A Method for Estimating Amounts of Acid-Soluble Phosphate in Carbonized Activated Sludge

TAFU Masamoto†* and CHOJI Tetsuji*

*Department of Ecomaterials Engineering, Toyama National College of Technology, 13 Hongo-machi, Toyama, 939-8630 Japan

(Received, September 22, 2005, Accepted May 22, 2006)

Wastewater containing organic substances is usually treated by the activated sludge process. With the expansion of sewage systems, the amount of activated sludge increases vastly. In Japan, it becomes increasingly difficult to secure landfill sites for disposing the increase amount of excess sludge. Carbonizing activated sludge has been developed as one of the methods to reduce and/or recycle wastes. Carbonized activated sludge is useful because it contains phosphorus to be extracted from it, and to be a good adsorbent. The amount of the acid-soluble phosphorus in carbonized activated sludge was experimentally estimated in this study.

Key Words: phosphate, activate sludge, resource recovery, carbonized sludge

1. Introduction

In Japan, waste water containing phosphorus is ordinarily treated by the activated sludge process, resulting in the production of excess sludge, which is increasing year by year. Thus the amount of excess sludge produced needs to be controlled and reduced. At present, most excess sludge is incinerated and disposed of in landfills. A fear has arisen in which the world’s phosphorous resources will be exhausted in the future. The excess sludge is rich in phosphorus, and so various resource extraction processes have been proposed to achieve the highly-efficient recovery of the phosphorus in excess sludge. Some methods for recovering phosphorous from activated sludge have been reported. However, the authors of these reports were interested in just the efficient recovery of the phosphorus in activated sludge.

By contrast, we are also interested in carbonized active sludge as a way of reducing the amount of excess sludge. If phosphorus is extracted from carbonized activated sludge to an acidic solution, it is expected that the material remaining can be utilized for soil improvement as with activated carbon.

In this study, we evaluated the content of acid-soluble phosphorus in carbonized activated sludge experimentally, and developed a method for estimating the amounts of extractable phosphorus in the sludge to an acidic solution.

2. Experimental

2.1. Sample and Reagents

Carbonized activated sludge produced in a carbonizing furnace from excess activated sludge was used for the experiments. The sample was dried in an electronic oven at 378 K until the weight of the sludge became fixed.

2.2. Assessment of acid-soluble phosphate content in carbonized activated sludge

Fig. 1 shows a schematic diagram of experimental procedure. 1g of dry sludge was added to 50cm³ of nitric acid adjusted to various pH values in a polypropylene bottle. The mixture was shaken in a reciprocal shaker (Type NR-1, TAIJEC, Japan) at a room temperature. The solution was filtered with a 0.45 μm membrane filter. The sample remaining on the filter was again mixed with 50cm³ of nitric acid. This process was repeated 5 times. The concentrations of phosphorous and another element in the solution were measured by induction coupled plasma atomic emission spectroscopy (ICP-AES,
nitric acid adjusted to pH 1.0 in a glass beaker with four baffle boards in it. The mixture was stirring by a mixer at 298K for various periods. The pH of the mixture was monitored by a pH electrode (Orion PerPHect 9207BN, Thermo Electron Corp., USA) and a pH meter (PCM 500, Thermo Electron Corp.). The concentration of phosphorous in the solution filtered with a 0.45 μm membrane filter was measured by ICP-AES.

3. Results and discussion

3.1 Assessment of acid-soluble phosphate content in carbonized activated sludge

The results from molybdenum blue absorption spectrometry and ICP-AES showed that all of the phosphorous eluted from the sludge was orthophosphate. Fig. 3 shows the concentrations of phosphate in the solution under various pHs. This figure shows that more phosphate was eluted at lower pH of the solution.

Fig. 4 shows the concentration of phosphate eluted from the carbonized active sludge at each washing process as detailed in 2.2 above. The pH was adjusted to 1.0 by nitric acid. It was clear that the entire amount of phosphate could not be completely eluted by washing 5 times. We found that the concentration of phosphate decreases in a geometrical ratio, so we estimated the total amount of phosphate (C) eluted from the sample by using the following geometric formula:

![Graph showing phosphorous concentration of solution containing 1.0 g of dry sample of carbonized activated sludge in 50 cm³ of nitric acid adjusted to various pH values after 1 hour mixing.](image)
Fig. 4  Total amount of phosphorous in elution (Cn) as a function of number of washings (n). Washing was carried out at room temperature for 1 hour.

\[ C_n = \sum_{n=1}^{\infty} ar^n = \frac{ar}{1 - r} \quad (1) \]

in this formula, a and r were estimated from Fig. 4. The result from Eq. (1) was that the content of acid soluble phosphorous in 1g of carbonized activated sludge was calculated to be 54.8 mg·g⁻¹.

Fig. 5 and Fig. 6 shows the concentration of aluminum and calcium ions eluted from the carbonized active sludge at each washing process as detailed in 2.2 above. These results indicated that not only the amount of phosphorus but also those of the other elements could be estimated by the same method. Table 1 shows acid-soluble amounts of various elements in 1g of carbonized activated sludge.

Fig. 5  Total amount of aluminum in elution (Cn) as a function of number of washings (n). Washing was carried out at room temperature for 1 hour.

Fig. 6  Total amount of calcium in elution (Cn) as a function of number of washings (n). Washing was carried out at room temperature for 1 hour.

Table 1  Content of components in carbonized activated sludge

<table>
<thead>
<tr>
<th>Elements</th>
<th>Parameters in Eq.(3)</th>
<th>Contents [mg·g⁻¹]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PO₄³⁻</td>
<td>1.47×10² 0.532</td>
<td>167.8</td>
</tr>
<tr>
<td>Al</td>
<td>2.19×10¹ 0.588</td>
<td>31.2</td>
</tr>
<tr>
<td>Ca</td>
<td>3.83×10¹ 0.444</td>
<td>26.9</td>
</tr>
<tr>
<td>Fe</td>
<td>1.94×10¹ 0.579</td>
<td>26.7</td>
</tr>
<tr>
<td>Mg</td>
<td>9.21×10⁰ 0.500</td>
<td>9.2</td>
</tr>
<tr>
<td>Ba</td>
<td>2.32×10⁰ 0.657</td>
<td>4.4</td>
</tr>
<tr>
<td>Si</td>
<td>2.50×10⁰ 0.631</td>
<td>4.3</td>
</tr>
<tr>
<td>K</td>
<td>1.95×10⁰ 0.588</td>
<td>2.8</td>
</tr>
<tr>
<td>Na</td>
<td>7.01×10⁻¹ 0.567</td>
<td>0.9</td>
</tr>
<tr>
<td>S</td>
<td>4.00×10⁻¹ 0.684</td>
<td>0.9</td>
</tr>
</tbody>
</table>

3.2. Recovery of phosphate from carbonized activated sludge

Fig. 7 shows changes in the pH and concentration of phosphate of the mixture which was prepared according to the procedure that previously described in 2.3. The concentration of phosphate reached a plateau (2000 mgdm⁻³), and the pH of the mixture exceeded 1.5 within 4 hours.

4. Conclusion

In this paper, the content of acid-soluble phosphorus
Fig. 7 Changes in pH and concentration of phosphate of the mixture containing carbonized activated sludge as a function of leaching time. Reaction temperature was 298K, and the 20 g sample was added to 1000 cm$^3$ of nitric acid adjusted to pH 1.0.

and various elements in carbonized activated sludge were experimentally evaluated, and a method of extracting phosphorus from the sludge in a solution was developed. We proposed a novel method to evaluate the amount of acid-soluble phosphorus and other elements. According to the results from the analysis by the adsorption spectroscopy, all of the phosphorous extracted from the carbonized sludge was recognized as orthophosphate, which has been used in various chemical industries. The phosphorous was easily extracted from the sample as orthophosphate by mixing in diluted nitric acid over a period of 4 hours.

It is expected that the resource recovery method proposed in this study has the potential to contribute to the preservation of phosphorus resources not only in Japan, but worldwide$^7$.

Acknowledgements

The authors deeply appreciate the helpful support with the experiments given by Ms. Yoshite Takai and Ms. Mikiko Kobayashi (Toyama National College of Technology). This study was supported in part by the Industrial Technology Research Grant Program from the New Industrial Technology Development Organization (NEDO), Japan (#04A19013).

References