

EVALUATION OF MERCURY ASSOCIATIONS IN TWO COALS OF DIFFERENT RANK USING PHYSICAL SEPARATION PROCEDURES

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Abstract

Mercury is considered to be a toxic metal of major concern, while coal combustion is recognized as one of the main anthropogenic sources of this element. Various processes have been investigated for mercury control in coal-fired utilities. However, until now, no cost-effective or efficient technology has been established for the removal of mercury emissions from coal combustion. For the development of such technologies a deep knowledge of the mode of occurrence and association of mercury compounds in different coals is necessary. Size fractionation, density separation, and oil agglomeration procedures were evaluated in this work not only to assess their efficiency as cleaning methods in mercury removal, but also to determine the associations of mercury with mineral/organic phases. Although the efficiency for mercury removal was found to be generally low, the results obtained provided valuable information for identifying the modes of occurrence of mercury in coal.

Keywords: mercury, coal combustion, physical separation

Introduction

The concentration of mercury in coal varies considerably, but in general [1] it is within the range of 0.02-1.0 $\mu\text{g g}^{-1}$. A recent work [2] concluded that $0.1 \pm 0.01 \mu\text{g g}^{-1}$ is the world-wide average mercury content in coal, and that when this value is expressed on an ash basis, the mercury average is 0.87 ± 0.08 and $0.62 \pm 0.06 \mu\text{g g}^{-1}$ in the ashes of bituminous and low rank coals respectively. Mercury, like most trace elements, may be present in coal in different modes of occurrence. Although the speciation of this element in a given coal is not always known, it is thought that mercury could occur in different coals as HgS, metallic mercury, as associated with pyrite and sphalerite, or organically bound to coaly matter [1].

Of the toxic air metals addressed in the 1990 Clean Air Act Amendments (CAAA), mercury is one of the elements of greatest concern because of its volatility, persistence and bioaccumulation as methyl mercury in the environment and its neurological health impacts. During coal combustion mercury compounds present in coal are mostly or entirely emitted to the environment in vapor phase. In spite of the low mercury content in coal, it has been recognized for years that coal-fired power plants in the United States are one of the largest sources of mercury emissions to the environment. As a consequence, on March 2005, the Environmental Protection Agency (EPA) issued the Clean Air Mercury Rule to permanently cap and reduce mercury emissions from coal-fired power plants. This rule makes the United States the first country in the world to regulate mercury emissions from utilities [3].

Mercury emission from coal combustion is also becoming a matter of growing interest in Europe. For the Development of an EU Mercury Strategy [4] the European Commission published in 2004 a consultation document inviting comments by stakeholders and other related persons in the field. This document identify large-scale coal combustion units as the largest emitters of mercury compounds into the air, and in January 2005, the Commission

adopted a mercury strategy that envisages a number of actions to protect the citizen's health and the environment [4]

Over the past few years, extensive efforts have been made to evaluate the quantity of mercury emitted from different sources and to reduce mercury pollution from coal [2, 5-9]. However at present, there is no universally accepted mercury control technology for coal-fired utilities, and the incorporation in coal power plants of the technologies already developed and in use in waste incinerators could enhance the cost of the power generating process considerably. One possible way of reducing the mercury content could be to clean coal as a previous step to its combustion. Coal cleaning methods are well established procedures, which are already extensively used for reducing the minerals in coal before combustion in order to increase its calorific value and to avoid some of the problems that mineral matter may typically cause (i.e. slagging, fouling, etc.). However, it is well known that the efficiency of mineral matter reduction from coal by means of these methods depends on the type of coal, making it difficult to predict the best method to use. Conventional physical coal cleaning techniques rely on differences in density or surface properties to separate the organic and inorganic components. Density separations and oil agglomeration are typical physical coal cleaning procedures. Density separation using dense medium baths is a method extensively used to reject the mineral impurities present in coal. This method is based on the differences in the specific gravity of the coal (organic or coaly matter) and its impurities (mineral matter). By mixing coal in dense medium baths of different densities it is possible to obtain, from a single coal, products with different organic/mineral matter proportions (density fractions). Separation of organic and mineral matter by oil agglomeration takes place when the solid particles of coal are added to a suspension water-oil. The oil preferably wets the organic particles (in practice the particles enriched in organic matter), which agglomerate. The mineral matter or more precisely, the particles enriched in mineral matter are rejected.

Density separation and oil agglomeration processes are effective in removing trace elements that are associated with major minerals, but ineffective for those that have high organic affinities [10-13]. In other words, the efficiency of these techniques for reducing mercury in coal depends on the speciation of this element. Some works have focused on trace elements or on mercury removal by using coal cleaning methods [6,10-17]. The mercury removal efficiency achieved so far depended on the type of coal and the cleaning procedure employed, the percentages of mercury rejection ranging from 1 to 99%. This uncertainty underlines the need for a better understanding of mercury behaviour in coal cleaning.

In the present work, a comparative study of the results obtained for mercury fractionation in coals of different rank using size fractionation and physical coal cleaning methods, was carried out with the aim of firstly contributing to a better understanding of the mode of occurrence of mercury in coals of different characteristics, and secondly exploring the possibility of reducing the mercury content in coal before combustion.

Experimental Section

Samples

Two coal samples of different rank from the Asturian (Spain) basin coalfield were used in this work: coal A (high rank coal) and B (bituminous coal). The proximate and ultimate analysis as well as the sulfur forms and ash compositions of these coals are given in Table 1. The samples were ground to sizes < 0.500 mm before the size and density separations and oil agglomeration were carried out.

Mercury analysis

The mercury content was determined, using an Automatic Mercury Analyzer (AMA). In order to assess the uncertainty of the results, the first part of the work consisted in evaluating the quality of the results obtained when Hg was analyzed with AMA equipment. Two MINTEK coal standards (SARM19 and SARM 20), and a NBS standard fly ash (1633 b), were used in this study as the reference materials.

Size fractioning

Size fractioning was carried out by wet screening. Nine fractions were separated from samples that had been ground to <0.500 mm. The size of the fractions was in the range of <0.020 to 0.500 mm. The ash and mercury contents of each fraction were determined and then calculated as cumulative ash and cumulative mercury contents.

Density separation

The density separations were carried out with 1 kg of coal sample, in dense medium baths. Eight and nine density fractions were obtained from coals A and B respectively, by consecutive immersion in dense medium baths of organic liquids (xylol, perchloroethylene and bromoform), of increasing density (from 1.40 to 2.60 g cm⁻³). This procedure, which is a conventional coal washability test (the float sink test) is described in ASTM Standard 4371-91. The ash and mercury contents of each fraction were determined and the cumulative ash and mercury contents were calculated.

Oil agglomeration

Oil agglomeration experiments were conducted in a commercial seven speed Waring blender, equipped with a 1000 ml glass vessel following a method previously developed [18-

19]. Crude (GIC) and refined (GIR) sunflower oils and crude (SOC) and refined (SOR) soybean oils were used as agglomerants. For each agglomeration test 400 ml of water and 16 g of coal was placed in the blender and mixed at 11 000 rev min⁻¹ for 5 min in order to disperse the particles. A specific amount of vegetable oil ranging from 10 to 40 wt% of oil was then added and mixed at the same speed for 60 s to produce agglomerates. The resultant agglomeration products were separated from the refuse, by filtration, water-washed, dried overnight at 50°C and finally analyzed for ash and mercury content. The results of the agglomeration were evaluated as in previous works [17] by the percentage of organic matter recovery (OMR), ash rejection (AR), and Efficiency Index (EI) as follows:

$$\%OMR = 100(wt_{\text{agglomerate}}/wt_{\text{coal}})[(100-ash_{\text{agglomerate}}) / (100-ash_{\text{coal}})]$$

$$\%AR = 100 [ash_{\text{coal}} - (ash_{\text{agglomerate}} * wt_{\text{agglomerate}} / wt_{\text{coal}})] / ash_{\text{coal}}$$

$$EI = \%OMR + \%AR - 100$$

ash_{coal} and ash_{agglomerate} being the percentages of ashes in the coal and agglomerate respectively

Results and discussion

The quality of the results obtained by the method used for mercury determination (AMA) was evaluated by analyzing three standard samples. The average of 10 determinations (n) of mercury per sample (x) was compared with the reference or certified values (ref /cert). Table 2 gives the results including the values of the standard deviation (SD), and relative standard deviations (%RSD). These analytical results may be considered as precise and statistically indistinguishable from the reference values, and they give an estimation of the uncertainty of the results to be discussed in this paper.

The mercury content of coals A and B is of the same order, 0.22 µg g⁻¹ and 0.26 µg g⁻¹ respectively (Table 1). In the nine size fractions separated from these coals (Figures 1-2), the

relationship between the distribution of the entire mineral content and the distribution of the mercury species according to sizes, is different in both coals. The ash content in coal A (corresponding to mineral matter), is maximum (40 % wt), in the fraction <0.020 mm, whereas in the other 8 fractions it is similar and close to 20% wt (Figure 1). In coal B, the ash content decreases slightly from the fractions of largest size to those of the smallest size, but finally it increases in the <0.020 mm fraction, where it reaches a maximum (Figure 2). A comparison of the mercury and ash contents in the different size fractions indicates that the mercury species in coal A do not follow the same tendency as the rest of the mineral matter. The range of mercury concentrations varies from 0.13 to 0.30 $\mu\text{g g}^{-1}$, and they increase as the sizes decrease, the concentrations being higher in the fractions of smallest particle size. As in the case of coal A the mercury content in the size fractions separated from coal B do not match the ash variation, and also increase in the fractions of a smaller size. In coal B the mercury concentrations in the size fractions range from 0.22 to 0.38 $\mu\text{g g}^{-1}$. In both coals the mercury compounds are, therefore, mainly concentrated in the minerals distributed in particles smaller than 0.100 mm, and the sizes of the mercury species in these fractions do not correlate with the sizes of the largest minerals, this tendency being more noticeable in the high rank coal A. As complementary information, Table 3 gives the values of the cumulative ash and mercury contents. From these data it can be inferred that a rejection of the <0.020 mm fraction, or even of the <0.045 mm fraction, would not produce any significant removal of mercury.

By means of density separations it is possible to assess the association of mercury species with organic or mineral matter in coal. If the mercury concentration in each one of the density fractions separated is compared to the ash content, significant differences may be observed between the two coals (Figures 3-4). In coal A, the mercury in the fractions increases in a similar way to that of ash. The mercury content is as low as 0.02 $\mu\text{g g}^{-1}$ in the light fractions

enriched in coaly matter with 2.54% ash content, and reaches a value of $0.67 \mu\text{g g}^{-1}$ in the fraction with an ash content of 79.4% (Figure 3). In coal B (Figure 4), the fractions enriched in ashes do not correspond to the fractions enriched with mercury, and the mercury concentration is maximum in the density fractions between 1.6 and 2.2 kg l^{-1} , which have an ash content of between 22.9 and 62.5%. It should be noted that the minimum values for mercury content in B are high compared to those for coal A. The mercury concentrations in the fractions with the lowest mineral matter contents are 0.02 and $0.19 \mu\text{g g}^{-1}$ in coals A and B respectively. The differences between the coals in terms of mercury association can be more clearly seen when the cumulative mercury content is compared to the cumulative ash content (Figures 5 and 6). Although in both cases, the cumulative mercury content increases with the cumulative ash content, the slope of the curve is steeper for coal A and tends towards the origin, suggesting that mercury is present in the mineral matter of this coal, whereas the slope of the curve corresponding to coal B is more gentle and does not tend towards the origin. This signifies that some of the mercury in this coal is associated with organic matter. If coal A were cleaned by using density separations in order to obtain a product with an ash content of around 10% wt, the product would have a mercury content of around $0.16 \mu\text{g g}^{-1}$ implying a reduction close to 35%.

The agglomeration of coals A and B with different types and concentrations of vegetable oils was found to be an efficient process in terms of %OMR, %AR and EI. Tables 4 and 5, show the results obtained by agglomeration with different concentrations of refined and crude sunflower and soybean oils. These Tables contain the percentages of the agglomerated yield (%wt), the ash content of the agglomerates (% ash), the %OMR, %AR and EI, and the mercury content on a coal and ash basis. In the high rank coal A, the %OMR increases like the oil concentration used in the agglomeration experiment, whereas in coal B the behavior is quite the opposite: maximum values of %OMR were obtained in the agglomeration

experiments performed with lower oil concentrations. In accordance with the %OMR values, %AR is higher in B than in A. The EI for coal A can be considered of the same order in all cases with values ranging between 28 and 34. The results for coal B reveal more remarkable differences for the different oil concentrations, the highest EI being for an oil concentration of 10% (more than 40), and the minimum (as low as 9), for an oil concentration of 40%. Although, oil agglomeration with vegetable oils proved to be an efficient process for cleaning coals, the removal of mercury was negligible. The ash content was reduced from 24.8 to 16.3 wt %, and from 28.7 to 12.1 in coals A and B respectively, but the mercury concentration in the agglomerates was found to be similar to those of the original samples. The mercury concentration in the agglomerated products obtained in the different conditions for both coals was of the same order, i.e. between 0.22 to 0.26 $\mu\text{g g}^{-1}$. Moreover, if mercury content is considered on an ash basis, it may be observed that it increased after the agglomeration processes (Tables 4 and 5).

Assuming that mercury is not efficiently rejected by the agglomeration procedures, the use of the recovered product enriched in coaly matter in a power station would enhance energy production efficiency, leading in turn to a slight reduction in mercury emission. The extent of the reduction was evaluated by calculating the index I.

$$I = \frac{[\text{Hg/om}]_{\text{coal}}}{[\text{Hg/om}]_{\text{agglomerate}}}$$

In this ratio Hg_{coal} and $\text{Hg}_{\text{agglomerate}}$ represent the concentrations of mercury in the coal and agglomerated product. The organic matter (om) was calculated as $100 - \text{ash \%wt}$. Values of I higher than 1 signify that total mercury emission can be reduced by burning the agglomerated product recovered instead of the raw coal. Figures 7 and 8 compare the values of index I for the products obtained by oil agglomeration using different types and concentrations of vegetable oils. In all cases I values are lower than 1.5 and in most cases close to 1, indicating that mercury emissions might be similar or slightly lower. If the I values of coals A and B are

compared, it can be concluded that the agglomeration of coal B will reduce mercury emission to a greater extent than in the case of coal A.

The results for mercury segregation in the fractions obtained by size fractionation, density separation and oil agglomeration, lead to two types of conclusion, one of which is related to the mode of occurrence of mercury, while the other concerns the possibility of mercury rejection by physical coal cleaning methods before coal combustion. As regards the mode of occurrence of mercury, major differences were observed. The mineral association of mercury in coal A and its partial organic association in B were assessed by means of density separation. Organic association in coal B may occur because: 1) the mercury is chemically bounded to coal organic matter or 2) it is finely distributed as minerals in the coaly matter. The second interpretation makes sense in the light of the results of size fractionation. The explanation that mercury in coal B was associated with organic matter agrees with the results obtained by agglomeration. If an element is organically bound or physically associated with organic matter, it will be difficult to reject by physical coal cleaning and, in the case of agglomeration it is concentrated in the agglomerated product. Assuming that mercury is associated to mineral matter in coal A, the behavior of this element when coal A is agglomerated could be explained if mercury is present as sulphur minerals or associated to pyrite. The difficulty of removing pyrite and other sulfides from coal by oil agglomeration may be due not only to the partial removal of this mineral when it is finely distributed in coaly matter, but also to its hydrophobic nature, which can vary widely from coal to coal. The differences between a coal and another will depend on factors such as morphology, origin and the state of oxidation of the sulfur minerals in different coals. Consequently, in coals in which mercury is associated with mineral matter, as in the case of coal A, a reduction in mercury content may be efficiently achieved by using density separation rather than by oil agglomeration procedures.

Conclusions

The results obtained demonstrate that in the coals studied, coal cleaning may reduce mercury emissions only-as a result of an increase in energy efficiency derived from organic matter recovery. Of the physical cleaning methods evaluated in this work, that of density separation may lead to a significant reduction in mercury, when this element is associated with mineral matter.

Physical cleaning methods give useful information on mercury speciation, and from the results obtained by these methods at laboratory scale, the different modes of occurrence of mercury in coals may be inferred.

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Table 1.- Characteristics of the coals

	Coal A	Coal B		Coal A	Coal B
<i>Proximate analysis (wt%)</i>			<i>Ash composition (wt% ash)</i>		
Moisture	1.2	1.1	SiO ₂	53.6	51.5
Volatile matter (db)	7.9	17.6	Al ₂ O ₃	23.6	28.8
Ashes (db)	24.8	28,7	Fe ₂ O ₃	6.94	6.86
<i>Elemental analysis (wt% daf)</i>			CaO	6.60	3.51
C	93.7	87.9	MgO	2.74	1.65
H	3.2	4.9	Na ₂ O	0.87	0.75
O (diff)	0.88	4.3	K ₂ O	2.96	3.94
N	1.3	1.5	TiO ₂	1.14	1.15
S _{total}	0.92	1.3	<i>Sulfur forms (wt% daf)</i>		
<i>Mercury</i>			Pyritic	0.57	0.49
Hg ($\mu\text{g g}^{-1}$)	0.22	0.26	Sulfate	0.02	0.02
			Organic	0.33	0.42

Table 2.-Results obtained from the analysis of mercury using AMA equipment

	SARM19	SARM20	1633b
n	10	10	10
Certified ($\mu\text{g g}^{-1}$ Hg)		0.25	0.14
Reference ($\mu\text{g g}^{-1}$ Hg)	0.2		
x ($\mu\text{g g}^{-1}$ Hg)	0.23	0.25	0.15
SD	-----	0.013	0.014
RSD %	-----	5.2	9.3

Reference is given as uncertified in the certificate analysis of the standard sample

Table 3.-Results of size fractionation calculated as cumulative weight, ash and mercury contents

Size mm	Coal A			Coal B		
	Cum wt %	Cum Hg $\mu\text{g g}^{-1}$	Cum. ash wt%	Cum wt %	Cum Hg $\mu\text{g g}^{-1}$	Cum ash wt %
0.355-0.500	13.4	0.13	21.6	15.1	0.22	33.4
>0.355	31.3	0.16	21.9	34.8	0.22	32.4
>0.250	44.5	0.16	22.0	53.4	0.23	30.2
>0.180	55.7	0.17	21.9	65.9	0.23	29.1
>0.125	61.1	0.18	21.9	70.9	0.23	28,7
>0.100	71.3	0.19	21.9	77.4	0.23	28.0
>0.063	76.0	0.19	21.9	81,9	0.23	27.6
>0.045	85.0	0.20	22.0	88,2	0.24	27.1
<0.500	100	0.22	24.8	100	0.26	27.8

$$\text{Cum wt\% (cumulative weight)} = \Sigma w_i = w_1 + w_2 + \dots + w_n$$

$$\text{Cum Hg } \mu\text{g g}^{-1} \text{ (cumulative mercury)} = [\text{Hg}_1 w_1 + \text{Hg}_2 w_2 + \dots + \text{Hg}_n w_n] / \Sigma w_i$$

$$\text{Cum ash wt\% (cumulative ash)} = [a_1 w_1 + a_2 w_2 + \dots + a_n w_n] / \Sigma w_i$$

Table 4.-Results of oil agglomeration for coal A

	Oil wt%	wt %	ash %	% OMR	% AR	EI	$\mu\text{g g}^{-1}$ Hg coal basis	$\mu\text{g g}^{-1}$ Hg ash basis
coal			24,8				0.22	0.89
Gir	10	62.2	16.3	69	59	28	0.23	1.41
	20	77.6	16.7	86	48	34	0.25	1.49
	40	81.5	17.3	90	43	33	0.22	1.27
Gic	10	58.2	15.9	65	63	28	0.21	1.32
	20	63.0	16.7	70	58	27	0.22	1.32
	40	76.6	18.7	83	42	25	0.24	1.28
Sor	10	63.4	16.5	70	58	28	0.21	1.27
	20	84.8	18.0	92	38	31	0.20	1.11
	40	87.8	19.2	94	32	26	0.25	1.30
Soc	10	63.1	16,5	70	58	28	0.23	1.39
	20	73.2	18,2	80	46	26	0.23	1.26
	40	79.5	19,6	85	37	22	0.25	1.27

Table 5.-Results of oil agglomeration for coal B

	Oil %	wt %	ash %	% OMR	% AR	EI	$\mu\text{g g}^{-1}$ Hg coal basis	$\mu\text{g g}^{-1}$ Hg ash basis
coal			28.7				0.26	0.91
Gir	10	60.8	12.1	75	74	49	0.22	1.19
	20	52.3	16.6	61	70	31	0.25	1.50
	40	37.8	21.8	41	71	13	0.26	1.19
Sor	10	70.9	12.2	87	70	57	0.24	1.97
	20	72.2	14.4	87	64	51	0.24	1.67
	40	36.5	17.8	42	77	20	0.25	1.40
Soc	10	77.9	18.1	90	51	40	0.23	1.27
	20	75.6	22.2	82	42	24	0.23	1.03
	40	48.8	24.9	51	58	9	0.25	1.00

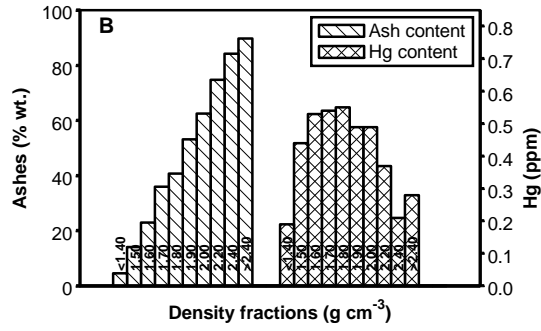


Figure 4.- Mercury and ash content in the density fractions of coal B

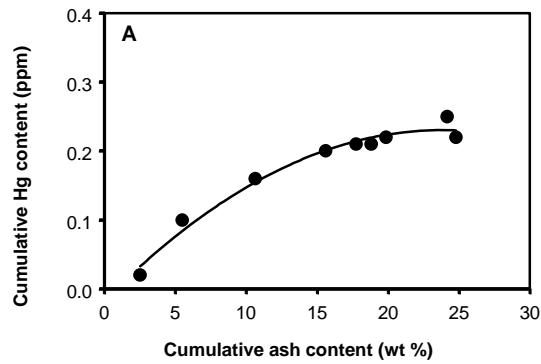


Figure 5.- Cumulative ash versus cumulative mercury contents in the density separations for coal A

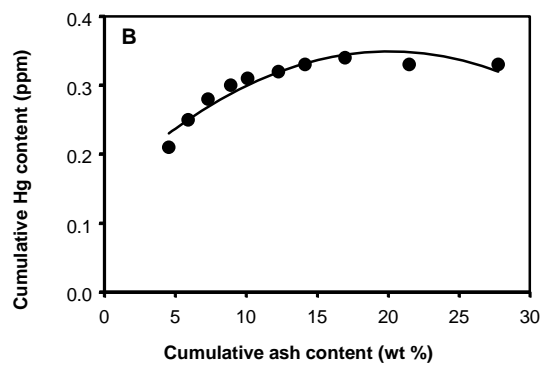


Figure 6.- Cumulative ash versus cumulative mercury contents in the density separations for coal B

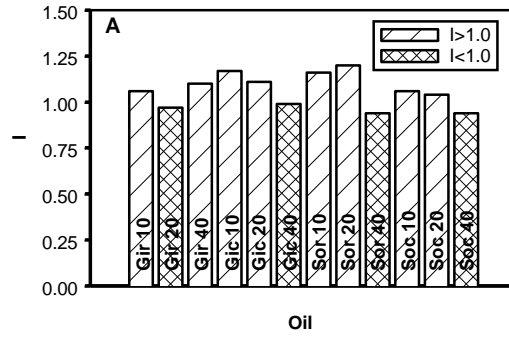


Figure 7.- I index for the agglomerates obtained using different concentrations of vegetable oils for coal A

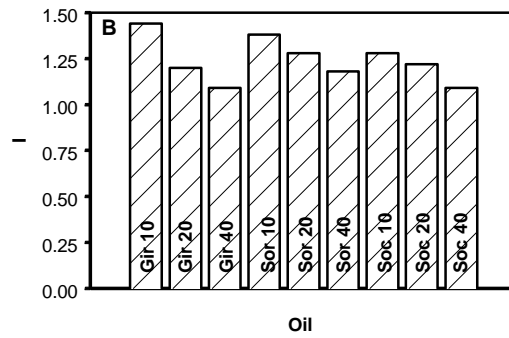


Figure 8.- I index for the agglomerates obtained using different concentrations of vegetable oils for coal B