An Experimental and Thermodynamic Equilibrium Analysis on the Leaching Process of Arsenic (As) from Coal Fly Ash

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The leaching process of arsenic (As) from coal fly ash collected from the coal-fired power plant in Japan was investigated with six different kind of paper sludge ashes (PS ash XA, XB, XC, YA, YB, and YC) as an inhibitor and their mass addition ratio 0 to 30%. To study the arsenic leaching mechanism, thermodynamic calculation (FactSage 7.2) was used to predict the possible As-bearing compounds in the fly ash and its distribution during the combustion and leaching process. The results indicated AlAsO₄(s) was the most probable species resulting compound from arsenic interaction with fly ash components under the combustion process. PS ash YB containing the highest calcium content shows a considerable immobilization ability for arsenic due to arsenic reaction with calcium compound in the leaching process. CaO in PS ash generates high pH leachate during the leaching process and promotes calcium with arsenic to form a precipitate. The arsenic leaching ratio by thermodynamic calculation and experimental, again PS ash YB, shows the best effect in the arsenic leaching concentration with addition ratio in the range 15-30%. It was found that the arsenic fixation capacity of PS ash increases as the calcium content in PS ash increases because the chemical reaction between calcium compounds and arsenic is accelerated during the leaching process. Predicting arsenic species based on the combustion and leaching process will be useful to choose the best available control technology to minimize the effect of arsenic into the environment.

Key Words
Coal fly ash, Inhibitors, Combustion and leaching process, Thermodynamic calculation
1. Introduction

Coal power plants generate large volumes of coal combustion products such as flue gas emissions, bottom ash, and fly ash. Fly ash is the most hazardous coal combustion product because it contains environmentally significant quantities of hazardous leachable trace elements such as As, Mo, and Se \(^1\). During the combustion process, the organic matter in coal was destroyed, and as a result, the concentration of trace elements in fly ash enhanced relatively according to their contents in the feedstock coal \(^2\).

Arsenic released from combustion is one of the environmental concerns due to its toxic, potential carcinogenic propensities and high volatility. In general, fly ash derived from coal combustion is subjected to disposal and reuse. The leaching of arsenic will cause a potential hazard on aquifer systems and soil, which thus causes a critical environmental problem. With an increase in awareness of the public in the environmental impact of the combustion of coal, it is vital to control the emission of arsenic during coal combustion and its leaching during fly ash treatment, these elements can be removed by washing with water \(^3\). Ogawa et al., reported that the treatment of fourteen different alkaline fly ashes under aging for 1-4 weeks at 10-30 °C, the concentration of arsenic tends to decrease after age for a week and decrease with an increase in aging temperature in some coal fly ash samples.

The highly soluble particles such as CaO dissolve when they come into contact with water. The addition of only small amounts of water to fly ash for aging might produce both alkaline conditions and strong supersaturation conditions for the formation of several types of Ca-bearing minerals in fly ash paste, such as calcite and ettringite \(^4\). In another study, the higher leachability of arsenic in acidic fly ash (HA) and alkaline fly ash (PD) related to the presence of larger fractions of water-soluble and/or exchangeable arsenic in serial leaching test \(^5\). In addition, the temperature of the leaching process also affects the leaching of arsenic from fly ash. At a temperature above 30 °C, arsenic can be stabilized completely regardless of the pH of the process \(^6\).

Recent publication from our laboratory studied the effect of paper sludge ash and blast furnace cements as an additive material on the leaching of arsenic, boron, fluorine and selenium. This additive is believed to be a promising method for controlling the leaching of heavy metals \(^7\). The leaching characteristics of each heavy metal are different and that the alkalinity of the leaching solution plays an important role. In addition, the pH of the leachate is believed to be correlated with the calcium content in the coal. Wang et al. \(^8\) stated that arsenic and selenium have a high mobility in alkaline pH, and Sri et al. \(^9\) stated that leaching of arsenic is related to calcium oxide content in coal fly ash.

The thermodynamic calculation of chemical reaction in a closed system was performed according to the second law of thermodynamics using the principle of minimizing the Gibbss (free) energy. The Gibbss (free) energy factors include the chemical composition and structure, condensed state (gas, liquid, and solid). A few studies adopted thermodynamic calculation to quantify As portioning behavior during co-combustion, thus providing information about interactions based on a particular condition (e.g., temperature, pressure, and major elements in the sample) \(^10\). Liu et al. reported that sewage sludge co-combustion system with the multiple oxides of SiO\(_2\), Al\(_2\)O\(_3\) and CaO. As reacted with CaO and Al\(_2\)O\(_3\) to form Al\(_3\)AsO\(_4\)(s) and Ca\(_3\)(AsO\(_4\))\(_2\)(s) which in turn inhibited As volatilization. An abundance of free CaO in fly ash generates alkaline leachate during the leaching test in another study. It successfully reduces arsenic leaching since the precipitate preferentially occurs via arsenic reaction calcium at high pH leachate \(^12\). In addition, the presence of mineral elements show that only potassium has a significant effect on arsenic species while effects of Ca, Fe, Na on species partitioning in equilibrium are not observed \(^13\).

This study aims to study the influence of different paper sludge ashes with different mass addition ratios as an inhibitor, the possibility of arsenic formation, and its mechanism during the leaching process by using Factsage software analysis. The experimental study with the same parameter analysis is also displayed for comparison. The application of these inhibitors in the leaching process was a promising method to prevent arsenic compounds into the environment and minimize its leachability from coal fly ash.

2. Experimental

2.1 Coal fly ash sample

The coal fly ash sample used in this research was obtained from coal-fired power plants in Japan, namely FA AA. The chemical composition of the coal fly ash sample was determined by X-ray fluorescence analysis (WDXRF S8 TIGER, Bruker AXS). Table 1 shows the chemical composition of the coal fly ash sample used in this study. Based on XRF analysis, the calcium content in FA AA is 1.13%, and arsenic concentration is 12.53 mg/kg, respectively.

2.2 Paper sludge ash

Paper sludge ash (PS ash) is a waste generated by the paper recycling industry. It is produced when dewatered waste paper sludge, a by-product of the de-inking and repulping of paper, is combusted to reduce waste volume and to produce energy. The utilization of paper sludge ash was
considered due to the relatively high calcium content. As mentioned before, the abundance of free CaO successfully reduces arsenic leaching since the precipitate preferentially occurred via reaction of arsenic with calcium. Based on these reasons, PS ash was used in this study to exhibit an inhibiting effect on the release of arsenic during the leaching test. The CaO content is determined by X-ray fluorescence analysis (WDXRF S8 TIGER, Bruker AXS). As shown in Table 1, the PS ash consists of different calcium content from the lowest (PS ash YA, 42.03%) to the highest calcium content (PS ash YB, 51.63%). It should be noted that PS ash also contains the arsenic compound, which may affect the leaching characteristic of arsenic and distribution of As-bearing compounds during the leaching process. Arsenic concentration in PS ash in the range of 1.5 to 11 mg/kg.

2.3 Leaching test
The leaching behavior of arsenic from coal fly ash was quantified using a leaching test according to the procedure of Environment Agency of Japan Notifications No. 13. Regarding the leaching procedure used in this work, a prepared coal fly ash sample of 5 g was mixed with 50 mL distilled water in a polyethylene bottle, and the slurry was stirred for 6 hours at room temperature. The solid-liquid sample was separated by filtration using a membrane filter of 0.45 µm. The concentration of arsenic infiltrate was carefully analyzed with HG-ICP AES.

2.4 Thermodynamic calculation analysis
In this study, the thermodynamic calculation was carried out using FactSage 7.2 during combustion and leaching process under different conditions. The FactSage covers a compound database of all coal fly ash components and provides information on the phases formed, their proportions and compositions.

To simulate the predicted possible arsenic species during combustion process, a pure O₂ and 100 g of coal were used for calculation. The calculation was performed in different temperatures interval between 100 to 1600 °C, at atmospheric pressure.

In the leaching process, the predicted arsenic after combustion process and paper sludge ashes were used as input data. In the case of leaching process, six different kind of paper sludge ashes (PS ash XA, XB, XC, YA, YB, and YC) and their mass addition ratio (0, 5, 10, 15 and 30%) were the key parameter in this study due to present of CaO as the important role on the leaching of arsenic. The data search used in this analysis includes FactPS and FToxid databases inbuilt in FactSage. During simulation analysis on the combustion and leaching process, aqueous, gases, and solid species are chosen as output.

3. Results and Discussion

3.1 Interaction of arsenic with fly ash components during combustion process
Arsenic behavior in a combustion process is dependent on the amount of arsenic, its interaction with gas-phase species, ash components, and temperature during the combustion process. Arsenic-fly ash interaction favors the formation of arsenates. The predicted possible arsenic species during combustion process are listed Table 2.

Table 2

| Chemical compositions | Coal fly ash Sample (FA A/A) | PS Ash XA | PS Ash XB | PS Ash XC | PS Ash YA | PS Ash YB | PS Ash YC |
|-----------------------|------------------------------|-----------|-----------|-----------|-----------|-----------|-----------|
| SiO₂                  | 59.38                        | 30.26     | 29.19     | 32.95     | 24.47     | 28.21     | 31.06     |
| Al₂O₃                 | 20.45                        | 15.89     | 15.59     | 16.52     | 14.10     | 15.61     | 16.88     |
| TiO₂                  | 0.62                         | 0.74      | 0.76      | 0.78      | 0.31      | 0.46      | 0.76      |
| Fe₂O₃                 | 15.65                        | 0.73      | 0.51      | 0.66      | 15.76     | 1.15      | 0.61      |
| CaO                   | 1.13                         | 46.78     | 48.86     | 42.77     | 42.03     | 51.63     | 47.46     |
| MgO                   | 0.61                         | 2.46      | 2.38      | 2.55      | 2.12      | 2.19      | 2.12      |
| Na₂O                  | 0.47                         | 0.12      | 0.08      | 0.24      | 0.38      | 0.04      | 0.12      |
| K₂O                   | 1.06                         | 0.54      | 0.50      | 0.59      | 0.10      | 0.13      | 0.15      |
| P₂O₅                  | 0.08                         | 0.50      | 0.43      | 0.45      | 0.31      | 0.23      | 0.15      |
| MnO                   | 0.07                         | 0.06      | 0.05      | 0.04      | 0.08      | 0.04      | 0.04      |
| V₂O₅                  | 0.01                         | 0.01      | 0.02      | 0.01      | 0.01      | 0.01      | 0.02      |
| SO₃                   | 0.49                         | 1.91      | 1.64      | 2.43      | 0.33      | 0.30      | 0.64      |
| Total                 | 100                          | 100       | 100       | 100       | 100       | 100       | 100       |
| Trace elements         |                              |           |           |           |           |           |           |
| Arsenic (As) [mg/kg]  | 12.53                        | 10        | 11        | 7.8       | 15        | 3.2       | 8.6       |
Interaction of As-Ca, As-Fe, As-Mg, and As-Na, the arsenates preferentially formed are Ca$_3$(AsO$_4$)$_2$(s), FeAsO$_4$(s), K$_3$AsO$_4$(s), Mg$_3$(AsO$_4$)$_2$(s), and Na$_3$AsO$_4$(s) (Fig. 1c-g), respectively. Finally, all the main fly ash component interactions have been introduced to study which are the dominant interaction with arsenic. As can be seen from Fig. 1h and interaction No. 15 in Table 2, although SiO$_2$ is the highest percentage component in coal fly ash, the presence of Al as Al$_2$O$_3$ inhibits the effect of other elements on the formation of arsenic-bearing species. Arsenic is predicted to form gaseous oxide and arsenates, at a temperature higher than 1400ºC small amount of AsO(g) is formed, and AlAsO$_4$(s) is formed and stable at temperature 1400ºC to a final temperature of the combustion process indicating that AlAsO$_4$(s) is the arsenate containing in fly ash. This process can be explained by the following reaction to generate AlAsO$_4$ through Eq. (1).

$$\text{Al}_2\text{O}_3 + \text{As}_2\text{O}_3 + \text{O}_2 \rightarrow 2\text{AlAsO}_4 \ (1)$$

3.2 Interaction and leaching behavior of arsenic in fly ash and paper sludge ash

The thermodynamic calculation was employed to predict the possible As-bearing compounds in fly ash and its distribution in water solution theoretically. Table 3 shows the possibility of arsenic formation during the interaction of predicted arsenate from the combustion process with paper sludge ash components during the leaching process. Based on the interaction, not only calcium compounds can inhibit the leached out of arsenic but also K and Mn. The possible mechanism of arsenic distribution during the leaching process is demonstrated in Fig. 2. Firstly, the mass of Al$_2$O$_3$ and H$_2$SiO$_4$ (as the results of reaction of SiO$_2$ and water) start to decrease, and CaAl$_2$Si$_2$O$_7$(OH)$_2$ increases by increasing of mass CaO in PS ash. Again, at the mass of CaO 0.4 gram CaAl$_2$Si$_2$O$_7$(OH)$_2$ start decrease to form Ca$_3$Al$_2$Si$_3$O$_12$, and that specie stable by increasing mass CaO. Finally, the formation of Ca$_3$(AsO$_4$)$_2$ due to reaction of AlAs$_2$O$_4$ with CaO and that species stable during the leaching process. Based on the results, calcium especially for free CaO, seemingly still plays an important role on the leaching of arsenic via the reaction of arsenic with calcium. To better understand the possible mechanism of arsenic formation during the leaching process can be explained through Eq. (2), (3), (4), and (5).

$$\text{SiO}_2 + 2\text{H}_2\text{O} \rightarrow \text{H}_2\text{SiO}_4 \ (2)$$

$$2\text{H}_2\text{SiO}_4 + \text{Al}_2\text{O}_3 + \text{CaO} + \text{H}_2\text{O} \rightarrow \text{CaAl}_2\text{Si}_2\text{O}_7(\text{OH})_2 + 4\text{H}_2\text{O} \ (3)$$

$$3\text{CaAl}_2\text{Si}_2\text{O}_7(\text{OH})_2 + 3\text{CaO} + 3\text{H}_2\text{O} \rightarrow 2\text{Ca}_3\text{Al}_2\text{Si}_3\text{O}_12 + \text{Al}_2\text{O}_3 + 4\text{H}_2\text{O} \ (4)$$

$$2\text{AlAsO}_4 + 3\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca}_3(\text{AsO}_4)_2 + \text{Al}_2\text{O}_3 + \text{H}_2\text{O} \ (5)$$

*No reactions between S/Si/Ti and As were observed*
Fig. 1 Equilibrium composition of arsenic (As) considering As-coal fly ash component interactions during combustion process: a) As-O₂, b) As-Al, c) As-Ca, d) As-Fe, e) As-K, f) As-Mg, g) As-Na, and h) As-all fly ash components
### Table 3 Interaction of predicted arsenate with paper sludge ash components in leaching process

| No. | Interaction | Phase | Reaction |
|-----|-------------|-------|----------|
| 1   | AlAsO₄ + H₂O | s     | AlAsO₄ + H₂O → AlAsO₄ + H₂O |
| 2   | AlAsO₄ + H₂O + SiO₂ | s     | AlAsO₄ + SiO₂ + H₂O → AlAsO₄ + H₄SiO₄ |
| 3   | AlAsO₄ + H₂O + Al₂O₃ | s     | AlAsO₄ + H₂O + Al₂O₃ → AlAsO₄ + H₂O + Al₂O₃ |
| 4   | AlAsO₄ + H₂O + TiO₂ | s     | AlAsO₄ + H₂O + TiO₂ → AlAsO₄ + H₂O + TiO₂ |
| 5   | AlAsO₄ + H₂O + Fe₂O₃ | s     | AlAsO₄ + H₂O + Fe₂O₃ → AlAsO₄ + H₂O + Fe₂O₃ |
| 6   | AlAsO₄ + H₂O + CaO | s     | 2AlAsO₄ + 3CaO + H₂O → Ca₃(AsO₄)₂ + Al₂O₃ + H₂O |
| 7   | AlAsO₄ + H₂O + MgO | s     | AlAsO₄ + H₂O + MgO → AlAsO₄ + Mg(OH)₂ |
| 8   | AlAsO₄ + H₂O + Na₂O | s     | AlAsO₄ + H₂O + Na₂O → AlAsO₄ + H₂O + Na₂O |
| 9   | AlAsO₄ + H₂O + K₂O | s     | 2AlAsO₄ + 3K₂O + H₂O → 2K₃AsO₄ + Al₂O₃ + H₂O |
| 10  | AlAsO₄ + H₂O + PO₄ | s     | AlAsO₄ + H₂O + PO₄ → AlAsO₄ + H₂O + PO₄ |
| 11  | AlAsO₄ + H₂O + SO₃ | s     | AlAsO₄ + H₂O + SO₃ → AlAsO₄ + H₂O + SO₃ |
| 12  | AlAsO₄ + H₂O + V₂O₅ | s     | AlAsO₄ + H₂O + V₂O₅ → AlAsO₄ + H₂O + V₂O₅ |
| 13  | AlAsO₄ + H₂O + MnO | s     | 2AlAsO₄ + 4MnO + H₂O → Mn₃(AsO₄)₂ + MnAl₂O₄ + H₂O |

**Fig. 2** Calcium effect in paper sludge ash (PS ash XA, addition ratio 5%) on arsenic leaching mechanism at 25°C atmospheric condition: a) all species formation and b) magnification on arsenate formation.
Based on the above discussion, Ca, K, and Mn are the compounds that can inhibit the leached out of arsenic. However, the presence of calcium still plays an important role in the leaching of arsenic and inhibits the effect of other compounds in the leaching process, especially for free CaO, which possesses a higher solubility during the leaching process. CaO promotes the combination of calcium and arsenic at high pH leachate to form a precipitate, which is slightly soluble in water. Paper sludge ash as the inhibitor contained high calcium that could enrich the calcium content in coal fly ash. The leaching behavior of arsenic into the environment was expected to be minimized.

Fig. 3 shows the arsenic leaching ratio in coal fly ash. Here arsenic leaching ratio was defined as the percentage of total mass/concentration of arsenic in the leachate to its total mass/concentration in fly ash and paper sludge ash. The experimental analysis with different paper sludge ash and mass addition ratio is also displayed for comparison. Fig. 3 a, in experimental analysis, PS ash YB containing the highest calcium content shows an enormous immobilization ability for arsenic. The arsenic leaching ratio of 99.77% was achieved by experimental compared to 99.98% as prediction results for paper sludge ash YB. In Fig. 3 b, in experimental analysis, increase in blending ratio of paper sludge ash YB also increase the arsenic leaching ratio (the arsenic leaching ratio increase 92.90% to 99.77% with addition ratio 5% to 30%, respectively). Only the arsenic leaching ratio from paper sludge ash YB is a line between experimental and FactSage analysis with an addition ratio in the range of 15% to 30%. Arsenic leaching ratio determined by experimental analysis is lower than that from a prediction by FactSage.

Related to these results, Sri Hartuti et al. also reported that leaching test results were compared with solution equilibrium calculation to consider the leaching mechanisms; however, the experimental results were significantly lower than the equilibrium calculation results. A possible reason is a kinetic control during the leaching test due to a short leaching duration. Also, thermodynamic calculation do not consider the reaction time, which is necessary for this theoretically equilibrium state to be reached.

3.3 Effect of paper sludge ash on pH of arsenic leaching process

The process of exhibits an inhibiting effect on arsenic release during the leaching test can be studied by the pH leachate. As reported, the increase in leachate’s pH exhibits an inhibiting effect on the release of arsenic during leaching tests and reducing the arsenic leaching process since the reaction of arsenic with calcium at high pH leachate [8]. This indicates the relatively higher CaO content of PS ash added into fly ash tend to increase the pH of the mixture leachates. Through comparing the pH leachate analysis from FactSage and experimental, as tabulated in Table 4, the effect of different paper sludge ash with different in mass addition ratio was studying.

Both in the analysis show that pH leachate increases by increasing the mass addition ratio in all paper sludge ashes. Again, the experimental study is lower than that from a prediction by FactSage. Based on the above discussion, an increase in the pH leachate exhibits an inhibiting effect on the release of arsenic during the leaching test, and the analysis of the results indicating that the success on the inhibiting process of arsenic release from

Fig. 3 Leaching behavior of arsenic in coal fly ash: a) effect of different kind of paper sludge ashes (addition ratio 30%) and b) effect of addition ratio of paper sludge ash (PS ash YB)
the coal fly ash by using paper sludge ash as an inhibitor.

4. Conclusions

The arsenic interaction from coal fly ash with paper sludge ash in the combustion and leaching process was carried out by FactSage analysis and experimental analysis for comparison. The results confirmed that arsenic interaction and the main fly ash, arsenic element preferentially formed mainly AlAsO$_4$ after the combustion process. Ca$_3$(AsO$_4$)$_2$ was predicted to be arsenate compound forming in leaching process of coal fly ash with paper sludge ashes as an inhibitor, PS ash YB with containing the highest calcium content, shows an enormous immobilization ability for arsenic and only the arsenic leaching ratio from paper sludge ash YB is a line between experimental and FactSage analysis with addition ratio in range 15% to 30%. Calcium especially for free CaO, seemingly still plays an important role on the leaching of arsenic. The result of the experiments are lower than that obtained by the prediction. A possible reason is a kinetic control during the leaching test due to a short leaching duration. Also, thermodynamic calculation do not consider the reaction time, which is necessary for this theoretically equilibrium state to be reached. Arsenic leaching process optimally working at alkaline pH during leaching test and successively reduces arsenic leaching since the precipitate preferentially occurred via arsenic reaction with calcium compound at alkaline pH leachate.

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| Addition Ratio (%) | Analysis | pH | Paper Sludge ashes |
|--------------------|----------|----|-------------------|
|                    |          | XA | XB                | XC | YA | YB | YC    |
| 5                  | Experimental | 11.005 | 10.890 | 10.180 | 9.855 | 10.340 | 10.500 |
| 10                 | Experimental | 11.275 | 11.335 | 9.835  | 10.090 | 11.285 | 11.080 |
| 15                 | Experimental | 11.525 | 11.375 | 11.385 | 10.885 | 11.420 | 11.380 |
| 30                 | Experimental | 11.850 | 11.280 | 11.455 | 11.085 | 11.870 | 11.520 |
| 5                  | FactSage  | 12.326 | 12.320 | 12.340 | 12.300 | 12.304 | 12.313 |
| 10                 | FactSage  | 12.371 | 12.358 | 12.400 | 12.294 | 12.297 | 12.322 |
| 15                 | FactSage  | 12.417 | 12.398 | 12.460 | 12.300 | 12.305 | 12.343 |
| 30                 | FactSage  | 12.548 | 12.515 | 12.614 | 12.318 | 12.330 | 12.408 |