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Trace Element Partitioning and Leaching in Solids Derived from Gasification of Australian Coals

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ABSTRACT

Trace element concentrations vary between coals from ppb to ppm levels and can depend on the rank of the coal and its geological origins. During gasification, some of the trace elements are volatilised at high temperatures and may condense and deposit in cooler downstream parts of the system or in quench water streams. Some species may appear in condensed phases such as slag or fly ash. Changes in the trace element concentrations in the slag and flyash from that of the parent coal are expected due to the reactions occurring at high temperatures and the different chemical activity of the trace element phases in the slag, fly ash, and syngas.

Four Australian coals were used in an entrained flow gasification test program conducted in the Siemens 5 MWth gasification test facility. Solid samples were collected from different points in the gasification process during each test. Compositions of these samples were analysed and the distribution of trace elements was studied.

The elements can be classified as follows, according to their tendency to appear in the slag and fly ash:
- Partitioned between slag and fly ash: Cu, W, Mo, Cd, Bi, Zn, Sn, Sb
- Partially volatile and depleted from either slag or fly ash: Be, Th, Sc, Y, Li, Mn, Ni, Sr, Ba
- Highly volatile (i.e. were not observed in either slag or fly ash): As, Se, B, Hg, F, Pb, V.

Comparison of these experimental results with equilibrium calculations of trace element appearance in the condensed phases suggests that the modelling approach is suitable only for certain elements. For several of the trace elements of significance in this study, kinetic factors have to be considered in conjunction with thermodynamic modelling.

The leaching behaviour of the trace elements in the slag was also studied. This work shows very low leachability for most of the trace elements except Zn and Sb, which, due to their relatively high volatility, reported to the slag samples in very low concentrations.

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1. Introduction

In addition to the main mineral components in coal (Si, Al, Fe, Ca, Mg, Ti, Mn, Na, K, P) and the minor elements such as S and Cl, there is a wide range of trace element species present at concentrations that vary extensively across coal types. The behaviour of these trace elements during gasification is of interest as their presence in solid waste streams from gasification processes can impact on disposal or utilisation strategies.

It has been reported (Roberts et al., 2010) that compositional changes of major elements occur in the slag and process water solids (mainly consisting of fly ash) during coal gasification. Therefore, it is likely that changes in the trace elements concentrations in the slag and flyash from the parent coal will also take place. Indeed, some of the trace elements will be volatilised at the high temperatures reached during gasification (Helble et al., 1996), or may be in liquid form. Trace element species have different chemical reactivity with the molten oxides in

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the slag formed on the wall of the gasifier or with molten flyash. Thus the trace elements partition between the slag, flyash and syngas and this depends on the occurrence of the elements in the coal and under gasification conditions (Font et al., 2010).

Various aspects of the mobilisation of trace elements during gasification have been studied (Helle et al., 1996; Richaud et al., 2000; Argent et al., 2002; Thompson et al., 2002; Alvarez-Rodriguez et al., 2007; Bunt et al., 2008, 2009, 2010; Font et al., 2010). Most of the studies are focused on the prediction of the phases in which trace elements appear. Ideally, techniques for predicting trace element behaviour would model the phase transformation of the trace elements, including their volatilisation during and after gasification as a chemical kinetic process. However, this approach is not practicable due to a lack of the necessary fundamental kinetic information and, therefore, most studies have adopted a thermodynamic equilibrium approach as a best approximation. Analysis of the distribution of trace elements in the slag, ash, and syngas is therefore useful for assessing the applicability of the thermodynamic phase equilibria approach for a particular trace element, and for determining future research needs.

The scope of this study is to analyse the distribution of the trace elements in entrained flow gasification solid wastes, such as slags and fly ash removed in the quench water, thereby providing some input for further development of trace element modelling under gasification conditions. Trapping of trace elements in the glassy phase has considerable consequences for the handling of solid wastes from the gasifier. In the present study, the behaviour of the trace elements in the slag was also investigated in terms of the elements leachability.

2. Experimental

2.1. Samples of slag and process water solids

Solid samples of slag and fly ash were obtained from the entrained flow gasification of four Australian coals: CRC701 is a low-rank (sub bituminous) coal from Western Australia, CRC702 is a high volatile bituminous coal from the Hunter Valley in NSW, CRC703 is a semi-anthracite from the Bowen Basin in Queensland, and CRC704 is a high volatile bituminous coal from the Surat Basin in Queensland. Coal characteristics and calculated operating temperatures for each trial are listed in Table 1. The pilot-scale test program (Harris et al., 2009) involved a range of gasifier operating conditions in order that the performance of the coals could be compared under a consistent set of conditions, and also that the effects of gasification conditions could be quantified for each coal in a comparable manner. Each test was performed under more than one set of steady state conditions (balance phases) during which the gasifier operated steadily—more details can be found elsewhere (Harris et al., 2009).

Solids present in the discharged quench water were collected, and these are referred to as ‘process water solids’ (PWS). Tapped slag samples were collected periodically throughout the trials, as batches using a lock-hopper system which removed slag from the bottom of the quench. PWS and slag samples from each balance phase were analysed for their chemical and physical characteristics.

2.2. Sample analysis

The concentrations of the trace elements were determined by ICP-AES and ICP-MS with the exception of As, Se (determined using hydride generation/atomic fluorescence techniques), and Hg (determined using a cold vapour/atomic fluorescence technique). Solid samples were either dissolved in mixed acids (including HF) or fused with lithium borate before dissolving in nitric acid. Liquid samples were acidic with nitric acid before measurement. Standards (for measurement) were prepared to match the matrix of the samples.

Leaching tests were undertaken by leaching slags for three days in distilled water at a liquid:solid ratio of 20:1, consistent with previous work done in this area (Jankowski et al., 2004, 2006; Ward et al., 2007). The leachates were then filtered through 0.22-µm Millipore cellulose acetate membrane filters. An unacidified portion of the filtrate was analysed for pH and conductivity. Another portion of the filtrate was acidified with approximately 1% v/v AR grade concentrated nitric acid and the concentrations of trace elements determined in the leachate using ICP-AES and ICP-MS.

3. Results and discussion

3.1. Distribution of trace elements between coal, slag and process water solids

The concentrations of the trace elements in the coals and their corresponding slag and PWS (which consists mainly of fly ash) is shown below in Figures 1–6. Based on these analyses, the trace elements can be categorised according to their appearance in the slag and fly ash:

1. Low volatile, partially lost from slag and fly ash
   - Known from the literature to be low or partially volatile
   - Some have escaped in gas phase or water
   - Some amount remains in PWS and slag

2. Volatile, recaptured on fly ash
   - Known from the literature to be volatile
   - Present in PWS but at higher concentration than in the parent coal mineral matter

3. Highly volatile from slag and/or fly ash
   - Lost to gas or water process streams
   - Low concentrations in PWS and/or slag

These categories can be further subdivided as per Table 2. The distributions of trace elements are discussed below in the context of the groupings shown in Table 2 and available thermodynamic modelling results.

Group 1.1. Present in both phases, some loss

First group of trace elements (Figure 1) are partially vapourised, and present in both the slag and the PWS at reduced concentrations. Generally, their concentration in the slag and PWS is about

---

Table 1
Coal characteristics and gasification operating temperatures. Temperatures are calculated based on heat loss through the cooling screen of the gasifier.

<table>
<thead>
<tr>
<th></th>
<th>CRC701</th>
<th>CRC702</th>
<th>CRC703f</th>
<th>CRC704</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture (%) as-received</td>
<td>6.4</td>
<td>6.7</td>
<td>9.0</td>
<td>2.3</td>
</tr>
<tr>
<td>Ash (%)</td>
<td>5.6</td>
<td>8.7</td>
<td>9.6</td>
<td>11.7</td>
</tr>
<tr>
<td>Volatile Matter</td>
<td>36.2</td>
<td>34.4</td>
<td>7.2</td>
<td>44.1</td>
</tr>
<tr>
<td>Fixed Carbon</td>
<td>51.8</td>
<td>55.6</td>
<td>82.3</td>
<td>41.9</td>
</tr>
<tr>
<td>Operating tempera</td>
<td>1460–1580</td>
<td>1550–1620</td>
<td>1480–1540</td>
<td>1670–1800</td>
</tr>
</tbody>
</table>
10–50% lower than in the parent coal mineral matter. However, the appearances of Mn, Ni, and Li have some exceptions: Mn concentration in CRC703 slag is nearly the same as in parent coal mineral matter, concentration of Li in CRC704 slag is higher than in the parent coal mineral matter, and concentration of Ni in CRC704 slag and PWS is higher than in the parent coal mineral matters. The reason for this is unclear, but could be associated with differences in the gasification conditions for each coal run (Table 1). Another reason for these exceptions may be enrichment with the trace elements from previous runs: concentrations of these elements are higher in coals previously tested, and slag samples may contain trace elements from the wall slags of previous runs.

Manganese is known to be a low-volatile element and Ni starts to vaporise in syngas atmospheres at temperatures above 1400 °C (Diaz-Somoano et al., 2003; Thompson et al., 2002). A notable loss of Ni in slag has been reported at temperatures above 1550 °C (Argent et al., 2002; Thompson et al., 2002) and at a temperature of 1800 °C 40%–80% of Ni is still concentrated in the condensed phases.
Group 1.2. (partially volatile) and Group 2 (volatile), recaptured on fly ash

These elements are those which have become volatile to some extent, but have been recaptured on the fly ash. Elements where, in general, a deficit in the slag is compensated by an increase in concentration in the PWS are shown in Figure 2. These elements are Cu, Mo, W, Cd, Bi, Zn, Sn, and Sb. The last five are highly volatile at high temperatures and nearly absent in the slag. It is likely they are captured by PWS at lower temperatures downstream of the gasifier. Modelling of Cd species indicates the high volatility at gasification conditions (Diaz-Somoano et al., 2003). However, Cd conservation in the slag indicates its moderate volatility in our experimental conditions. It is likely that for Cd species in the slag the residence time was not long enough to achieve equilibrium.

Copper appears in the slag at temperatures above 1100 °C, but starts to vaporise from the slag between 1350–1400 °C, and can coexist in gaseous and condensed phases at temperatures up to 2000 °C (Argent et al., 2002; Thompson et al., 2002). According to modelling results, Zn and Sn are mainly present in a gaseous form.

Fig. 2. Concentration of trace elements in coal mineral matter and redistributed between slag and PWS (groups 1.2 and 2: PWS dominant.)
at temperatures above 1000 °C (Argent et al., 2002) and 1300 °C (Thompson et al., 2002), respectively, while Sb gradually increases in the gaseous phase in the temperature range of 1300–1900 °C (Thompson et al., 2002).

Within group 1.2 some exceptions are observed: the PWS sample of CRC703 is not Cu-enriched. There is no loss of Mo from CRC702 slag, and some W loss from CRC702 and CRC703 slags. This is likely due to the high concentration of these elements in ‘previous’ CRC701 coal.

Group 1.3. Partially-volatile, recaptured on slag

Only Cr has a higher concentration in the slag than in the PWS and the related coal minerals, as shown in Figure 3, indicating a high solubility of Cr in the molten oxides at high temperatures. Thermodynamic modelling shows some losses of Cr in the condensed phase, which agrees with Cr concentration in PWS, but contradicts with Cr appearance in some slags (Diaz-Somoano et al., 2003). It is possible that this enhanced Cr concentration could be from materials used in the manufacture of gasifier wall materials. Further work here is required.

There is a very low concentration of V in the PWS, compared with the slag and the parent coal mineral matter. Vanadium species are captured by the molten oxides in the slag and became more chemically stable than in the fly ash. According to thermodynamic modelling (Diaz-Somoano et al., 2003), V$_2$O$_5$ is the most stable phase at temperatures higher than 500 °C.

Group 3.1. Highly volatile and depleted only from the slag

Two elements, As and Se, are expected to be highly volatile (Diaz-Somoano et al., 2003; Thompson et al., 2002) and are not present in the slag phase (Figure 4). As distinct from the Group 2 elements, which are also expected to be highly volatile (Bi, Zn, Sn and Sb), they are only partially recaptured by fly ash. These elements are predicted to be at a very low concentration in the slag at temperatures above 1000 °C (Diaz-Somoano et al., 2003; Thompson et al., 2002) but would most likely condense on the heat transfer surfaces that cool the syngas (Ratafia-Brown et al., 2002), which may explain their concentration in the fly ash. Another reason for their presence in fly ash may be that in this study the ash residence time in the gasifier was insufficient for complete As and Se volatilisation.

Group 3.2. Highly volatile: significant loss from solid phases

This group consists of the trace elements that are highly volatile and are not found in either the slag or PWS (Figure 5). The concentration of Hg, B, F, and Pb are significantly lower in the PWS than in the parent coal mineral matter and even less in the slag: Hg and F are nearly absent from the slag. Concentration of F in CRC702 coal is higher than the typical concentrations of trace elements found in coals (ppm level). Mercury is always considered as a highly volatile element at gasification temperatures and is mostly emitted in the flue gas (Diaz-Somoano et al. 2003). According to modelling, B is highly volatile at temperatures above 1600–1700 °C (Argent et al., 2002; Thompson et al., 2002), Pb is highly volatile at temperatures above 1000 °C (Argent et al., 2002; Diaz-Somoano et al., 2003) and could be expected to be totally in the gas phase under gasifier conditions. The appearance of Hg and B in fly ash indicates that it is likely that fly ash did not have enough residence time to achieve phase equilibria in the system or they are partially captured during the cooling.

Group 4. Questionable: coal/condition specific

Trace element concentrations are different in bituminous, sub bituminous, high volatile bituminous and semi-anthracite coals. In general, the partitioning of the trace elements between the slag and fly ash, especially partially and highly volatile elements, are very similar.
for all four coals. Exceptions are Co, Nb, U, and Be (Figure 6), whose distributions in the coals and waste products seems to be dependant on the parent coal or processing conditions (e.g. temperature).

Cobalt is predicted to be captured most strongly by the slag (Thompson et al., 2002). Beryllium is predicted to be in the gaseous form Be(OH)$_2$ at temperatures $>$1000 °C and this becomes the dominant Be-species at temperatures $>$1300 °C, and it is expected to condense before gas cleaning systems (Diaz-Somoano et al., 2003). On the other hand, thermodynamic modelling of Be species with other elements and phases under gasification conditions indicates that only about 2–10% of Be will be vaporised in the temperature range 1350–1900 °C (Thompson et al., 2002).

The results presented above indicate that the appearance of trace elements in the gasification solid residues does not always follow

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**Fig. 5.** Concentration of trace elements in coal mineral matter, PWS and slag (group 3.2: highly volatile, significant loss from solid phases).

**Fig. 6.** Concentration of Co, Be, Nb and U in coal mineral matter, PWS and slag (group 4: coal/conditions specific).
thermodynamic modelling predictions. Table 2 lists the trace elements where the thermodynamic approach is not applicable (label “N”) or is questionable. The relatively-long residence time of slag in a gasifier means that most of the trace elements in slag can be predicted by equilibrium modelling. Fly ash, however, has a significantly shorter residence time and, therefore, phase equilibria may not be achieved, and the trace elements appearance in fly ash needs also to be linked with kinetic considerations. Such modelling requires further systematic experimental data to be used as the basis of ongoing development.

### Table 2

Distribution of trace elements in slag and PWS. ”Y”- indicates that the behaviour is consistent with thermodynamic modelling, ”N”- indicates that the behaviour is inconsistent with thermodynamic modelling, ”?”- not clear. Arrows indicate how trace elements are re-distributed.

<table>
<thead>
<tr>
<th>Group</th>
<th>Appearance</th>
<th>Experimental slag</th>
<th>Experimental fly ash</th>
<th>Modelling slag</th>
<th>Modelling flyash</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Low volatile, some loss in slag/fly ash</td>
<td>Th, Sc, Y, Li, Mn, Ni, Sr, Ba</td>
<td>Cu, W, Mo, Cd</td>
<td>?</td>
<td>?</td>
</tr>
<tr>
<td>1.2</td>
<td>Low volatile: recaptured on fly ash</td>
<td>Cr, V</td>
<td></td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>1.3</td>
<td>Low volatile: concentrated in slag</td>
<td>Bi, Zn, Sn, Sb</td>
<td></td>
<td>N</td>
<td>?</td>
</tr>
<tr>
<td>2</td>
<td>Volatile, recaptured on fly ash</td>
<td>As, Se</td>
<td></td>
<td>Y</td>
<td>?</td>
</tr>
<tr>
<td>3.1</td>
<td>Highly volatile: depleted in slags only</td>
<td>B, Hg, F, Pb</td>
<td></td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>3.2</td>
<td>Highly volatile: significant loss from solid phases</td>
<td>Nb, U, Co, Be</td>
<td></td>
<td>?</td>
<td>?</td>
</tr>
<tr>
<td>4</td>
<td>Questionable: coal-specific</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.2. Slag leaching

The overall leachability (or solubility in leachate) of elements from the slag is low. However, since the trace element concentrations in the slag are different from those in the parent coal mineral matter, the leachability of each element has to be assessed in terms of the ratio to the total amount of the trace element in the slag before the leaching test. Data presented in Table 3 and Table 4 shows the absolute concentration of the trace elements in the leachates and the corresponding % of the trace element extracted from the slag. Slags s101–104 refer to slags from coals CRC701–704 respectively.

The leachability of major slag elements, such as Mg, Na, K, S, and P, is very low; the amount of each element in the leachates is generally less than 0.001% of the total amount in the slag.

The trace elements can be grouped into categories, depending on their leachability:

1) **High leaching elements** are the elements whose amount in the leachate is ~ 1% or more of the amount in the slag (in bold, Table 3). These elements are Mo, W, B, As, Zn, Se, and Sb. However, the last five correspond to trace elements which are generally vapourised from the slag, so only Mo and W are likely to be of concern.

2) Elements with **moderate leaching** properties, where the amount of element in the leachate is between 0.01–0.5% of the amount in the slag. These elements are Ni, Co, Li, Sc, and Cd.

3) Element with **low leaching** properties (< 0.01% of amount in the slag), such as Sr, Ba, Mn, Y, Th, and U.

4) Element with **leachability not detected** (and not shown in the table) are Cu, Cr, Sn, Bi, Hg, F, and Pb. Within this list, only Cu and Cr are conserved in the slag; all others are in the slag with very low concentrations.

### Table 3

Concentration of trace elements in leachates.

<table>
<thead>
<tr>
<th>Element</th>
<th>s101</th>
<th>s102</th>
<th>s103</th>
<th>s104</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>0.3</td>
<td>0.4</td>
<td>0.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Mg</td>
<td>0.29</td>
<td>0.18</td>
<td>0.22</td>
<td>0.22</td>
</tr>
<tr>
<td>Na</td>
<td>0.21</td>
<td>0.13</td>
<td>0.37</td>
<td>0.42</td>
</tr>
<tr>
<td>K</td>
<td>0.2</td>
<td>0.1</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>P</td>
<td>0.04</td>
<td>0.003</td>
<td>0.21</td>
<td>0.20</td>
</tr>
<tr>
<td>Zn</td>
<td>0.009</td>
<td>0.014</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Sr</td>
<td>0.03</td>
<td>0.024</td>
<td>0.012</td>
<td>0.014</td>
</tr>
<tr>
<td>Ba</td>
<td>0.014</td>
<td>0.012</td>
<td>0.001</td>
<td>0.014</td>
</tr>
<tr>
<td>Ni</td>
<td>0.016</td>
<td>0.035</td>
<td>0.006</td>
<td>0.014</td>
</tr>
<tr>
<td>Mn</td>
<td>0.04</td>
<td>0.004</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Co</td>
<td>0.003</td>
<td>0.003</td>
<td>0.001</td>
<td>0.002</td>
</tr>
</tbody>
</table>

(Elemet concentration in leachate, ppm, l:s = 20:1)

<table>
<thead>
<tr>
<th>Element</th>
<th>s103</th>
<th>s104</th>
</tr>
</thead>
<tbody>
<tr>
<td>Li</td>
<td>2.1</td>
<td>15.0</td>
</tr>
<tr>
<td>Mo</td>
<td>3.7</td>
<td>5.3</td>
</tr>
<tr>
<td>B</td>
<td>2.7</td>
<td>3.4</td>
</tr>
<tr>
<td>W</td>
<td>0.4</td>
<td>0.43</td>
</tr>
<tr>
<td>Sc</td>
<td>0.3</td>
<td>0.55</td>
</tr>
<tr>
<td>As</td>
<td>0.2</td>
<td>0.56</td>
</tr>
<tr>
<td>Sb</td>
<td>0.04</td>
<td>0.11</td>
</tr>
<tr>
<td>Cd</td>
<td>0</td>
<td>0.015</td>
</tr>
<tr>
<td>Y</td>
<td>0.046</td>
<td>0.027</td>
</tr>
<tr>
<td>Se</td>
<td>0</td>
<td>0.26</td>
</tr>
<tr>
<td>Th</td>
<td>0</td>
<td>0.006</td>
</tr>
<tr>
<td>U</td>
<td>0</td>
<td>0.02</td>
</tr>
</tbody>
</table>

(Elemet concentration in leachate, ppm, l:s = 20:1)
A summary of these leaching results is:
- There is relatively high leaching of Zn from slags from CRC701 and CRC702 coals.
- Antimony is leached from slags from CRC702 and CRC704.
- There is high leaching of As, Se, and Mo from CRC703 and CRC704 slags.
- The same elements with moderate leaching or low leaching were observed for almost all four slag samples.

There are coal-specific issues that are influencing the leaching results presented above. The data presented here are not suitable for an analysis of coal properties and their impacts on trace element partitioning and leaching from gasification residues; however, such information is important and will form the basis of ongoing work in this area.

The appearance of trace elements in fly ash is different than in the slags. Therefore, the leaching of some of the trace elements from gasification-derived flyash could be considerably different to that from the slags. In order to be able to confidently manage the handling, disposal, or utilisation of solids from entrained flow gasification, ongoing work in this area is required.

4. Conclusions

This work has presented some preliminary data regarding the behaviour of trace elements during entrained flow gasification of Australian coals. Based on analyses of slag and fly ash samples, trace elements have been categorised into three groups based on their volatility under entrained flow gasification conditions:
- non-volatile or low volatile,
- volatile and,
- highly volatile;
and three groups based on their reactivity/solubility in the condensed phase:
- present in slag only;
- present in fly ash;
- present in both slag and fly ash.

A thermodynamic modelling approach for predicting trace element behaviour during gasification seems to be suitable for selected trace elements only, and usually only for their behaviour in the slag. Appearance of the trace elements in fly ash is often very different than the appearance in the slag, and occurs in a significantly shorter time frame; consequently, kinetic considerations have to be taken into account in order to accurately model their behaviour under entrained flow gasification conditions.

Leachability of trace elements from the slag samples was found to be very low for the majority of species. Only Mo, Zn, As, Se, and Sb concentrations in the leachate are in the level of a few percent of the concentrations in the slag, but the concentrations of last four elements in the slags are very low due to their high volatility.

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### Table 4

<p>| Trace element concentration in slag and the % of the element extracted. Leaching values exceeding 1% are in bold. |
|----------------------------------|------------------|------------------|------------------|</p>
<table>
<thead>
<tr>
<th>Concentration in slag, ppm / % of leached</th>
<th>s101 ppm</th>
<th>s102 ppm</th>
<th>s103 ppm</th>
<th>s104 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>16600</td>
<td>16800</td>
<td>11000</td>
<td>10500</td>
</tr>
<tr>
<td>Mg</td>
<td>10500</td>
<td>5900</td>
<td>6800</td>
<td>9600</td>
</tr>
<tr>
<td>Na</td>
<td>3300</td>
<td>2000</td>
<td>6000</td>
<td>6700</td>
</tr>
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Ilyushechkin et al. / Coal Combustion and Gasification Products 3 (2011)
References


