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Removal of arsenic in coal fly ash by acid washing process using dilute H_2SO_4 solvent

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ABSTRACT

Coal fly ash emitted from coal thermal power plants generally contains tens ppm of arsenic, one of the hazardous elements in coal, during combustion and their elution to soil or water has become a public concern. In this study, the acid washing process developed by the authors was applied to the removal of arsenic from coal fly ash. Laboratory- and bench-scale investigations on the dissolution behavior of arsenic from various coal fly ash samples into dilute H₂SO₄ were conducted. Arsenic in the coal fly ash samples were dissolved into H₂SO₄ solutions rapidly. However, its concentrations decreased with an increase in the PH of H₂SO₄ was estimated as H₃AsO₄, and its anionic species was considered to adsorb with the elevation of PH under the presence of ash particle. Such adsorption behavior was enhanced under the presence of Fe ion in the solution. The sufficient removal of arsenic was achieved by controlling pH and avoiding the adsorption of arsenic on the surface of coal fly ash particles, and the elution of arsenic from coal fly ash sample was successfully below the regulation limit.

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1. Introduction

Electric power consumption in Japan has increased in this decade [1]; consequently, this has led to an increase in the demand for coal as an alternative energy source for electric power generation [2]. As a result of this increase in coal consumption, the amount of coal ash emission has also increased and exceeded 10 million tons per year in Japan in recent years [3].

Coal ash is roughly classified into coal fly ash and coal clinker ash, and their generation ratio is reported to be approximately 90:10 [4]. Coal fly ash is generally collected using a bag filter with electrostatic precipitation, while coal clinker ash accumulates at the bottom of the combustion furnace.

The recycling of coal fly ash has been promoted in various industries in order to comply with "The Basic Law for Establishing the Recycling-based Society", which has been in force in Japan since

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2000. This law encourages the minimization of the amount of landfilled coal fly ash because of the shortage of landfill sites in Japan [4]. Approximately 70% of coal fly ash has been utilized as raw material for fly ash cement in recent years, as authorized by the Japan Industrial Standard (JIS). The mixing ratio of coal fly ash and the mechanical properties of the fly ash cement are strictly regulated [5,6]. However, excessive input of coal fly ash into cements is no longer expected in Japan [7] as shown in Fig. 1. The stacked bar graph expresses the 'acceptable amount of coal fly ash' calculated by the production amount of Portland cement, fly ash cement, and the maximum mix rate of coal fly ash into Portland cement (5%) and fly ash cement (30%). Clear dissociation of the amount of recycled coal fly ash and acceptable coal fly ash could be observed since 2000, and this trend may imply that the mixing rate of coal fly ash into Portland cement has been beyond the JIS standard. Therefore, alternative approach for coal fly ash recycling needs to be developed and promoted in Japan.

Various types of environmentally hazardous substances in raw coal are known to condense on the surface of coal fly ash particles during their formation from coal, depending on their chemical nature and the combustion process [8–10]. Some of these substances tend to easily elute into the environment, and this elution has made it difficult to promote and popularize an alternative approach to coal fly ash recycling.

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Fig. 1. The relationship among the amount of coal fly ash emission, coal fly ash in cements, and the acceptable amount of coal fly ash for cements.

Above all, boron, fluorine, and arsenic have been recognized as the most troublesome hazardous elements in coal fly ash. It has been reported that the elution concentration of these elements from coal fly ash is often beyond the regulation limit, and therefore, new technologies need to be developed for their removal or stabilization. One of the probable techniques for the removal of these elements from coal fly ash is the acid washing process, which was proposed by the authors in a previous study [11]. In the first stage of this development, we focused on the removal of boron, which has been listed as an environmentally regulated substance since 2001. The removal of boron was successfully carried out in the practical phase of the acid washing process.

In this study, the removal of arsenic from coal fly ash samples was conducted in a bench-scale test plant for the acid washing process. In order to ensure that the margin of safety for industrial wastes including coal fly ash was within that for environmentally regulated substances, we employed the authorized elution test method "Notifications #18" and "#19" of the Soil Contamination Control Law in Japan [12]. Each method defines the elution concentration and the total contents of hazardous substances in a soil specimen. The regulated values for contents and elution of arsenic in coal fly ash are 150 mg kg⁻¹ and 0.01 mg L⁻¹, respectively. In many cases, arsenic was effectively removed by this process. However, in some cases, the removal of arsenic was insufficient. Furthermore, the elution of arsenic from the washed ash became easier after washing, and its concentration was greater than the regulation limit. In order to clarify the reason for such behavior

of arsenic during washing, some laboratory-scale tests were also conducted. According to the results, the bench-scale test plant was modified, and a new process to remove arsenic from coal fly ash was established.

2. Experimental

2.1. Bench-scale acid washing test

The coal fly ash samples were provided by an electric power company in Soma city in Fukushima prefecture, Tohoku region, Japan. Ash-A, B, C, and D listed in Table 1 were used for benchscale acid washing test. Like general coal fly ash, the coal fly ash samples used in the present work were mainly composed of SiO₂ and Al₂O₃, which are usually in the form of guartz and mullite. The coal fly ash samples also contained iron, alkali, and alkali earth as minor elements. The contents and elution concentration of arsenic are also shown in Table 1. As per "Notifications #18" and "#19", were utilized for determining the elution concentration and contents of arsenic, respectively. In the test method of Notification #18, 500 mL of distilled water with a pH range of 5.8-6.3 and 50 g of a coal fly ash sample were mixed and shaken for 6 h. On the other hand, in the method of Notification #19,6g of a coal fly ash sample was mixed and shaken with 200 mL of 1 M HCl for 2 h. Contents of arsenic were below the regulation upper limits established by Notification #19, while its elution concentration from coal fly ash samples exceeded the limits of elution concentration established by Notification #18 in many cases as it will be presented in the later section.

In the bench-scale test, 60 kg of Ash-A, B, C, and D were charged in the centrifuge whose capacity was 70 L and agitated with 60 L of 0.8 M of dilute H_2SO_4 for 10 min [10]. In this washing condition, the liquid–solid ratio was set at 1:1. The solution was then dehydrated by centrifugation at around 200 rpm. It was difficult to completely separate the H_2SO_4 solution from the slurry-like washed coal fly ash. Therefore, in the next step, 120 L of water was introduced into the centrifuge for rinsing. After running, the total content and the elution concentrations of arsenic in the as-supplied and washed coal fly ash samples were measured by hydride generation atomic absorption spectrometry (HG-AAS).

2.2. Laboratory-scale leaching test

A laboratory-scale leaching test was conducted in order to reveal the dissolution mechanism of arsenic from coal fly ash samples into the H_2SO_4 solvent. Ash-E, F, and G were selected for the test. 100 g of each coal fly ash sample was weighed and mixed with 1 L of H_2SO_4

Table 1

The chemical composition of 8 kinds of coal fly ash samples considered in this study. The contents and elution concentrations of arsenic are also listed. The bold values in elution show the excess over the regulation limit.

	А	В	С	D	Е	F	G
Chemical composi	ition (XRF, wt%)						
SiO ₂	71.8	75.5	62.2	61.3	60.8	62.5	62.2
A1 ₂ O ₃	21.7	17.9	20.8	24.3	28.5	28.6	20.8
Fe ₂ O ₃	1.66	2.64	3.44	3.31	4.22	3.82	3.44
CaO	1.07	1.09	8.52	6.68	1.61	1.15	8.52
MgO	0.42	0.57	1.57	1.13	0.67	0.56	1.57
Na ₂ O	0.38	0.37	0.37	0.34	0.67	0.43	0.37
K ₂ O	1.36	0.65	1.14	0.92	0.98	0.55	1.14
SO3	0.27	0.23	0.42	0.46	0.38	0.29	0.42
Total	98.66	98.95	98.46	98.24	99.0	99.1	98.46
Contents (mg kg ⁻¹)						
As	10	10	44	38	14	24	44
Elution by #18 (m	g L ⁻¹)						
As	0.098	0.05	0.006	0.006	0.17	0.19	0.006

solvent adjusted at 1 or 0.1 M, and leaching was carried out immediately by switching on the magnetic stirrer. In order to monitor the pH and the redox potential, pH and redox electrodes (Inlab410 and InlabRedox, Mettler Toredo) were connected with a laptop PC, and changes in the pH and the redox potential during the leaching test were continuously monitored at intervals of 10 s. For each sampling time, 10 mL of solution was picked up by the sampler and immediately filtrated within 30s using glass-fiber chromatography paper (GF-A, Whatman). Terminate leaching time was 10 min for each coal fly ash sample. 1 mL of the filtrates was collected by a micropipette and diluted in a 10 mL mess flask. 1 mL of 13.6 M nitric acid was added into the mess flask in order to maintain the pH around 0 and prevent any adsorption of arsenic onto the wall of the mess flask. An internal standard technique by 0.2 mgL^{-1} of cobalt addition was employed for the determination of arsenic concentration with an inductively coupled plasma mass spectroscopy (ICP-MS) in order to improve the accuracy.

High performance liquid chromatography (HPLC) measurement was also conducted in order to identify the ionic species of arsenic in the H_2SO_4 solution. 1 g of coal fly ash samples (Ash-F and G) and 10 mL of the H_2SO_4 solvent whose concentrations were 1 and 0.1 M were mixed and agitated for 5 min. Filtrates were then diluted by the procedure described above. Standard solutions containing As(III) and As(V) were prepared using distilled water and powders of As₂O₃ and KH₂AsO₄ (Wako Chemicals), respectively. The analysis of HPLC (HP 1200, Agilent) was carried out for all the standard solutions and specimens, and the separation of each species was achieved using ammonium phosphate buffers whose concentrations were 10 and 200 mM [13].

3. Experimental results

3.1. Bench-scale acid washing test

The relation between the total content and the elution concentrations determined by Notification #18 of arsenic was observed for the as-supplied and the washed ash. The results are shown in Fig. 2. The total content and elution concentrations of arsenic from both acid-washed Ash-A and B decreased after the benchscale test of acid washing process. The elution concentration of arsenic from washed Ash-A was less than the regulation limit, while that from washed Ash-B was still a little greater than the regulation limit. However, drastic decreases in the elution concentrations of arsenic from both washed Ash-A and B were clearly observed. In contrast, the elution concentrations of arsenic from acid-washed Ash-C and D increased and became greater than the regulation limit in spite of the acid washing. One of the most important aims of this study is to make the elution concentra-



Fig. 2. The relationship between contents and elution concentrations of arsenic from coal fly ash obtained from the bench-scale washing test. S in the bracket stands for 'as supplied', while W in the bracket shows 'washed'. The dotted line exhibits the environmental regulation limit by Notification #18.

tion of arsenic from coal fly ash less than the regulation limit of Notification #18 after the treatment with the acid washing process, and therefore, the increase in elution concentrations, defined as 'Re-elution', was undesirable for the acid-washing process.

3.2. Laboratory-scale leaching test

Fig. 3a and b shows the variation in the dissolution concentration of arsenic into acid solution from Ash-E, F, and G with leaching time. The dissolution behaviors of arsenic in Ash-E, F and G are partially different. When 1 M of the H₂SO₄ solvent having a liquid-solid ratio of 10 was used, the pH of each solution was almost invariant with time. In these cases, most of the arsenic in all fly ash samples was rapidly dissolved within 1 min. Then, its dissolution concentration was kept constant at the maximum level, which was calculated from the initial arsenic contents in each coal fly ash, shown in Table 1. Similar dissolution behavior was observed in the dissolution of arsenic from Ash-E and F with the 0.1 M H₂SO₄ solvent. In contrast, the dissolution of arsenic components from Ash-G with the 0.1 M dilute H₂SO₄ solvent exhibited quite a different dissolution behavior. The dissolution concentrations of arsenic initially increased rapidly and then decreased with time. Such behavior is called as "returning phenomenon" in this paper. When the returning phenomenon occurred, an increase in pH was also observed at the same time mainly because of dissolution of CaO contents. According to the difference of dissolution behavior of arsenic described above, we divided the coal fly ash samples into Group-I (Ash-E and F) and Group-II (Ash-G), where



Fig. 3. Changes in pH and arsenic concentrations from Ash-E, F, G and H at the laboratory-scale leaching test.



Fig. 4. The HPLC charts of arsenic from Ash-F and G in H₂SO₄ solution.

"returning phenomenon" was not been observed in Group-I but it occurred in Group-II.

Fig. 4 shows the HPLC charts of arsenic eluted from Ash-F or G by the dilute H_2SO_4 solvent. It is obvious that only As(V) was detected in the solution. From the viewpoint of peak strength, As(V) peaks from Ash-F show a similar profile, while the peak strength of As(V) in Ash-G with 0.1 M H_2SO_4 is considerably smaller than that of As(V) in Ash-G with 1 M H_2SO_4 . These detected peak signals were consistent with the dissolution concentration of arsenic measured by ICP-MS, shown in Fig. 3. These results indicate the exclusive existence of As(V) in the H_2SO_4 solution.

4. Discussion

4.1. The dissolution of arsenic in coal fly ash

A number of studies have been conducted on the behavior of arsenic in the coal combustion process [14–17]. Through these studies, it has been revealed that arsenic in original coal is completely volatilized in the oxide form, e.g., As_2O_3 (g). Thus, the mode of occurrence of arsenic in original coal seldom affects its speciation in the flue gas and coal fly ash particles [18]. Since the concentration of arsenic in coal fly ash samples is extremely low, the X-ray absorption fine structure (XAFS) method with the synchrotron orbit radiation is usually employed for the speciation analysis of arsenic in coal fly ash [18–21]. From these studies, it has been concluded that the main species of arsenic in coal fly ash is calcium arsenate (Ca₃(AsO₄₎₂). Ohki et al. [22] also detected (Ca₃(AsO₄₎₂) in the coal fly ash by X-ray photoelectron spectroscopy (XPS), and this result was in accord with the analysis by XAFS.

It is reported that there is a correlation between the arsenic content per unit weight and the particle size for a given type of coal fly ash [10,23–29]. These studies have annotated that the content of arsenic per unit weight is inversely proportional to the particle radii of coal fly ash particles. Since the specific surface area of a fine particle is inversely proportional to the particle radius, such a trend strongly indicates the deposition of vapor species of arsenic onto the surface of coal fly ash (hereafter referred to as the surface-



Fig. 5. The Eh–pH diagram of As–H₂O system. The values of redox potential were referenced from standard hydrogen electrode. The plots show the change in pH and redox potential of solution among 0.1 M H₂SO₄, Ash-F, and G.

deposition mechanism). If this surface-deposition mechanism is reasonable, arsenic components are condensed on the surface of coal fly ash particles as calcium arsenate; further, these arsenic components are considered to dissolve easily into an acid solvent.

At the early stage of leaching, a liberation reaction of calcium arsenate in coal fly ash may occur as per Eq. (1) given below:

$$Ca_{3}(AsO_{4})_{2} + 3H_{2}SO_{4} = 2H_{3}AsO_{4}(in H_{2}SO_{4}) + 3CaSO_{4}$$

log K (at 298 K) = 65.6 (1)

The presence of H_3AsO_4 in the H_2SO_4 solution at the early stage of leaching is consistent with the result of the HPLC chart shown in Fig. 4 and the Eh–pH diagram of As–Fe–S–H₂O shown in Fig. 5. The plots in Fig. 5 represent the change in pH and the redox potential of solution in the solution among Ash–F, G and the 0.1 M H₂SO₄ solvent. H_3AsO_4 in H_2SO_4 solution was considered to remain in the solution with no speciation changes in Ash–F solution. On the other hand, H_3AsO_4 in Ash–G–H₂SO₄ solution will transform into $H_2AsO_4^-$ as the leaching proceeds.

4.2. Mechanism of returning phenomenon

One of the possible mechanisms of the returning phenomenon is the adsorption of arsenic's monovalent oxyanions $(H_2AsO_4^-)$ onto the surface of coal fly ash. Wang et al. [30] found that the leachability of arsenic in coal fly ash samples was greatly affected by the pH of solution in acidic condition. They proposed the adsorption model based on the assumption of that H_3AsO_4 does not adsorb onto the surface of coal fly ash particles, while $H_2AsO_4^-$ and $HAsO_4^{2-}$ readily adsorb because of their negative charge. In their model, $[As(V)_{sol}]$, the concentration of arsenic species remained in the solutions, is expressed as Eq. (2):

$$[As(V)_{sol}] = \frac{[As(V)]}{1 + S_{T\gamma}(K_{s2\alpha_2} + K_{s3\alpha_3})}$$
(2)

where [As(V)] and S_T stand for the maximum concentration of arsenic in the solutions and total site concentration, respectively, α_2 and α_3 denote the fractions of As(V) as H₂AsO₄⁻ and HAsO₄²⁻. γ is the ratio of [H⁺]/([H⁺] + K_H). K_H , K_{s2} and K_{s3} are the equilibrium constant of Eqs. (3)–(5) below:

$$S-OH + H^+ = S-OH_2^+; K_H$$
 (3)

$$S-OH^{2+} + H_2AsO_4^{-} = S-H_2AsO_4 + H_2O; \quad K_{s2}$$
(4)



Fig. 6. The changes in arsenic concentration from Ash-G with pH. Fitting curve by modeling of surface adsorption is also drawn.

$$S-OH^{2+}+HAsO_4^{2-} = S-HAsO_4^{-} + H_2O; K_{s3}$$
 (5)

The predicted relations between arsenic concentration and pH in the solution calculated by Eq. (2) are drawn in Fig. 6 with the experimental result of Ash-G. The parameters of $pK_H = 3.5$, $S_T = 2.4 \times 10^{-4} \text{ mol g}^{-1}$, $\log K_{s2} = 4.4$, and $\log K_{s3} = 7.9$ were utilized from Wang's study. The calculated curve of the adsorption model could well fit the experimental data plots of 0.1 M H₂SO₄ solution in the pH range of less than 2. However, the experimental results exhibited smaller values than the calculated ones in the pH range from 2 to 4, indicating that another mechanism may also contribute to the returning phenomenon.

4.3. The possibility of formation of ferric arsenate in the H₂SO₄ solutions

Nishimura and Tozawa [31] measured and compared the solubility products of FeAsO₄, Ca₃(AsO₄)₂, and Mg₃(AsO₄)₂, and they concluded that the maximum rate of arsenic removal from wastewater was governed by the formation of FeAsO₄. According to the literature values, the solubility of FeAsO₄ drastically decreases in the pH range from 0 to 4. However, it is very hard to precisely predict the equilibrium arsenic concentrations at FeAsO₄ asturation because the reported solubility products of FeAsO₄ are widely scattered in the range from $10^{-19.9}$ to $10^{-24.6}$ [31–36].

In order to comprehend the degree of FeAsO₄ formation over the adsorption of arsenic acid species, further experiments have been conducted in the present work as described below. 100 g of Ash-F was washed thrice using 1 M of an HCl solvent with the liquid–solid ratio of 10 for 1 h, and rinsed thrice by 2 L of distilled water. Through this washing, it was preliminary confirmed that most of the leachable elements in Ash-F could be removed. Then, the washed Ash-F was dried for 2 days or longer. The test solutions with the pH of 0 and 3 were prepared by mixing of distilled water, 18 M of concentrated H₂SO₄, and Na₂HAsO₄·7H₂O reagent. The concentrations of Ash-F, and/or 100 μ L of 1000 mg L⁻¹ of iron(III) standard solutions were added into the prepared test solution with the following three cases:

Case 1: only Fe(III) solution was added. Case 2: only washed Ash-F was added. Case 3: both washed Ash-F and Fe(III) solution were added.

In Cases 1 and 3, the concentration of iron(III) ion in each solution was set to 10 or 100 mg L^{-1} . The mixtures were continuously agitated with a magnetic stirrer during the experiment. The sampling period was 10, 60, and 360 min. After the sampling, the pH of



Fig. 7. Changes in arsenic concentration of 2 types of test solutions with the addition of washed Ash-F and Fe(III) standard solution. The numbers in legend stand for Fe(III) concentrations in mg L⁻¹ in each test solution.

each solution was confirmed at 0 or 3.3. The obtained solutions by the sampling were filtrated and supplied for ICP-MS measurement.

The experimental results are shown in Fig. 7. The values of arsenic concentration in test solution with a pH of 0, no change in arsenic concentration could observe in any cases. In the runs at pH = 3, the addition of Fe(III) (Case 1) had no effect on the arsenic concentration. This fact strongly suggested that the formation of FeAsO₄ was not the main mechanism of returning phenomenon. On the other hand, a drastic decrease of arsenic concentration was observed by the addition of washed Ash-F into the test solution. In Case 2, the arsenic concentration in the test solution of pH = 3 quickly decreased after the addition of washed Ash-F, and approached to constant value of around 0.5 mg L⁻¹. This value was very close to the calculated arsenic concentration by adsorption model proposed by Wang et al. [30].

The important finding in this experiment was that the decrease in arsenic concentration in the solution at pH=3 was enhanced with the Fe(III) ion under the presence of fly ash particles (Case 3). Moreover, the rate of returning phenomenon was also accelerated under the presence of Fe(III) ion in the solution, while no remarkable difference was observed between 10 and 100 mg L⁻¹ of Fe(III). Taking these discussions into account, it was deduced that the difference between experimental arsenic concentration and the calculated one by adsorption model was due to the contribution of the formation of ferric arsenate surface complex [37].

4.4. Re-elution of arsenic from acid-washed fly ash

In the bench-scale test mentioned earlier, the pH of Ash-D solution at the stage of acid washing was around 2.5. Ash-C and D have higher CaO contents than Ash-A and B. Due to high CaO content, the leaching behavior of arsenic in Ash-C and D may be sorted by Group-II, and therefore, the adsorption of negative ionic species of arsenic acid would occur. When the washed fly ash samples were supplied for the Notification #18 test, the pH of test solution became around 10. It is deduced that almost all of adsorbed $H_2AsO_4^-$ may desorb from the surface of fly ash particles at these pH conditions and re-elute into the solution by Eq. (6):

$$S-H_2AsO_4 + OH^- = S-O^- + 2HAsO_4^{2-} + H_2O$$
(6)

In order to confirm the occurrence of this desorption reaction, the test solution containing 0.8 mg L^{-1} of arsenic at pH=4 was prepared, and Fe(III) standard solution and washed fly ash were charged into this solution. After 10 min of agitation, the concentration of arsenic in the solution was very low as expected. Then, NaOH solution was added to increase pH to around 10 and the concentration of arsenic was again measured. It was found that the concentration of arsenic in NaOH-added solution was returned to the initial level, indicating the adsorbed arsenic was released at higher pH of solution.

5. Modification for bench-scale acid washing process

In the previous section, we revealed that arsenic was mainly present as adsorbed S-H₂AsO₄ under the coexistence of coal fly ash particles in the pH range of 3-4 and became soluble HAsO₄²⁻ in the pH range of 9-10. This finding gives us an important indication that a pH range of 3-4 in the solution should be avoided in the final stage of acid washing process. Consequently, the acid washing process was conducted using Ash-B and highly concentrated H₂SO₄ (2 M) to keep the pH at sufficiently low level. Other conditions were similar to that described in the "bench-scale acid washing test" in Section 2. In this experiment, the pH of solution was kept below 0.2. The eluted arsenic concentrations by Notification #18 test from as-supplied Ash-B, washed Ash-B by conventional process, and washed Ash-B by modified process were 0.050, 0.017, and 0.010 mg L⁻¹, respectively. It is obvious that the elution concentration of arsenic from Ash-B decreased by washing with 2M of H₂SO₄, and therefore, the suppression of elution concentration of arsenic could be achieved by controlling the concentration of H₂SO₄ solvent.

The aim of acid washing process is to remove hazardous elements on the surface of coal fly ash particles by washing with diluted acid. After dehydration by centrifuge, caustic soda is added into eluted acid solution containing hazardous compositions for neutralization. The sludge, mainly composed of Na₂SO₄, is formed during the neutralization, and the hazardous elements are collected in the sludge. Collected sludge is dried, fired and finally sent to the controlled landfill site. Generation ratio of sludge is generally less than 10 kg per ton of supplied fly ash. The material balance and behavior of other trace elements such as selenium will be discussed in our next paper.

6. Conclusion

Laboratory- and bench-scale dissolution experiments were conducted to remove arsenic from several kinds of coal fly ash samples by acid washing to reveal the dissolution mechanism of arsenic and achieve sufficient removal of arsenic from coal fly ash samples. Most of the arsenic contents determined by Notification #19 dissolved rapidly in laboratory-scale experiments; however, we found that there was a decrease in arsenic concentration in H_2SO_4 solution with an increase in pH. The possibility of adsorption of arsenic acid species on the surface of coal fly ash particles was considerable in equilibrium condition of acid washing. Adsorbed arsenic acid species on the surface of coal fly ash particles was considered to desorb in an alkaline condition, and we succeeded in achieving sufficient removal of arsenic by ensuring that the pH of the slurry was not in the range of 3–4 in the practical phase leaching test.

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