Recovery of Sodium Phosphate from Incinerated Ash of Sewage Sludge by Gel Formation

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Abstract: In order to develop a phosphorus recovery method, investigation of a phosphorus recovery from incinerated ash of sewage sludge through gel formation was carried out. Aluminum phosphate was extracted from ash of sewage sludge using acidic extraction. The recovered aluminum phosphate was dissolved using aqueous NaOH solution, and sodium silicate was added to the solution. Sodium phosphate was recovered through gel formation followed by solid-liquid separation.

Key word: Aluminum phosphate, sodium phosphate, gelation, sodium silicate.

1 Introduction

A significant amount of phosphorus is contained in municipal waste water. Almost all the phosphorus is discharged with sewage sludge. In order to recover the phosphorus from sludge, some studies have been carried out [1-6]. The phosphorus that is recovered by acidic treatment contains a lot of aluminum [7]. To make a wide utilization of recovered phosphorus, the removal of the aluminum component is considered a very important matter, and some methods were investigated [8].

The aluminum component in the recovered phosphorus is considered to exist mainly as a form of aluminum phosphate. Aluminum reacts with alkali metal silicate to form a silicate gel, which is widely known as zeolite synthesis. Aluminum ions phosphate is dissolved by the addition of an aqueous solution of alkali metal hydroxide, and is also expected to react with alkali metal silicate to make an alkali metal silicate gel. Using this reaction (1), alkali metal phosphate is recovered [9]. Sodium phosphate was recovered by this method using reagent of AlPO₄ and sodium silicate [10]. We investigated the recovery of sodium phosphate from ash of sewage sludge instead of the reagent.

\[ \text{M}_2\text{SiO}_3 + \text{mAlPO}_4 + \text{nMOH} \rightarrow \text{M}_2\text{O} \cdot \text{xAl}_2\text{O}_3 \cdot \text{ySiO}_2 \cdot \text{zH}_2\text{O} + \text{M}_3\text{PO}_4 \] (1)

M: alkali metal; m, n, x, y, z: indefinite number.

2. Method

2.1 Preparation of the Raw Materials

As a raw material for this experiment, aluminum phosphate was recovered from the incinerated ash of sewage sludge which is discharged from the Yokkaichi municipal waste water treatment facility. And 400 g of the ash was mixed with 4 L of aqueous and sulfuric acid solution (H₂SO₄ 150 g), and filtrated. In order to adjust the pH 4.5, 100 g of sodium carbonate (Kanto chemical Co., INC, assay minimum 97%) was added to the filtrate, then aluminum phosphate in the extract was precipitated, and recovered (as raw AlPO₄, dry weight 134 g) by filtration using filter paper (ADVANTEC, No.2, Toyo Roshi Co., LTD). Chemical composition of the raw AlPO₄ is shown in Table 1. Aqueous aluminum phosphate solution was prepared by dissolving the 50 g of raw AlPO₄ with 35 g of NaOH (Kanto chemical Co., INC, assay minimum 97%) to 700 mL of water. Almost all the AlPO₄ was
Table 1  Composition of the raw AlPO₄

<table>
<thead>
<tr>
<th>Component</th>
<th>Composition (W%)</th>
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<th>Composition (W%)</th>
<th>Component</th>
<th>Composition (W%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>1.39</td>
<td>K₂O</td>
<td>1.55</td>
<td>CaO</td>
<td>0.88</td>
</tr>
<tr>
<td>Na₂O</td>
<td>8.99</td>
<td>SO₃</td>
<td>6.93</td>
<td>ZnO</td>
<td>0.8</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>13.23</td>
<td>Fe₂O₃</td>
<td>8.59</td>
<td>others</td>
<td>1.03</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>55.26</td>
<td>MgO</td>
<td>1.35</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Experimental Procedure.**

dissolved, and other components were precipitated as brown sediment (8.4 g) [11].

An aqueous solution of sodium silicate was prepared using a reagent of sodium silicate [Kanto chemical Co., INC (assay minimum SiO₂: 37%, Na₂O: 18%)]. And 100 g of the sodium silicate was dissolved in 100 mL of water (total volume 155 mL).

2.2 Experimental Procedure

The experiment was carried out as shown in Fig. 1. An aqueous sodium hydroxide solution was added to the raw AlPO₄ and dissolved aluminum phosphate was separated using filter paper (as mentioned). Aqueous sodium silicate solution was added to the filtrate, and gel was formed at room temperature. To make rapid dehydration, a freezing and de-freezing method was applied to the dehydration of sludge [12], the gel which was dehydrated through de-freezing, was separated by filtration, and the residue was dried at 105 °C for analysis. The phosphorus component was recovered from the extract through evaporation.

2.3 Addition Rate

In order to find a proper addition rate, 3 mL to 15 mL of aqueous solution of sodium silicate as mentioned was added to 100 mL of aqueous AlPO₄ solution at room temperature (about 25 °C), and the mixture formed a gel in a few minutes. The formed gel was cooled in an ice box (under -10 °C) for 24 hours, and the frozen gel was retained in a room for de-freezing. Dehydration took place through de-freezing, and the dehydrated gel was separated using filter paper, and the gel residue was dried at 105 °C for analysis.

2.4 Recovery of Phosphorus

A 200 mL aqueous solution of aluminum phosphate was mixed with a 24 mL aqueous solution of sodium silicate (expected as most probable addition rate) at room temperature, and a gel was for medina condition of pH 11.5. The formed gel was cooled in an ice box, and dehydration was carried out as mentioned before. The dehydrated gel was dried and the phosphorus containing extract was evaporated for analysis, and gel (17 g) and phosphorus (23 g) were recovered.

3. Result and Discussion

3.1 Addition Rate

The relation of the amount of the formed gel and the addition rate of the sodium silicate is shown in Fig. 2. The amount of the formed gel, increased by the addition of sodium silicate, and when the addition rate
reached 4 g (solution of sodium silicate 6 mL), the amount of the gel tends to plateau, and is considered to be most probable addition rate.

3.2 Properties of Recovered Materials

The recovered gel is a fine white powder with diameter of 0.1 mm. The chemical component of the recovered gel and phosphorus was analyzed by X-ray analyzer, (XRF, Rigaku Corporation SPECTRO XEPOS) and the result is shown in Fig. 3. The recovered gel was mostly composed of Na$_2$O, SiO$_2$, Al$_2$O$_3$, and it was considered to be a compound of alumino-silicate. The elemental ratio of the gel was calculated from the analyzed data (X-ray analyzer), and the approximate molecular ratio was considered as Eq. (2).

\[
\text{Na}_2\text{O} \cdot x\text{Al}_2\text{O}_3 \cdot y\text{SiO}_2; \frac{x}{y} = 1/9
\]  

(2)

The ratio of $x/y$ corresponds to the chemical composition of a zeolite (< 0.5) [13]. However, XRD (X-ray diffraction) of the gel shows the pattern of an amorphous compound (Fig. 4), and further investigation is needed to identify some properties and physical structure.

The amount of P$_2$O$_5$ component in the gel is very small, and almost all of the phosphorus was separated from the gel as an extract. The recovered phosphorus was mainly composed of P$_2$O$_5$ and Na$_2$O, and is considered to be a form of Na$_3$PO$_4$ by the XRD analysis (Fig. 5), and the recovered condition pH 12.5, in this condition, phosphorus can make any form of HPO$_4^{2-}$ or PO$_4^{3-}$ [14]. A small amount of SiO$_2$ and SO$_3$ component was found in the recovered phosphorus. The SiO$_2$ component is considered non-reacted sodium silicate which remained in the recovered phosphorus, and SO$_3$ component is also considered to be a contaminant in the raw AlPO$_4$. The residue is mainly composed of Fe$_2$O$_3$, and contained a significant amount of P$_2$O$_5$, Al$_2$O$_3$ and Na$_2$O. The raw AlPO$_4$ that contained FePO$_4$, FePO$_4$ reacts with NaOH, forms a Na$_3$PO$_4$ [15] and results in forming a Fe$_2$O$_3$. The other components as Al$_2$O$_3$, Na$_2$O are considered to be the non-reacted AlPO$_4$ compound in raw AlPO$_4$.

![Fig. 2 Relation of sodium silicate and formed gel.](image)

![Fig. 3 Chemical composition of recovered materials.](image)
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Fig. 4  XRD spectrum of the gel.

Fig. 5  XRD spectrum of the recovered crystal.
4. Conclusions

In order to find a wide utilization of aluminum phosphate which is recovered from an incinerated ash of sewage sludge, a conversion method of aluminum phosphate to sodium phosphate was investigated. Aluminum phosphate which was recovered from the ash of sewage sludge, was dissolved by sodium hydroxide, and reacted with aqueous solution of sodium silicate, forming a silicate gel. Aluminum phosphate was converted to sodium phosphate through the formation of alumino-silicate gel. The result indicates the possibility to develop a recovery method of sodium phosphate from sewage sludge.

References


