low Hg concentrations with 20 ppb for rice straw, 28 ppb for wheat straw, and 32 ppb for whole-tree wood chips. The ashes of these three principal feedstocks all show low average mercury concentrations (6 ppb), just above detection limit of Hg without any apparent dependence on ashing or slagging temperature (to 1,525°C). The only dependency on ashing temperature observed is that the Hg concentrations for the initial 525°C ash for all straw feedstock are systematically higher than for the ashes produced at higher temperatures. This suggests that ashing temperatures below $\sim 575^\circ\text{C}$ may be too low to completely remove volatile Hg from these fuels.

To allow an evaluation of the Hg losses, the analytical results were evaluated on a fuel basis at calculated concentration levels well below the analytical capability (Table 1). In addition to the direct loss of Hg, the analyzed concentrations of Hg also reflect the removal of other volatile components. These are most notably the organic components (C-H-N-S-O), but also other inorganic components such as the halogens and alkali metals (Cl, K, and Na; Thy et al. 2006, 2008). The elemental losses are potentially both composition and temperature dependent. Mercury concentrations depend on the total composition (total sum effect). If the mass or temperature changes (as during dehydration, combustion, decomposition, crystallization, and/or melting), the relative concentration of Hg will also change. In order to evaluate the actual loss (or gain) in

Hg, the measured concentrations are corrected for the effects of mass losses at the actual ashing temperature using the moisture and ash contents of the raw fuels. On a wet basis, ash contents are 20.4% for rice straw, 9.0% for wheat straw, and 1.1% for wood, all at 525°C (Thy et al. 2006). The stepwise additional losses above this baseline ashing temperature are approximated from the loss-on-ignition determined on ash or slag by Thy et al. (2006) (Table 1), yielding Hg concentration on an original fuel basis (Table 1).

From these temperature corrected ash contents, the retained percentage of Hg in the ash or slag can be compared to the initial concentrations of Hg in the corresponding three raw fuels. The results of these calculations are summarized in Table 1 as well as in Fig. 1. There is little if any correlation between ashing and slagging temperatures and residual Hg contents, save for a significantly higher content for the low temperature (525°C) straw ashes, as noted previously.

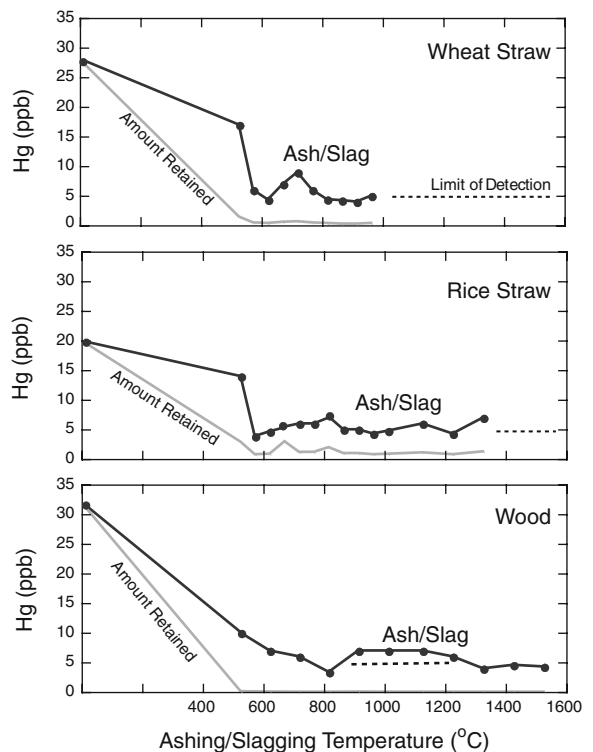


Fig. 1 Mercury content (Hg ppb) as a function of ashing and slagging temperature ($^\circ\text{C}$) for each of the three main types of biomass feedstock (wood, rice straw, and wheat straw). The analyzed concentrations are shown as *solid points*. The raw fuel compositions are plotted at a temperature of 25°C . The estimated retained amount relative to the raw fuel content is indicated by the *gray curve* (see text for calculations)

The Hg concentrations in the ash are only moderately dependent on fuel (or ash content) as reflected in the strongest decrease for the wood ash (1% total fuel ash content), followed by wheat straw (9% ash), and a less marked decrease for rice straw (20% ash). The amount of Hg lost from the ashes or slag relative to the original feedstock is 98% for rice straw, 99% for wheat straw, and ~100% for wood for temperatures above ~600°C. These losses correspond to relative enrichment factors calculated following Meij and te Winkel (2007) as of 0.06, 0.02, and 0.002, respectively, and classify Hg as a highly volatile Class III element (together with Cl, F, and Se).

There is little variability in the Hg content of rice straw (Table 1). We analyzed three different rice fuels acquired over a period of several years from three different locations in the Central Valley of California and were only able to detect differences (17–20 ppb) near the analytical standard deviation between individual fuels (~2 ppb). The geographic and temporal variability of rice and other feedstock, however, requires a systematic study that was not intended by the present survey. The Hg contents further appear to be unaffected by leaching (Table 1). Of the herbaceous materials (rice, creeping wildrye) and woods (athel, eucalyptus) for which matching data sets are available, the effect of leaching appears dominantly to be a marked enrichment (10–30%) in Hg contents that reasonably correspond to the predicted effect of the mass losses during leaching (9–23% on a dry basis). The exception is rice straw that suggests a nearly 50% loss of the original Hg content during leaching, assuming a reasonable 30% mass loss.

Mercury is variably concentrated in the fly ashes analyzed in this study (Table 1). A study using a laboratory scale, fluidized bed combustor with cyclonic particle separation (cyclone ash) yields a modest two- to fivefold enrichment in the fly ash relative to the feedstock fuel blends. The highest Hg content was detected in fly ash collected from a commercial biomass boiler during an intake-controlled experiment. The four fly ashes contained from 479 to 1,166 ppb Hg (Table 1), with the highest concentrations detected in the fly ash collected using wood baseline fuels, and straw blended wood fuels resulting in the lower Hg concentrations. This suggests enrichment factors of well above 20–40 times compared to the known (Table 1) or estimated fuel compositions.

5 Discussion

Mercury is a toxic trace element in many plants and occurs in concentrations often well below 100 ppb dominantly to have been absorbed by the root systems from water solutions and the bulk soil matrix. Mercury is believed to occur dominantly as in amino acids or organometal compounds in plant tissues (Kabata-Pendias and Pendias 1992; Arienzo 2005). The mercury concentrations of biomass are thus expected to be dependent on local and regional soil and bedrock as well as irrigation and anthropogenic sources.

A regional US survey of the potential emission from wood fuels by Mentz et al. (2005) concluded that the average mercury content of stemwood was 2.3 ppb (range 1–4 ppb) and similarly found that bark contained much elevated concentrations of 12.5 ppb (range 1–37 ppb). The somehow higher concentrations of 11–32 ppb found in the present study in wood fuels may be attributed to the volcanic soil and hydrothermally active environment (Coolbaugh et al. 2002) of the slopes of the Shasta volcano on which the wood material was collected, plus perhaps atmospheric deposition from global coal combustion (Sanders et al. 2008; Selin et al. 2008). Rather similar results of 17–23 ppb were obtained in this study from stemwood of salt-tolerant trees (athel and eucalyptus).

Information on mercury in herbaceous materials is limited (Kabata-Pendias and Pendias 1992). A study by Obrist et al. (2008) gave an average concentration of mercury in rice straw as 9 ppb that is low compared to the 17–28 ppb obtained in this study (Table 1). Other herbaceous materials analyzed in this study included a selection of saline-irrigated perennials that indicated only slightly higher concentrations of 31–38 ppb (wild rye, wheat grass). The three rice straw feedstock (R, HELA, EER; Table 1) that were collected within a narrow geographical area in the northern Central Valley of California over a span of 5 years, between 1995 and 2001, revealed no significant variation in Hg content. This limited variation may be related to limited local soil variation and consistent irrigation methods over the years of harvest. Further work specifically designed to address the temporal and geographical factors are clearly needed, but were beyond the goals of the present study.

There appear to be no previous studies on the effects of biomass feedstock leaching on mercury concentration. Our experiments suggest that mercury mobility

was unaffected for most feedstock by water leaching at ambient temperature, including vigorous agitation during extraction. This observation supports the common belief that mercury is mainly bonded in an organic structure of the plant tissue and to a lesser extent related to adsorption to plant surfaces and adhering particles.

Mercury is strongly released to the flue gas during combustion (Carpi 1997; Meij and te Winkel 2007; Park et al. 2008). The release that we observe is largely independent on firing temperature and amounts to nearly 100% from wood and decreases slightly to 98–99% from straw materials (Fig. 1). Very similar results were obtained by Friedli et al. (2001, 2003). The fate of vaporized mercury in the combustor stack is controlled by cooling-controlled heterogeneous reactions and chemical deposition on ash particles or boiler stack surfaces as oxides, sulfates, or halides (Schofield 2008). The result is a strong concentration of mercury in fly ash (e.g., Querol et al. 1995).

The Toxicity Characteristic Leaching Procedure published by the US Environmental Protection Agency in Method 1311 (EPA 2009) determines the mobility of both organic and inorganic compounds present in solid wastes. The toxicity analysis simulates landfill conditions and determines the concentrations of contaminants present in extracted leachate and determines if a waste meets the definition of toxicity and will require handling as a hazardous waste. For solid wastes (like fly ash and slag), the regulations allow the use of a total constituent analysis instead of the more involved extraction analysis. The result of a total constituent analysis is divided by 20 to convert into an equivalent leachable concentration that thus carries the toxicity characteristics. The EPA mercury toxicity limit is 200 ppb that is well above maximum equivalent concentrations obtained for fly ashes in this study (<58 ppb).

6 Conclusion

An exploratory survey of the mercury content of common biomass feedstock shows that the concentrations are moderately low (11–38 ppb). The results do not support significant temporal and geographical variability and further do not imply anomalous concentrations from saline irrigation used in soil remediation experiments, although mercury is not thought to be a primary contaminant of the soils in the region. Likewise, water leaching has no detectable

effects on mercury mobility for the feedstocks tested. Combustion results in effectively complete removal of mercury to the flue gas and leaves solid ash and slag residuals with mercury content at or near the lower limit of detection. Mercury is strongly concentrated in fly ash and can reach concentrations up to 40 times the corresponding fuel concentrations. There are no indications in this study for mercury concentrations in feedstock or combustion residues that exceed federal toxicity limits, although elevated levels in fly ash suggest careful evaluation and monitoring for any higher levels of mercury or new feedstock sources.

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