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Phosphorus recovery from digested sewage sludge as MAP by the help of metal ion separation

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ABSTRACT

This study was designed to solve metal ion influence problem on phosphorus recovery from digested sewage sludge as MAP. The experimental steps were proceeded to maximize MAP production and its quality. Used experimental steps were:

(a) Acidic dissolution of phosphorus,
(b) Removal of metal ions from phosphorus rich water phase,
(c) Recovery of phosphorus as MAP,
(d) Separation of MAP.

All digested sewage sludge samples were taken from Stuttgart University sewage treatment plant for research and education (LFKW). Four different forms of LFKW digested sewage sludge were used as feeding sample. These were: original digested sludge, diluted digested sludge, centrifuged digested sludge and incinerated digested sludge.
A Donnan membrane unit having a Nafion 117 (DuPont) cation exchange membrane was used to remove metal ions from the samples used. Highest metal ion removal efficiencies, which were 98%, 97%, and 80% for Al, Ca and Fe ions, respectively, were obtained from incinerated digested sludge run.
Incinerated digested sludge run was used as preliminary step for MAP production and high quality MAP was produced. Produced MAP fulfils all requirements related with Düngemittelverordnung 2003 and it could be used as a fertilizer in Germany.

1. Introduction

Phosphorus is an essential nutrient for all forms of life. Phosphorus consumption in the world in year 2006 was around 142 million tons of phosphate rock concentrate (U.S. Geological Survey, 2008), which is annihilating economically extractable phosphate rocks. Therefore, the reliance on phosphate rocks should be stopped and sustainable ways to use phosphorus should be researched.

A sustainable way to use phosphorus can be recovery of phosphorus from digested sewage sludge (will be referred as digested sludge) since almost all the removed phosphorus accumulates in treatment sludge in a conventional wastewater treatment plant whether phosphorus in wastewater is removed by biological phosphorus removal or by chemical phosphorus removal. On the other hand most of the metal ions and heavy metals accumulate in treatment sludge too. Al, Ca and Fe (main metals) ion concentrations in digested sludge

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are usually high, especially where phosphorus removal is made by addition of metal salts.

Since mineral fertilizers account for approximately 80% of phosphates used worldwide (Steen, 1998), it would be beneficiary to recover phosphorus from digested sludge as a fertilizer. One of the most popular phosphorus recovery applications in this sense is converting phosphorus from digested sludge into magnesium ammonium phosphate hexahydrate (MAP or struvite).

MAP is a slow releasing fertilizer with a chemical formula of $MgNH_4PO_4 \cdot 6H_2O$. The quality of MAP as a fertilizer has been researched and usually found as in average to high quality (Ahmed et al., 2006; Ghosh et al., 1996; Scope Newsletter, 2001).

Minimum MAP solubility is observed between pH 8.0 and pH 10.0 (Buchanan et al., 1994; Booker et al., 1999; Momberg and Oellermann, 1992). During pH arrangement of digested sludge samples for MAP production, main metal ions react with available phosphorus and precipitate as metal phosphates at pH above 4.0 (Jaffer et al., 2002; Weidelener et al., 2005), reducing phosphorus concentration which is available to form MAP. Therefore, to prevent the re-precipitation of phosphorus with metal ions, interfering metal ions have to be either removed or inactivated.

Weidelener et al. (2005) investigated the handling of main metal ions from digested sludge of Stuttgart University sewage treatment plant for research and education (Lehr- und Forschungsklärwerk Büsnau-LFKW) to increase recovery of phosphorus as MAP, by using complexing agents. But they found that, despite the application of complexing process, MAP production efficiency was affected negatively by high concentration of aluminium.

Prakash et al. (2004) investigated removal of Al from water treatment plant residuals by using Donnan membrane process which includes a Nafion 117 (DuPont) cation exchange membrane and observed 81% Al removal. Schaum (2007) used Donnan membrane process which includes a Nafion (DuPont) cation exchange membrane to remove Al ions out of $AlPO_4$ solution which has 8 g P/l. He observed almost 60% Al removal and almost 14% P removal in 16 h of membrane unit operation. Therefore using a Donnan membrane process to remove metal ions by the help of a Nafion 117 membrane was thought to be a promising idea.

This research was focused on metal ion removal prior to MAP production. LFKW digested sludge samples were used in different forms to recover phosphorus as MAP. First, the best metal ion removal method was investigated and then MAP production was processed by using it as preliminary step. As final step, usability of produced MAP as a fertilizer in Germany was investigated.

The following steps are proceeded in order to maximize metal ion removal and therefore maximize phosphorus recovery and MAP production:

(a) Acidic dissolution of phosphorus and obtaining phosphorus rich water phase,
(b) Removal of metal ions from phosphorus rich water phase,
(c) Recovery of phosphorus as MAP by chemical precipitation in liquid phase at pH 8.5 after selecting and applying best metal ion removal method,
(d) Separation of MAP.

2. Materials and methods

2.1. LFKW digested sludge

Digested sludge samples were used in four different forms: original digested sludge (OLDS), diluted digested sludge (DLDS), centrifuged digested sludge (CLDS), and incinerated digested sludge (ILDS).

Phosphorus removal in LFKW is being made chemically. LFKW eliminates phosphorus by simultaneous precipitation mainly with sodium aluminate and sometimes with Fe salt additions. Al, Ca and Fe concentrations are usually the highest metal ion concentrations in digested sludge. The properties of digested sludge are stated in Table 1.

2.2. Mixing and pH control unit

Each sample was put into a beaker and mixed with a paddle or magnet mixer. During mixing, sample pH was controlled by adding 25% sulphuric acid or 32% caustic soda automatically by using Metrohm 725 Dosimat and Pectec pH-mV Regler M8832N pH meter.

2.3. Acidic dissolution

Dissolution of phosphorus was made with addition of 25% sulphuric acid into the respective sample mainly for 24 h. During the acidic dissolution, samples were continuously stirred and sulphuric acid was added automatically.

Dissolution of phosphorus increases rapidly between pH 3.5 and pH 1.0 by decreasing pH of the sludge sample. More than 90% phosphorus dissolution can easily be achieved at pH between 1.8 and 1.9.

2.4. Experimental setup and methods for metal ion separation

Metal ion separation, especially separation of Al, Ca and Fe ions, was needed to minimize metal ion interference on MAP production.

Table 1 – The properties of LFKW digested sewage sludge (mf: membrane filtrated)

$HRT_{Digester}$ [Days]	60–65
pH	7.4
Temperature [°C]	35–37
P_{Total} [mg/l]	2550
$P_{Total\ mf}$ [mg/l]	93
Phosphorus in dry solids [%]	4.65
COD [mg COD/l]	47000
$NH_4–N_{mf}$ [mg/l]	1724
Dry solid [%]	5.5
Loss of ignition [%]	62.5
Al [mg/l]	2720
Al_{mf} [mg/l]	<2
Ca [mg/l]	2655
Ca_{mf} [mg/l]	54
Fe [mg/l]	505
Fe_{mf} [mg/l]	<2