

Phosphorus Recovery from Charcoal of Sewage Sludge by Incineration Treatment

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Abstract: The phosphorus recovery from charcoal of sewage sludge by an incineration method was investigated. The charcoal of sewage sludge was mixed with alkali metal hydroxide and incinerated at 750 °C in aerobic conditions. The phosphorus was successfully recovered as an alkali metal phosphate from the incinerated ash through water extraction. The recovery rate of the phosphorus reached about 75% to 80%. The appropriate condition of the phosphorus recovery and composition of the recovered phosphorus were investigated.

Key words: Sewage sludge, sodium hydroxide, potassium hydroxide, phosphorus recovery.

1. Introduction

Sewage sludge contains significant amounts of phosphorus, in order to recover phosphorus, some methods are investigated with the incinerated ash or charcoal of the sludge using acid [1] or alkali [2]. The phosphorus in the sludge is considered to exist in many forms of phosphate compounds (AlPO_4 , FePO_4 , $\text{Ca}_3(\text{PO}_4)_2$, etc.). In an acidic treatment, almost all of the phosphorus can be extracted, however, the recovered phosphorus contains high amount of aluminum [3-5] which is considered to be inconvenient for the usage of the recovered phosphorus. The alkali treatment can possibly recover the phosphorus of the low content of aluminum, but the extraction rate is low because of low solubility of the $\text{Ca}_3(\text{PO}_4)_2$ component of the sludge in alkali conditions [6]. In order to obtain the high extraction rate through the alkali treatment, phosphorus extraction at high temperatures is regarded to be effective [7, 8]. We investigated phosphorus recovery from the charcoal of sewage sludge using NaOH or KOH through the incineration of the charcoal [9].

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2. Method

2.1 Recovery Method

The charcoal of sewage sludge made by heat treatment at 850 °C in anaerobic condition, was used. As alkali materials, NaOH or KOH was used. The aqueous solution of the alkali was mixed with the raw charcoal, and incinerated at 750 °C for 1 hour after drying at 105 °C. Later, in order to extract the alkali metal phosphate which is formed through the incineration, washing water was added to the incinerated mixture (ash) with S/L (solid/liquid) ratio 1:10, and filtrated using filter paper (Toyo Roshi Kaisha, LTD. Advantec). The phosphorus was recovered from the filtrate by drying at 105 °C as shown in Fig. 1.

2.2 The Alkali Addition Rate

In order to decide a proper addition rate of the alkali, 5 g of the raw charcoal was mixed with some concentrations of the aqueous solution of NaOH or KOH. The mixture was dried at 105 °C, and incinerated at 750 °C in aerobic condition. In order to extract the phosphorus, water was added to the incinerated mixture, and was filtrated using filter

paper. The residue was dried at 105 °C, and the chemical composition was examined using an X-ray analyzer (Rigaku Cooperation SPECTRO XEPOS). The phosphorus content of the residue was decreased by the extraction of the phosphorus, and the decreased amount of the phosphorus tended to be increased with the addition rate of the alkali (Fig. 2). Contrarily, the component of Na_2O was increased by the addition of the alkali. In other components (like SiO_2 , Al_2O_3 ,

Fe_2O_3), a significant change of the composition was not found through the treatment. The degreasing rate of the phosphorus tends to plateau when the addition rate reached 2 g (in the case of NaOH, shown in Fig. 3) or 3 g (in the case of KOH, shown in Fig 4). From the result, we decided the best addition rate of the alkali for the phosphorus recovery was as follows.

The addition rate: 5 g of the charcoal to 2.5 g of NaOH 2.5 g or 3 g of KOH.

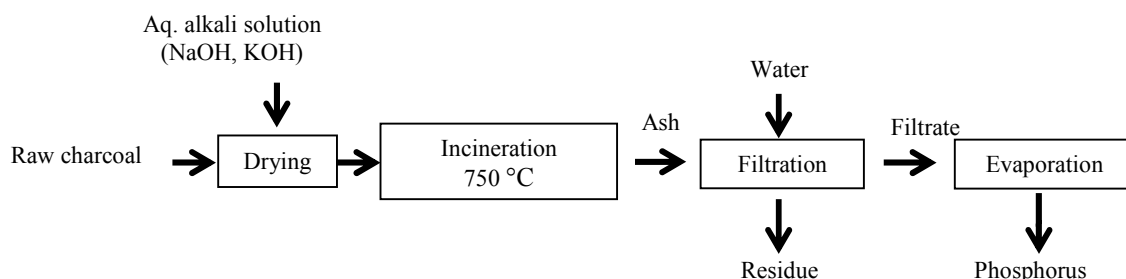


Fig. 1 Recovery method.

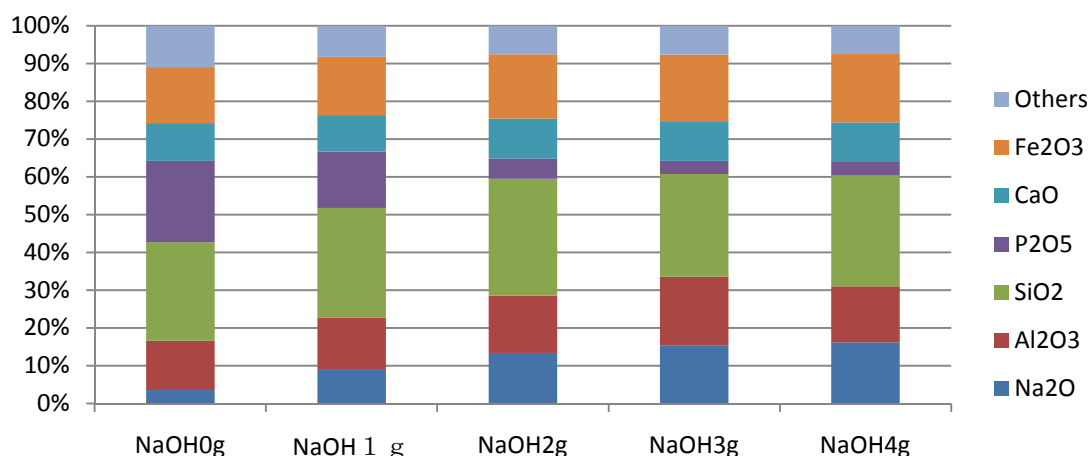


Fig. 2 Chemical composition of the residue.

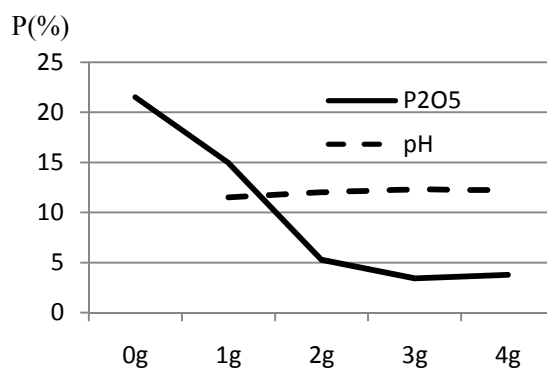


Fig. 3 The relation between the amount of phosphorus in the residue and the NaOH addition rate.

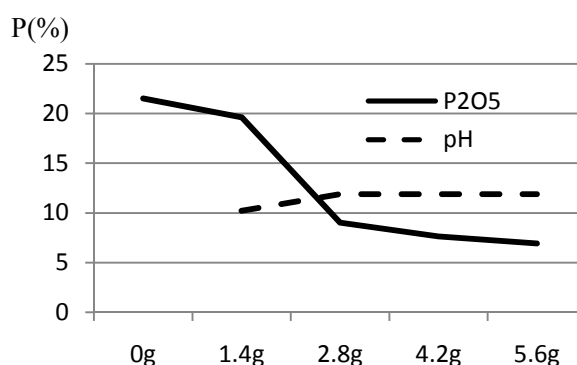


Fig. 4 The relation between the amount of phosphorus in the residue and the KOH addition rate.

3. Results and Discussion

In order to confirm the phosphorus recovery, the aq. solution of NaOH (10 g of NaOH was mixed with 17 mL of water) was added to the 20 g of raw charcoal, and incinerated after drying. Later, 200 mL of water was added to the incinerated mixture, and was filtrated using filter paper. The pH of the filtrate reached about 12. The possibility of Cr^{6+} formations through incineration or heat treatment is reported [10, 11]. In order to confirm the formation of the Cr^{6+} through this treatment, concentration of the Cr^{6+} was checked by a simple analysis (using PACKTEST, KYORITSU Co. Ltd), 2 mg/L of the Cr^{6+} was detected. The filtrate and residue were dried for analysis at 105 °C, and recovered.

The same experiment was carried out using 20 g of the charcoal and 15 g of KOH. The small amount of Cr^{6+} (0.5 mg/L) was found in the filtrate. In the case of KOH, the addition rate of the KOH was expected to be high compared with the amount of the phosphorus in the raw charcoal, and excesses of KOH in the recovered phosphorus absorbed the humidity in the air, resulting in the difficult condition of the analysis of the phosphorus. To remove the excess KOH, neutralization using 4 mL HCl was carried out, and later, dried at 105 °C. The amounts of recovered materials are shown in Table 1 (the amount of added HCl is not included).

These recovered materials were analyzed using an X-ray analyzer (as shown in Fig. 5). Almost all of

phosphorus was removed from the charcoal, and recovery rate was considered 80% (in the case of NaOH) or 75%. (in the case of KOH) from the phosphorus composition, compared with the ash of the raw charcoal and the residue.

The recovered phosphorus is considered to be mainly sodium phosphate or potassium phosphate from the chemical composition, and contains small amounts of aluminum or silicon compounds.

The ash of the raw charcoals is composed of many components (P_2O_5 , SiO_2 , Fe_2O_3 , CaO and Al_2O_3), and from the result of the X-ray analysis, these components are considered to exist in a form of FePO_4 , $\text{Ca}_3(\text{PO}_4)_2$, $\text{Mg}_3(\text{PO}_4)_2$, SiO_2 by the X-ray diffraction pattern (using XRD, Rigaku Co.) shown in Fig. 6. Significant amount of alkali metal components (Na_2O or K_2O), was contained in the residue, and these compounds are mainly to be a form of sodium aluminum silicate (Fig. 7) or potassium aluminum silicate (Fig. 8) by the X-ray diffraction pattern. Almost the same amount of the residue compared with the raw charcoal will be discharged, therefore, finding the usage is a very important matter. The alkali metal aluminum silicate contained in the residue has a chemical composition like zeolite [12], and it is expected to have an ability like zeolite, and further investigation will be needed in order to find a usage for them. The other matter, formation of the Cr^{6+} compounds was found, and elimination of them is necessary.

Table 1 The amounts of the addition rate and recovered materials.

	Addition rate of the alkali	Amount of the incinerated (treated) ash	Amount of the residue	Amount of the recovered phosphorus
NaOH	10 g	19.8 g	11.6 g	8.2 g
KOH	16 g	24.4 g	12.3 g	12.1 g

The amount of the raw charcoal: 20 g.

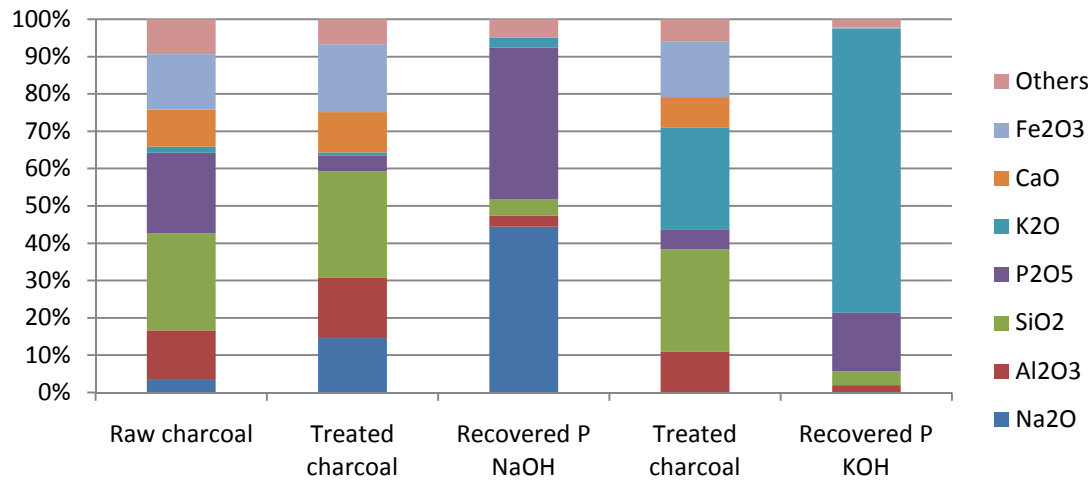


Fig. 5 The composition of the recovered materials.

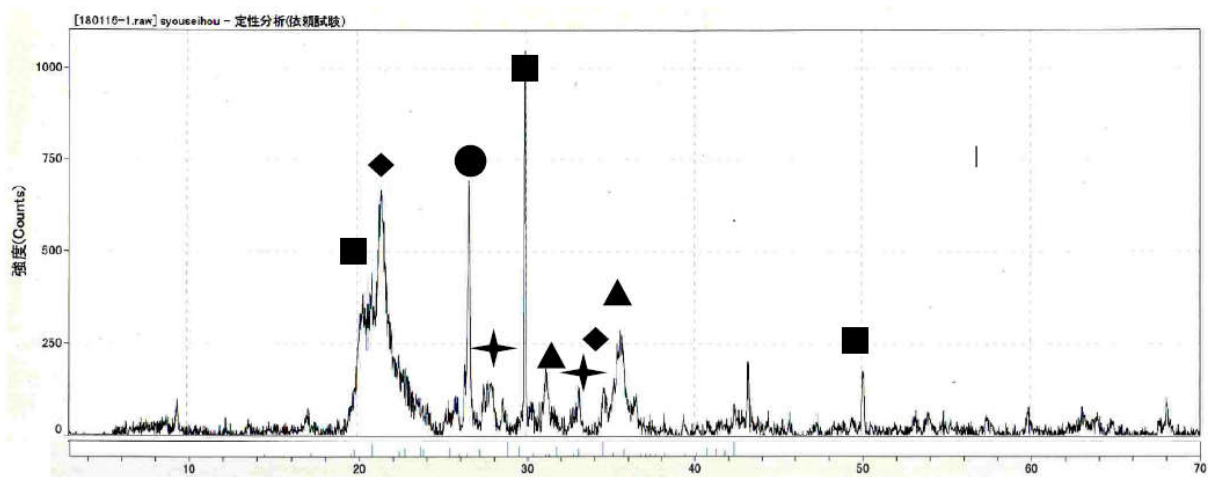


Fig. 6 X-ray diffraction patter of the ash of raw charcoal.

Source: ●: SiO₂; ▲: Fe₂O₃; ■: Mg₃(PO₄)₂; ◆: Sodium aluminum silicate; ✦: Ca₃(PO₄)₂.

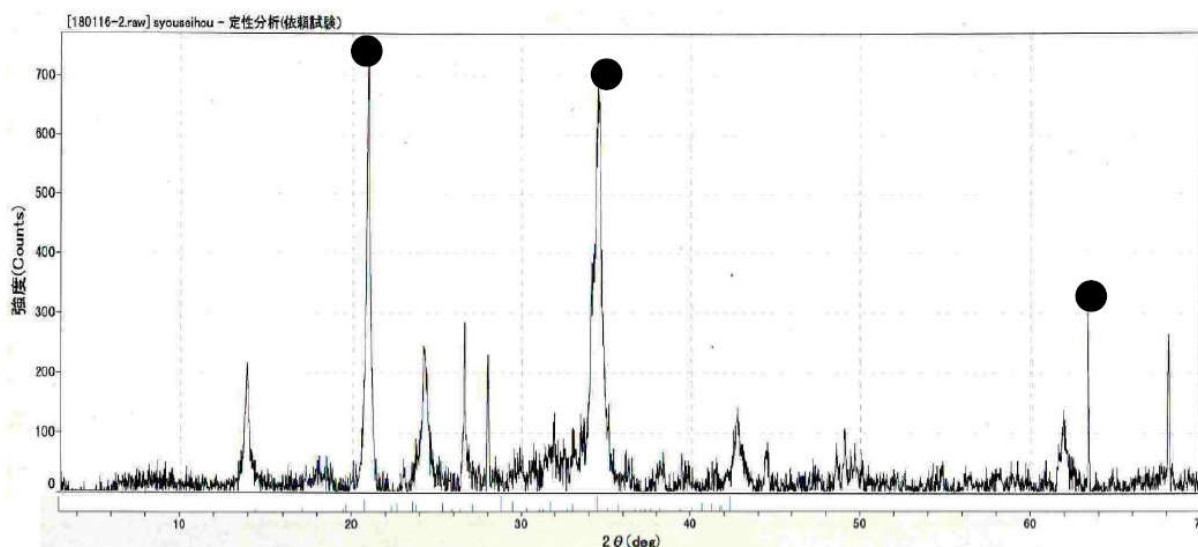


Fig. 7 X-ray diffraction patter of the residue (NaOH).

Source: ●: Sodium aluminum silicate.

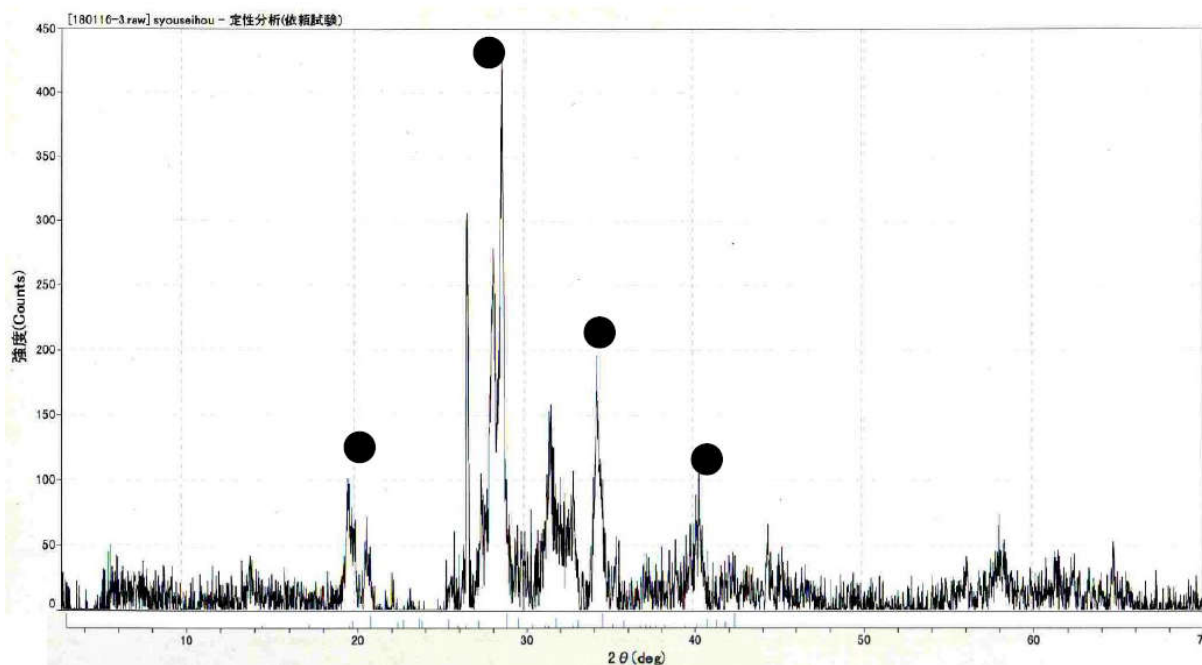


Fig. 8 X-ray diffraction pattern of the residue (KOH).

Source: ●: Potassium aluminum silicate.

4. Conclusions

Phosphorus was recovered from the charcoal of the sewage sludge with a high recovery rate through incineration by the addition of alkali metal hydroxide. Almost all of the recovered phosphorus is considered to be alkali metal phosphate which can be used in

many ways. However, much investigation will be needed to determine the best method for practical usage.

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References

- [1] Shima, H., and Takahashi, M. 1997. Gekkan Water 39-7 (552), 36-40. (in Japanese)
- [2] Yosida, K., Takahashi, Y., and Hatano, M. 2001. "The Experiment on the Recovering Sodium Phosphate from Incinerated Ash of Sewage Sludge." In *Proceedings of 12th Annual Conference of the Japanese Society of Waste Management Expert*, 280-1.
- [3] Ingham, J., Ryan, J., Keyakida, E., and Ri, J. 1996. "Phosphorus and Metal Recovery from Sewage Treatment Sludge." In *Proceedings of the 7th Annual Conference of the Japan Society of Waste Management Expert*, 280-2. (in Japanese)
- [4] Takahashi, M., Kato, S., Shima, H., Sarai, E., Ichioka, T., Hayakawa, S., and Miyajiri, H. 2004. "Technique for Recovering Phosphorus Salt from Incinerated Ash of Sewage Treatment Sludge." *Transactions of Materials Research Society of Japan* 29 (5): 2149-52.
- [5] Takahashi, M., Kato, S., Shima, H., Sarai, E., Ichioka, T., Hayakawa, S., and Miyajiri, H. 2001. "Technology for Recovering Phosphorus from Incinerated Wastewater Treatment Sludge." *Chemosphere* 44: 23-9.
- [6] Sugawara, R., Abe, T., Sato, K., Moriya, Y., and Hatsuyama, S. "Hai alkali riyo niyoriu rinn kaisyuu zissyuu siken oyobi rinn kaisyuu plant kadou kosuto sisan." ("Phosphorus Recovery Test Using a Waste Alkali Materials and Running Cost Estimation of the Recycle Plant.") Accessed Feb. 5th 2018, https://www.jstage.jst.go.jp/article/jsmcwm/23/0/23_311/_pdf.
- [7] Takahashi, M., Sato, K., Kato, S., and Enjyoji, H. 2004. "Technique for Recovering Sodium Phosphate from Ash of Sewage Sludge." In *Proceedings of the Sixth International Conference on ecobalance*, 629-32.
- [8] Takahashi, M., Sato, K., Kato, S., and Enjyoji, H. 2004. "Technique for Recovering Phosphorus Salt from Incinerated Ash of Sewage Sludge." *Journal of Advanced Science* 16 (2): 66-70.
- [9] Takahashi, M., Sato, K., and Nakahara, K. 2005. "Recovery of Phosphate Salt from Charcoal of Sewage Sludge." In *Proceedings of the 15th Annual Conference of the Japan Society of Waste Management Expert*, 386-8. (in Japanese)
- [10] Takahashi, M., and Takemoto, Y. 2015. "Formation of Cr^{6+} through Drying Processes." *Journal of Material Science A* 5 (5-6): 230-2. (in Japanese)
- [11] Sogawa, K., and Yamamura, S. *Surveillance Study to Recycle Wood Pellet Combustion Ash Properly and Safely*. Annual Report of Kochi Prefectural Environmental Research Center 26, 17-23. (in Japanese)
- [12] Tomonaga, H., and Tatsumi, T. 1991. "Zeolite Science and Technology—Application for Development of Natural Resources." Accessed Apl. 2nd 2018. https://www.jstage.jst.go.jp/article/shigentosozai/1989/10/7/1/107_1_2/_pdf.