Chapter

Development of the Phosphorus Recovery System (PRS) Utilizing Ultrasonic Wave in Incinerated Sewage Sludge Ash (ISSA)

Ye Duk Choi, Jun Yeon Lee, Zoo Ho Jang, Jung Gone Joung, Kyu Mun Han, Mok Young Lee and Kweon Jung

Abstract

This study was performed to develop a Phosphorus Recovery System(PRS) for the recovery of phosphorus from incinerated sewage sludge ash using struvite precipitation. Fly ash generated at the Seonam Sewage Treatment Plant(SSTP) has a high P_2O_5 content (13.9%). We developed a PRS consisting of an ultrasonic extractor, solid-liquid separator, mixing tank, and phosphorus recovery tank. The ultrasonic extractor had a 28 kHz vibrator for high speed and efficiency, which could perform the extraction in one-quarter of the time required in the conventional stirring method. Results of tests on the ultrasonic extractor showed that up to 0.044 g of P per gram of ash could be extracted with 1 N NaOH at an L/S ratio of 10 mL/g and an ultrasonic output of 500 Wh for 0.5 hr. The PRS is needed to improve the operation method and economic analysis to commercialize the technology and its application through further studies.

Keywords: phosphorus, recovery, ash, sludge, ultrasonic

1. Introduction

Incineration ash generated from sewage treatment facilities is known to contain a large amount of phosphorus, about 10% [1, 2]. Republic of Korea relies entirely on imports of phosphorus, which is referred to as the 3rd element of fertilizer, in the form of phosphate rock. Phosphate rock is expected to run out of resources within the next 50 to 100 years because of the increase in world population and demand for fertilizers [1, 3, 4].

Recently, due to the prohibition of dumping of sewage sludge at sea, the rate of onshore disposal is greatly increasing. Phosphorus recovery from incineration ash has many advantages in terms of improving economic efficiency and minimizing volume of t reaction tank through high concentration of phosphorus concentrated in incineration ash. Most of the conventional phosphorus recovery technologies eluted phosphorus from sewage sludge under anaerobic and high-temperature conditions, but commercialization of the technology was not easy due to high water content and

low phosphorus content. Water content of sludge in incineration facility is more than 80%, and percentage of organic matter is higher than 60%.

Looking at the previous research results, phosphorus recovery is mostly in the form of MAP (Struvite, $MgNH_4\ PO_4\ 6H_2O$ or $MgNH_4\ PO_4\ H_2O$) and HAP (Hydroxyapaptite, $Ca_5(PO_4)_3OH$) [5]. The principle of precipitation or crystallization is applied. Struvite can be expected to have a fertilizer effect because it contains essential elements necessary for plant growth, and excessive input is possible because it does not dissolve instantly due to the slow-release rate of nitrogen and phosphorus [6]. On the other hand, Struvite decomposes when heated and has high solubility in acidic conditions.

2. Materials and methods

Currently, the S sewage treatment facility incinerates about 150 ton/day of sewage waste by putting it into a fluidized bed incinerator at 800°C or higher. As an incineration by-product, incineration ash is generated at approximately 10 ton/day, and incineration ash is largely classified into fly ash and bottom ash. Particle size analysis was performed using a particle size analyzer (Sympatec GmbH, HELOS/HAER), and the chemical composition was analyzed with an X- ray fluorescence spectrometer (Rigaku, NEX-CG) and a scanning electron microscope (Shimadzu, SS-550). First, the L/S ratio (Leaching solution/Solid ratio, mL/g) was fixed at 10, which is the same as the experimental conditions suggested by other researchers [7]. fly ash 200 g and eluate 2 L was put into a standard Jar(2 L), and after stirring, solid-liquid separation was performed at 2000 rpm for 20 min using a centrifuge. The supernatant was analyzed. Heavy metals were prepared by pretreating the sample. Analyzed by ICP-OES (Varian, VISTA PRO) and PO_4^{3-} and SO_4^{2-} Anions such as were analyzed using IC(Metrohm, CH/Advanced IC) .

The phosphorus recovery process largely consists of two steps: phosphorus extraction and crystallization. First, the phosphorus extraction method is classified into an acid extraction method and an alkali extraction method, and the phosphorus crystallization step is MAP and HA depending on the input cation.

Phosphorus Recovery System(PRS), which was developed for the first time in Korea by applying the above phosphorus recovery principle, is made of acrylic

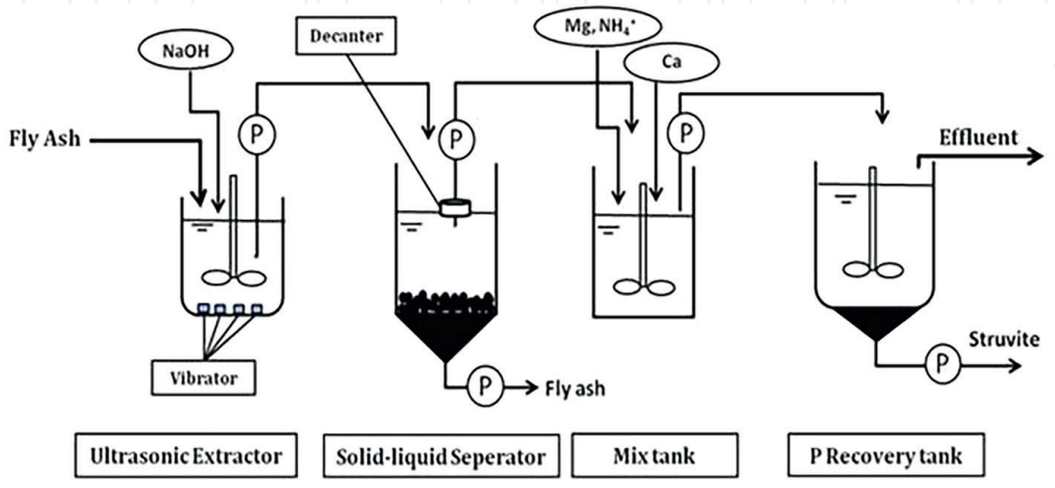

Figure 1.
Configuration of the PRS.

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material except for the ultrasonic extraction tank, and the ultrasonic extractor, Solid-Liquid Seperator, Mixing Tank, and P-Recovery Tank (**Figure 1**) .

Ultrasonic extraction tank that elutes phosphorus contained in the incineration ash is made of stainless material to prevent corrosion, and the fly ash and An agitator was installed to mix the eluate (**Figure 2**). In addition, by installing a cover on the top, evaporation of moisture due to temperature change during operation is minimized. The internal volume of the reactor is 48 L (0.4 m × 0.4 m × 0.3 m), and a total of 20 vibrators (4 × 5) with a frequency of 28 kHz are installed on the floor (**Figure 3**). The maximum ultrasonic power is 700 W, and the fly ash and of the eluate When mixing The L/S ratio (leaching solution/solid ratio, mL/g) was set to 10.

The solid-liquid separation tank has an inner diameter at the rear end of ultrasonic extraction tank. It is a cylinder with a height of 190 mm and a height of 500 mm and has an internal volume of 14 L. It separates the leached mixture of incineration ash. The remaining incineration ash is transferred to the hopper at the bottom by a pump, and the supernatant is transferred to the mixing tank through the Decanter located on the surface. It is a cylinder with a height of 150 mm and a height of 205 mm, and the

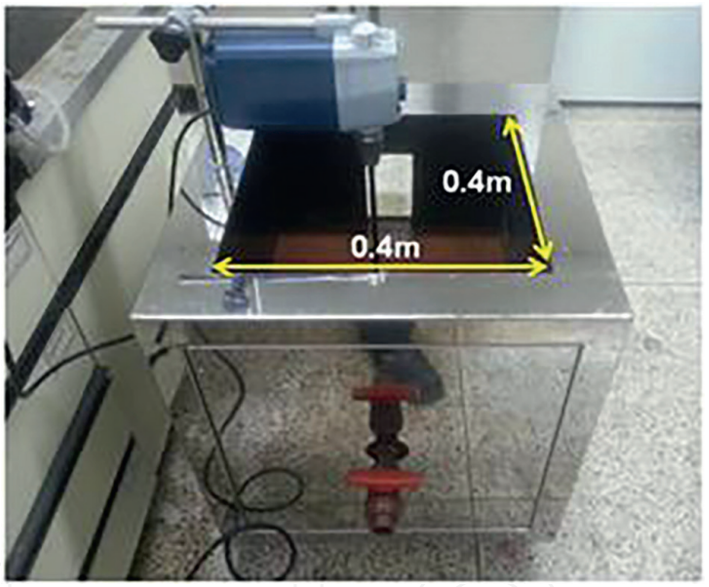

Figure 2.
Photo of ultrasonic extractor.

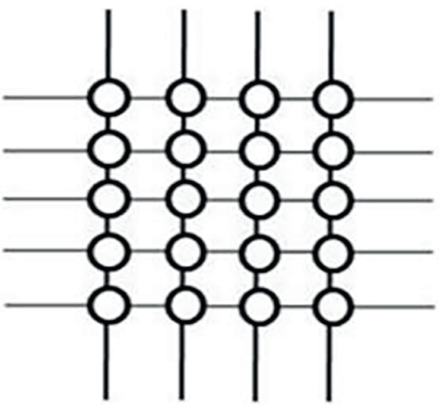

Figure 3.
Arrangement of vibrators.

Component	Volume(L)	Stirring velocity(rpm)	Remark
Ultrasonic Extractor	48.0	200	Vibrator(28 kHz)
Solid-Liquid Seperator	14.0	—	Decanter
Mixing Tank	3.6	150	—
P-Recovery tank	6.4	—	—

Table 1.
Specification of the PRS.

internal volume is 3.6 L, and an agitator is installed in the center for complete mixing. During MAP crystallization, 2 M $MgCl^{2+}$ and 2 M NH_4Cl were used as Mg^{2+} and NH_4^+ sources, respectively, and $CaCl_2$ was used as Ca^{2+} source during HAP crystallization. Phosphorus (P) recovery tank inner diameter It is a cylinder with a height of 190 mm and a height of 250 mm, and the internal volume is 6.4 L. In addition, it was designed so that phosphorus crystal (Struvite) precipitated at the bottom was transferred to the storage tank through a pump. The specifications of the P(phosphorus) recovery system (PRS) are shown in **Table 1**.

The recovered precipitate was analyzed using an X- ray diffractometer(XRD, Shimadzu, JP/XRD-7000) and an X- ray fluorescence analyzer (XRF, Rigaku, NEX-CG).

3. Result

3.1 Fly ash and bottom ash

3.1.1 Particle size

Ash generated from sewage treatment facilities is largely divided into fly ash and bottom ash, and in particular, fly ash accounts for more than 97%. Fly ash and bottom ash generated from the incinerator were collected and the distribution by particle size was analyzed with a particle size analyzer.

Figures 4 and **5** shows the particle size distribution of fly ash and bottom ash, respectively. X_{50} is Fly ash and bottom ash were 31.02 μm and 42.61 μm, respectively,

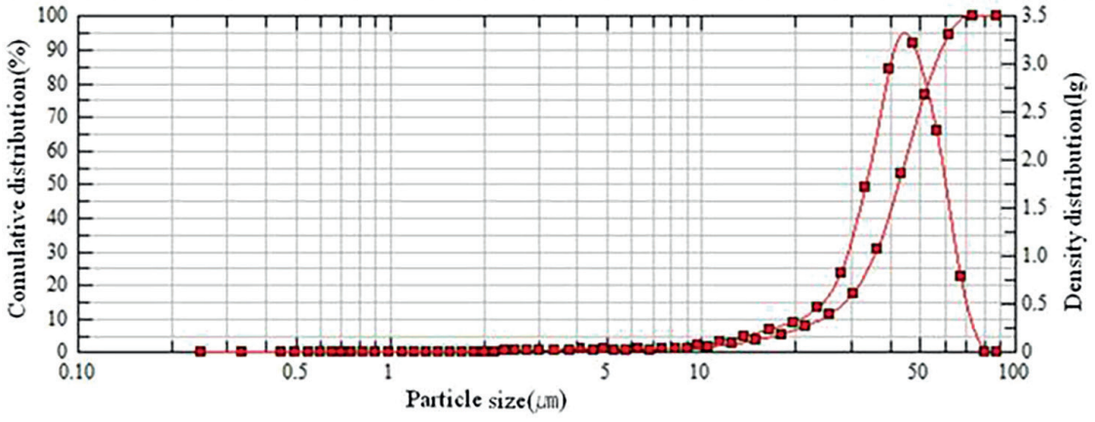

Figure 4.
Grain size distribution of fly ash.

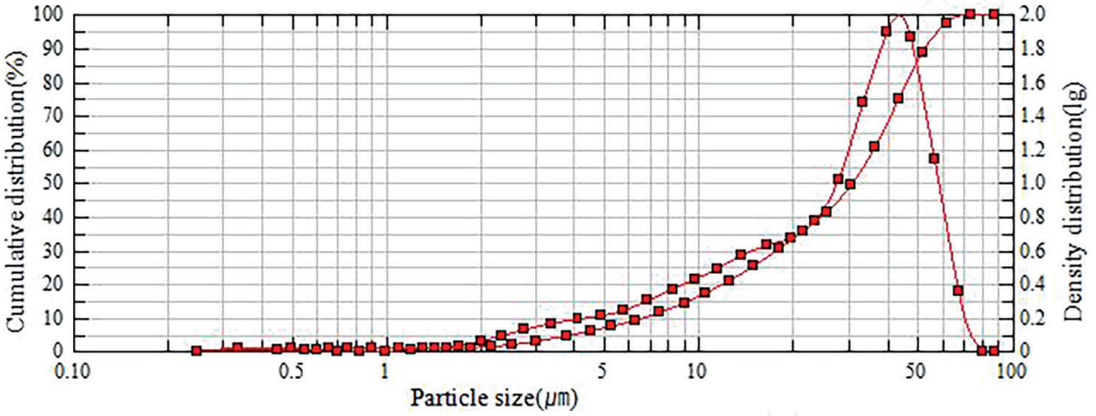

Figure 5.
Grain size distribution of bottom ash.

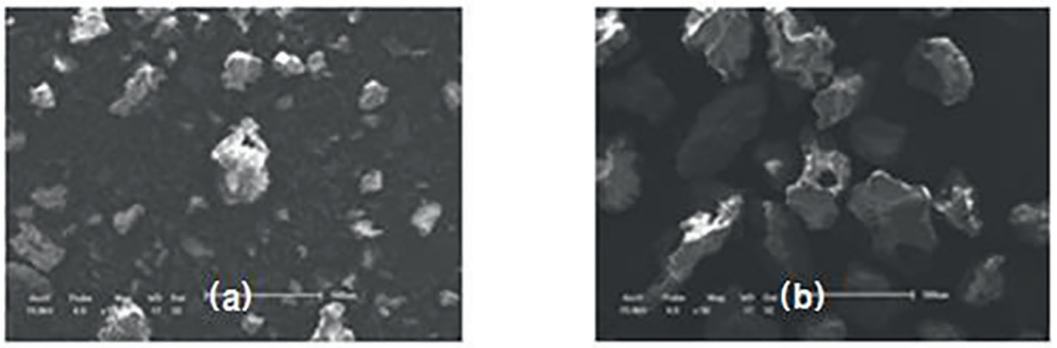

Figure 6.
SEM photographs (×50) of ash; a) Fly ash, b) bottom ash.

and a large number of relatively small particles were distributed in the fly ash. In addition, as a result of specific surface area (Sv) analysis, fly ash and bottom ash were 0.46 m^2/cm^3 respectively and 0.17 m^2/cm^3, the specific surface area of the fly ash was more than 2.5 times larger. Considering the experimental results of Roy [8] and the amount of incinerated ash, fly ash is judged to be more suitable for P elution. Phosphorus (P) contained in incineration ash is mostly in the form of Whitlockite and is known to be thermally stable and not volatilized even at high temperatures of 800 to 900°C [9].

Figure 6 is a photograph (magnification ratio: 50 times) of the surface of fly ash and incineration ash using a Scanning Electron Microscope (SEM), and it was confirmed that a large number of relatively large particles were distributed in the bottom ash.

3.1.2 Chemical composition

Incineration ash, a by-product of incineration, is mostly emitted in the form of metal oxides along with combustion gases. **Table 2** shows the test results of fly ash and bottom ash analyzed using X- ray fluorescence spectrometry (XRF). First of all, the P_2O_5 of the fly ash was 13.65 ± 2.35%, indicating that it contained more P than the bottom ash (10.23 ± 2.38%). Merino et al. [10] reported that P_2O_5 of sewage sludge incineration ash in Spain was 14.2%, and Donatello et al. [11] reported P_2O_5 of incineration ash as 14.8%. Fly ash is also believed to contain similar levels of

Element	Fly ash	Bottom ash
P^2O^5	13.65 ± 2.35	10.23 ± 2.38
Al_2O_3	16.90 ± 1.19	11.50 ± 1.90
SiO_2	24.15 ± 2.95	13.75 ± 2.45
Fe_2O_3	19.40 ± 2.60	42.45 ± 9.45
CaO	16.45 ± 1.55	12.73 ± 5.38
MgO	3.40 ± 0.27	3.83 ± 0.61
K_2O	3.31 ± 0.35	2.49 ± 1.34
ZnO	0.64 ± 0.31	1.04 ± 0.71
MnO	0.24 ± 0.04	0.29 ± 0.23
TiO_2	1.32 ± 0.20	0.95 ± 0.53
Na_2O	1.12 ± 0.19	0.58 ± 0.58
CuO	0.60 ± 0.33	0.72 ± 0.72

Table 2.
Chemical composition of ash by XRF. (unit: %).

phosphorus. In addition, Al_2O_3 of fly ash was 16.90 ± 1.19%, and Al_2O_3 of bottom ash was 11.50 ± 1.90%, indicating that fly ash contained more Al. According to literature, Donatello et al. [11] reported 13.1% Al_2O_3 in incineration ash, and Adam et al. [12] reported 10.8% Al_2O_3 in incineration ash. P contained in incineration ash is mostly a Whitlockite type($Ca_3(PO_4)_2$) orthophosphate.

Incineration ash As a result of analysis by X- ray fluorescence spectrometry (XRF), the P_2O_5 of the fly ash was 13.65 ± 2.35% and contained more phosphorus (P) than the bottom ash (10.23 ± 2.38%). In addition, MgO, a component of MAP precipitation, was 3.40 ± 0.27% and 3.83 ± 0.61% for fly ash and bottom ash, respectively, and there was no significant difference.

SiO_2(24.15 ± 2.95%) was the oxide that occupied the largest proportion in fly ash, which was attributed to the large amount of soil contained in sewage sludge. SiO_2 in incineration ash reported by other researchers Looking at the content, Huacheng Xu et al. [13] and Donatello et al. [11] reported that SiO_2 was the most present in incineration ash, with 43.1 ~ 49.1% and 31.6%, respectively. On the other hand, Fe_2O_3 in the bottom ash accounted for the largest proportion at 42.45 ± 9.45% due to the iron-based coagulant mainly used in the return water treatment process of the S sewage treatment facility. Comparing the CaO content, fly ash was 16.45 ± 1.55% and bottom ash was 12.73 ± 5.38%, similar to the 15.7% CaO content reported by Adam et al. [12].

3.2 Jar-test

3.2.1 Eluted phosphorus

P elution efficiency was compared by adding sulfuric acid and sodium hydroxide to the fly ash. For the Jar-test, 0.4 N and 1 N sulfuric acid solutions and sodium hydroxide solutions were prepared as extraction liquids, and the stirring time was It was set to 120 min. **Figure 7** shows P eluted from 1 N sulfuric acid solution and sodium hydroxide solution.

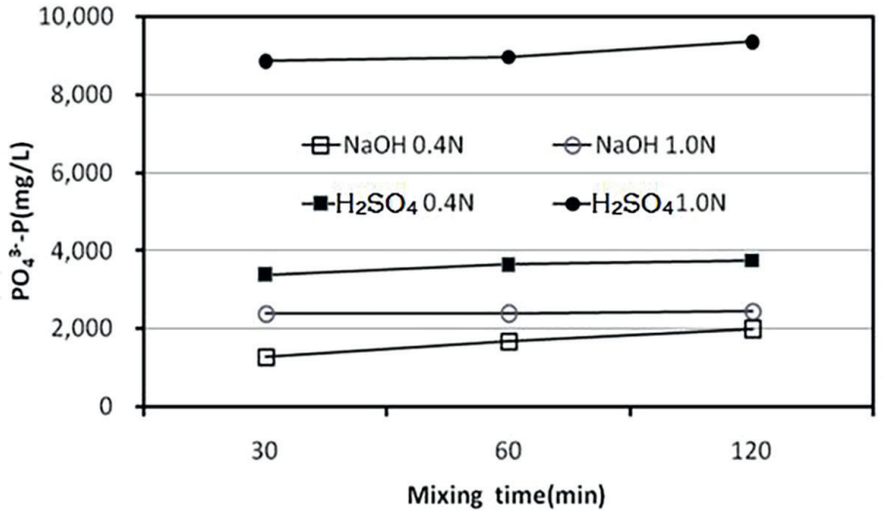

Figure 7.
Comparison of H_2SO_4 and NaOH as extraction liquid.

1 N sulfuric acid and 1 N sodium hydroxide were used, the eluted P was 9362.2 mg/L and 2462.7 mg/L, respectively, and the elution efficiency of sulfuric acid was about 3.8 times higher than that of sodium hydroxide. Through the above experimental results, when using sulfuric acid The maximum recovery of phosphorus that can be eluted is It was more than 0.094 g of PO_4^{3-}-P/g ash. P eluted when 0.4 N sulfuric acid and 0.4 N sodium hydroxide were used under the same conditions were 3747.4 mg/L and 1986.1 mg/L, respectively, and P eluted when 1 N sulfuric acid was used as the eluent. P increased 2.5 times in proportion to 0.4 N sulfuric acid. Biswas, B et al. reported that when sodium hydroxide is used for phosphorus elution from incineration ash, about 40% of phosphorus elution is possible compared to acid [14].

3.2.2 Stirring speed

Figure 8 shows P eluted at stirring speeds of 150 rpm and 200 rpm when 1 N sulfuric acid solution was used as the eluent. In order to select the optimal stirring speed for elution of incineration ash, It was fixed at 120 min. Stirring speed PO_4^{3-}-P

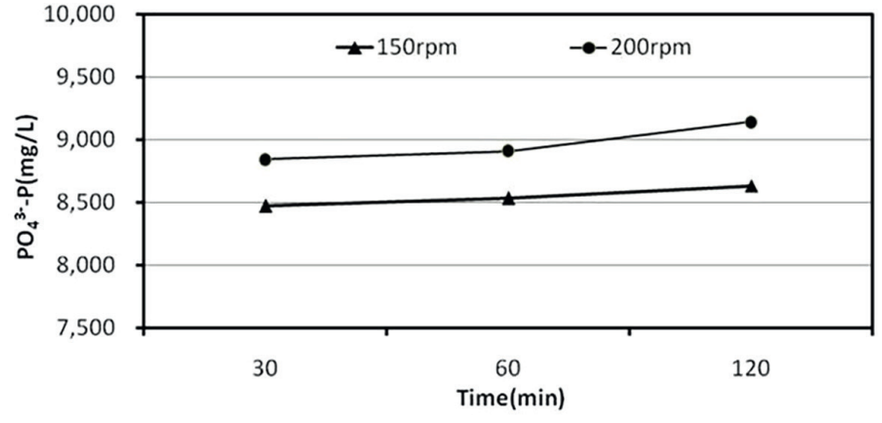

Figure 8.
Variation of PO_4^{3-}-P with different stirring speed.

eluted at 200 rpm was 9141.0 mg/L, showing stirring speed It was slightly higher than P(8,634.9 mg/L) eluted at 150 rpm.

Based on the above experimental results, the phosphorus elution efficiency was determined by the stirring speed. At 200 rpm, Agitation speed through the superiority of contact area and the number of collisions It is judged to be better than 150 rpm.

3.2.3 Heavy metal

When using 1 N sulfuric acid solution Al, Ca and Mg concentrations are shown in **Figure 9**. After 120 minutes, the Al concentration was 2790.1 mg/L and Mg concentration was 1781.4 mg/L. Al and Mg concentrations increased with stirring time, but Ca concentration continuously decreased from 2404.1 mg/L(5 min) to 1211.3 mg/L(120 min).

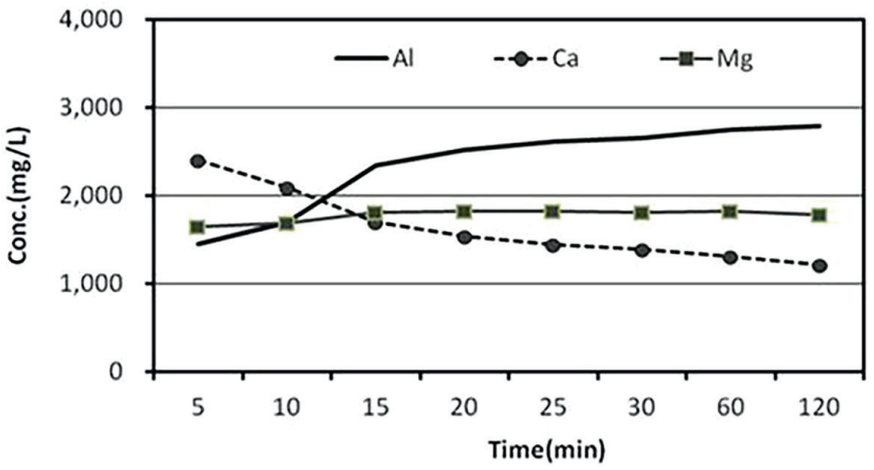

Figure 9.
Variations of heavy metals with H_2SO_4.

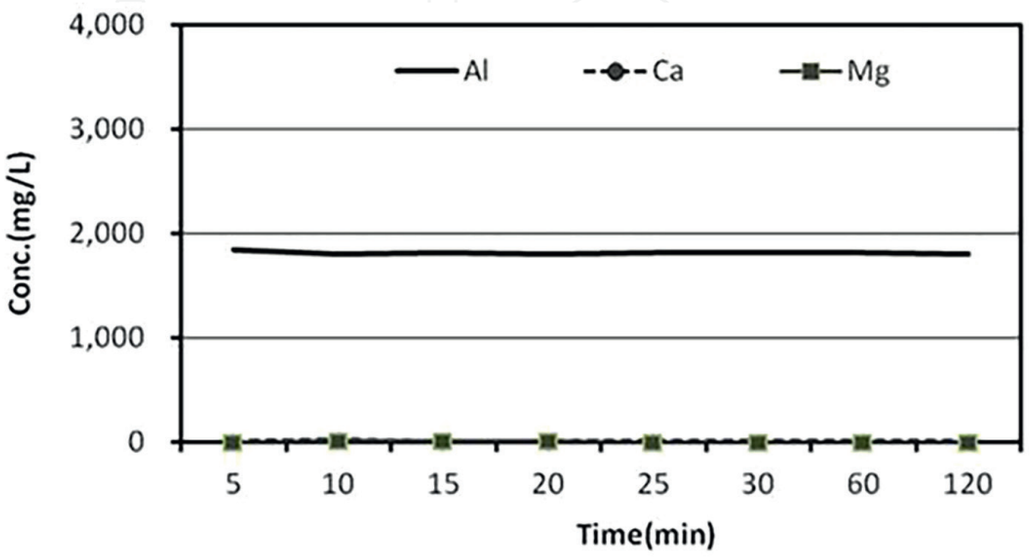

Figure 10.
Variations of heavy metals with NaOH.

This phenomenon is thought to be due to chemical precipitation of sulfate and calcium contained in the eluate as calcium sulfate ($CaSO_4$). In addition, K and Na concentrations were produced below 400 mg/L, and As, Cr, Cd, Pb and Se were detected below the limit of quantification.

On the other hand, when using 1 N sodium hydroxide solution Al, Ca and Mg concentrations are shown in **Figure 10**. The Al concentration appeared constant at a level of 1800 mg/L, and Mg and Ca concentrations were maintained at a low concentration of 20 mg/L or less compared to when sulfuric acid was used as an eluent. Schaum et al. argued that phosphorus extraction efficiency is as low as 30% or less, but heavy metal elution is low when phosphorus is separated by applying alkali extraction method from sewage sludge incineration ash [15]. When sulfuric acid is used, phosphorus elution efficiency is excellent, but a large amount of other heavy metals are generated as by-products, and phosphorus crystallization. It is necessary to select an effective phosphorus recovery method in consideration of the process of adjusting above pH 9.5 and recovery cost.

3.3 Operating result of PRS

3.3.1 Phosphorus

Figure 11 shows PO_4^{3-}-P according to ultrasonic power in 1 N sodium hydroxide. Same as 12. L: S ratio of 10 without ultrasonic irradiation When stirred for 30 min, PO_4^{3-}-P was 2117.7 mg/L, and when ultrasonic waves were irradiated at 100 W, PO_4^{3-}-P was 3194.8 mg/L at same stirring time during ultrasonic irradiation.

It was found that the elution concentration of phosphorus increased by more than 50%. when ultrasonic power was increased to 500 W, PO_4^{3-}-P was 4104.6 mg/L, indicating that phosphorus elution amount Improved over 94%. This is judged to be the result of an increase in the mass transfer rate of phosphorus adsorbed on fly ash or bonded in the form of a compound by the shear force of ultrasonic waves. The reason why the phosphorus elution concentration is high according to ultrasonic output after

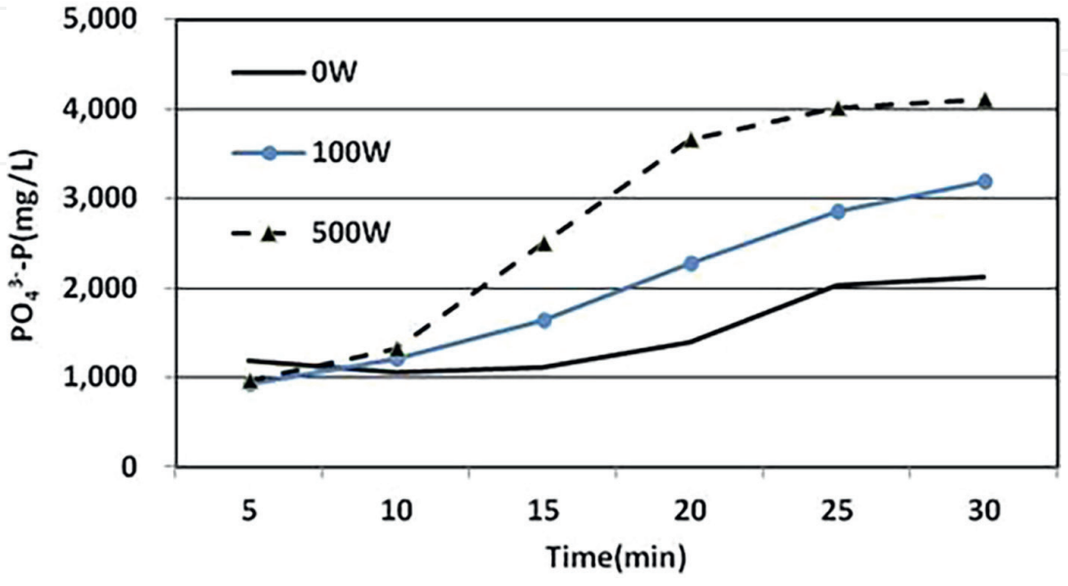

Figure 11.
Variations of PO_4^{3-}-P at different ultrasonic power with NaOH.

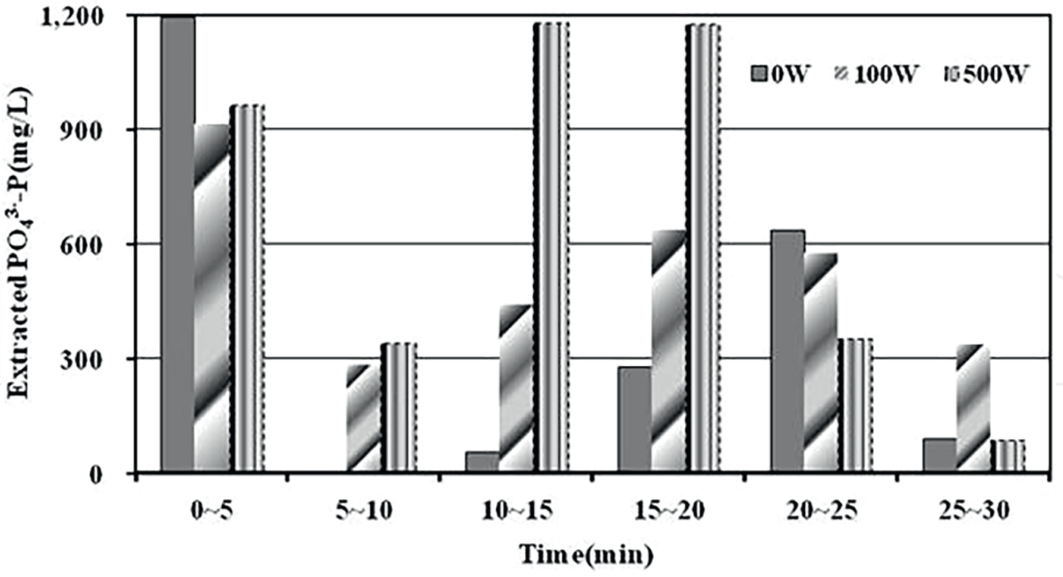

Figure 12.
Extracted PO_4^{3-}-P at different ultrasonic power with NaOH.

ultrasonic irradiation time of 10 min is that the P adsorbed on the surface of the fly ash is easily separated in the beginning, but P bonded in the form of P compound is thought to be because elution is possible through a strong shear force such as ultrasound and long exposure.

The amount of PO_4^{3-}-P dissolution by time according to the ultrasonic output was **Figure 12**. In the 0–5 min section, P adsorbed on the surface of the fly ash is easily separated, and it is found that a lot of P(phosphorus) over 900 mg/L (0.9 g P/100 g Ash) is eluted. On the other hand, ultrasonic waves were irradiated at 500 W, phosphorus elution amount It was significantly higher than 1100 mg/L (1.1 g P/100 g Ash) in the intervals of 10 ~ 15 min and 15 ~ 20 min.

3.3.2 Dissolution rate

The dissolution rate of PO_4^{3-}-P per unit time according to the ultrasonic power in 1 N sodium hydroxide is shown in **Figure 13**. When stirring for 5 min without ultrasonic irradiation, PO_4^{3-}-P dissolution rate was 238.4 mg/L-min, and when the ultrasonic wave was irradiated at 100 W, PO_4^{3-}-P dissolution rate was 183.2 mg/L-min. Appeared as On the other hand, when ultrasonic waves were not irradiated (0 W), the dissolution rate decreased to less than 100 mg/L-min, but when ultrasonic waves were irradiated at 100 W, it was maintained constant at 100 mg/L-min.

Compared to when ultrasonic waves were not irradiated, P elution concentrations increased by 2.1 times and 2.7 times when ultrasonic power was irradiated with 100 W and 500 W, respectively. The effect was found to be large. Based on these results, it is judged that the phosphorus elution effect can be maximized if the operating conditions are derived in consideration of the phosphorus elution amount and the ultrasonic irradiation cost when sodium hydroxide is used for phosphorus recovery from fly ash.

3.3.3 Heavy metal

The concentration of heavy metals eluted from 1 N sodium hydroxide according to the ultrasonic power is shown in **Figure 14**. Without ultrasound irradiation When

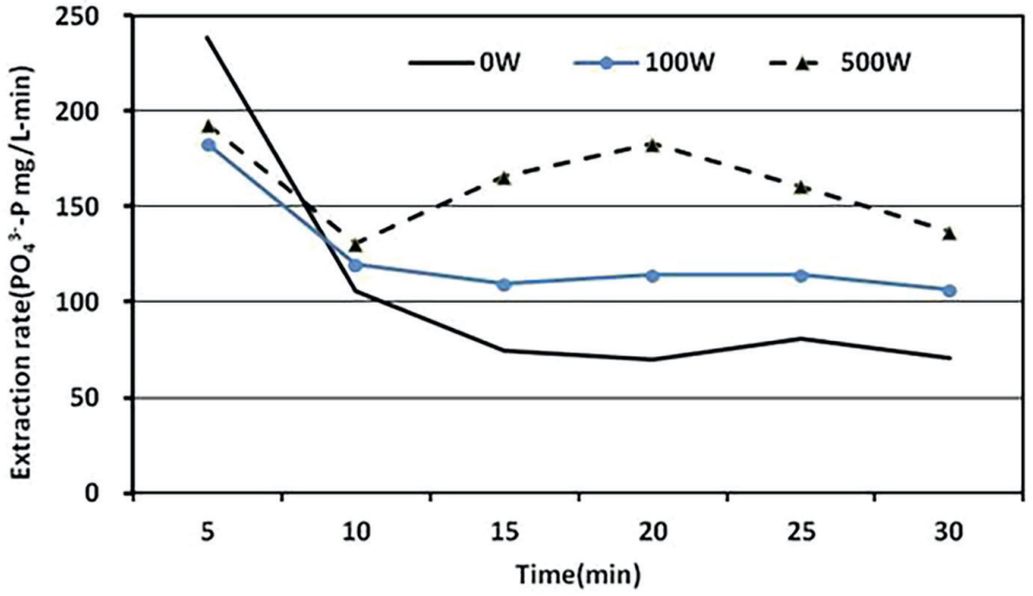

Figure 13.
Variations of extraction rate at different ultrasonic power with NaOH.

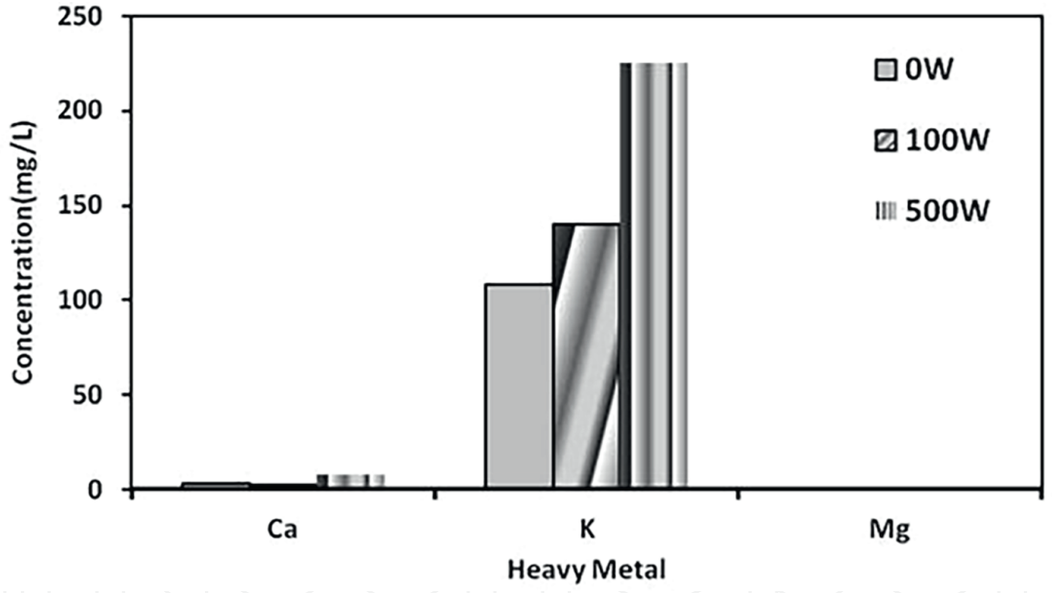

Figure 14.
Variations of heavy metals at different ultrasonic power with NaOH.

stirring for 5 min (0 W), Ca concentration was 3.5 mg/L, and when ultrasonic waves were irradiated at 100 W, the Ca concentration was 2.6 mg/L. Also, the Mg concentration was measured without ultrasonic irradiation.

When stirred for 5 min and when irradiated with ultrasonic waves at 100 W, 0.2 mg/L and non-detection were found, respectively, indicating that the Ca and Mg concentrations were insignificantly affected by ultrasonic irradiation. On the other hand, the K concentration was measured without ultrasonic irradiation. Concentrations were 108.5 mg/L and 224.9 mg/L, respectively, when stirring and when ultrasonic waves were irradiated at 500 W, confirming that the K concentration increased as the ultrasonic power increased.

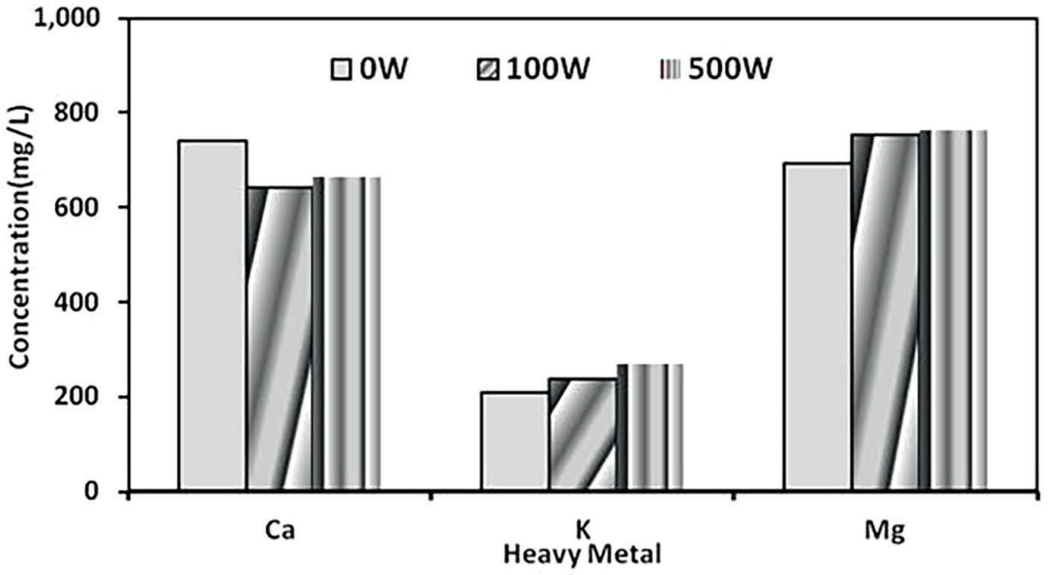

Figure 15.
Variations of heavy metals at different ultrasonic power with H_2SO_4.

The heavy metal concentration according to the ultrasonic power in 1 N sulfuric acid is shown in **Figure 15**. Mg concentration was measured without ultrasonic irradiation. When stirring and ultrasonic irradiation at 100 W were 691.8 mg/L and 754.9 mg/L, respectively, effect of ultrasonic irradiation was not significant. On the other hand K without ultrasonic irradiation When stirred and when irradiated with 500 W of ultrasonic waves, it was 209.7 mg/L and 269.0 mg/L, respectively, and the K concentration slightly increased with the increase in ultrasonic power. On the other hand, Ca and Mg concentrations showed a significant increase in heavy metal elution concentration in sulfuric acid eluate compared to sodium hydroxide.

When sulfuric acid is used as eluent, Cu concentration more than 10 times higher than that of sodium hydroxide is detected. It is expected that the purity of struvite will be lowered or secondary contamination will occur. On the other hand, when sodium hydroxide was used, As, Ba, Cd, and Ni were not detected, and Cr, Pb, Se, and Zn concentrations were detected at low concentrations of less than 1 mg/L. When sulfuric acid is used as an eluent, phosphorus elution efficiency is high, but heavy metals such as Cu, Zn, and As are eluted at high concentrations, causing other side reactions.

3.3.4 Optimization of operating condition

In order to determine the elution conditions of P in fly ash, an experiment was conducted by setting the stirring time and irradiation time to 3 hr. On the other hand, without ultrasound Stirring time when only stirring at 120 min, PO_4^{3-}-P was 4360.3 mg/L, compared to P(4,402.0 mg/L) eluted at 30 min at 500 W ultrasonic irradiation. It could be drastically reduced to 1/4. Considering this fact, fly ash When irradiated with 500 W ultrasound for 30 min in 1 N sodium hydroxide, P elution of up to 0.044 g P/g ash is possible. Compared to experimental results of recovering more than 0.094 g of P/g ash in 1 N sulfuric acid, about 46.8% or more of phosphorus

Element	Content(%)	Element	Content(%)
Mg	47.84	Si	3.9
P	22.6	Fe	1.2
Al	17.5	K	0.5
S	4.0	Zn	0.1

Table 3.
Main chemical composition of precipitated struvite by XRF.

contained in incineration ash can be recovered by using ultrasonic waves in 1 N sodium hydroxide. Based on the above experimental results, the optimal operating condition of ultrasonic elution tank is to minimize the concentration of harmful heavy metals and recovery cost when irradiating for 30 min at ultrasonic output of 500 W using 1 N sodium hydroxide at L/S ratio of 10.

3.4 Phosphorus crystallization analysis

Table 3 shows the results of X-ray Fluorescence Spectrometry (XRF) analysis of recovered struvite. Main components Mg and P were 47.84% and 22.6%, respectively, and Si and Fe were 3.9% and 1.2%. Struvite recovered through the above analysis results contains a large amount of Mg and P at more than 70%, so it is expected that it can be used as a fertilizer when cultivating crops. In addition it was confirmed through X-ray Diffractometer (XRD) analysis that Mg and P are the main components constituting phosphorus crystallization, considering the position of the main peak appearing in struvite.

4. Conclusion

1. Fly ash included in the incineration ash generated from the S sewage treatment facility was suitable for phosphorus recovery due to its large amount, specific surface area and high P_2O_5 content.

2. Phosphorus Recovery System(PRS) consisting of an ultrasonic extraction tank, a solid-liquid separation tank, a mixing tank, and a P recovery tank was developed to recover phosphorus from sewage sludge incineration ash.

3. As a result of operated ultrasonic extraction tank, compared to the existing agitation method, P(phosphorus) Separation time can be drastically reduced by 1/4, and phosphorus elution of up to 0.044 g P/g ash is possible when irradiating 500 W ultrasonic waves for 30 min in L/S ratio 10 and 1 N NaOH solution.

4. Phosphorus from incineration ash recognized as waste It is expected that the recovery will contribute to the expectation of import substitution effect of phosphate rock facing the crisis of resource depletion.

4.1 Discussion

When recovering phosphorus from incineration ash, the biggest problem is considered to be economic feasibility. It is still difficult to secure economic feasibility because the recovery cost is higher than that of phosphate rock. However, natural resources are finite and resource recovery technology needs to be developed to prepare for resource depletion. In particular, when recovering phosphorus from incineration ash, it is possible under acidic and basic conditions.

Although acidic conditions are favorable for phosphorus extraction efficiency from incineration ash, there is a disadvantage in that the purity of phosphorus crystallization is low due to the elution of many other heavy metals. In addition, the process is complicated because a separate pH control is required under acidic conditions. On the other hand, in basic conditions, phosphorus extraction efficiency is low, but phosphorus crystallization with high purity can be obtained because the elution concentration of heavy metals is low. In the future, considering these problems, it is judged that the phosphorus recovery method should be applied according to the site conditions of the sewage treatment plant and the e of phosphorus crystal (MAP or HAP).

Author details

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