Phosphate Recovery from Sewage Sludge for Application as a Substitute to Phosphate Rock

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Abstract

Huge amounts of sewage sludge, a form of solid waste, are being generated on a daily basis from municipal sewage treatment plants. This sewage sludge if not properly treated can cause landfilling problems that can result in climate change effects due to greenhouse gases emissions. This generated sewage sludge is rich in phosphorous and other nutrients which has potential to be recovered. Phosphorous can be recovered and used as an alternative to the in fertilizer production or applied directly to crops. The current study evaluates the potential to recovery phosphorous from sewage sludge using gasification as a thermal method. The sewage sludge was first dewatered to 60 wt.% moisture content before it was gasified. Gasification at 800°C was considered as the waste management option for the sewage sludge due to its flexibility and energy efficiency. At most 50% of the phosphate was recovered with a phosphorous nutrient composition of 6.5-7.5 wt. %. Gasification of sewage sludge to recover phosphate promotes environmental preservation at the same time creating a resource for fertilizer production.

Keywords
Gasification, phosphorous recovery, sewage sludge and waste management

1. Introduction
Phosphorous is a key element which is non-renewable for fertilizers production and the mineral resource are depleting hence the need for alternative sources. Alternative sources of phosphorous such as sewage sludge must then be considered for sustainable availability of this resource where it is available in large quantities (Stendahl et al. 2002). In addition, if sewage sludge is left unmanaged, it will result in methane generation in overall negative climate change effects. Sewage sludge in its dried form has also been widely applied as a fertilizer due to the presence of phosphorous as well as other minerals like lime, organic compounds and nitrogen. Although traditionally, sewage sludge has been managed through incineration to recover phosphorous, gasification has become a topical alternative technology (Pareset al. 2017).

Gasification therefore becomes an attractive potential due to its flexibility and energy efficiency through utilization of gas produced through heat integration (Samolada and Zabaniotou 2014). In addition, gasification also allows for destruction of available pathogens in the sewage sludge. During the process of gasification, the carbon based material in the sewage sludge is converted to fuel through partial oxidation at temperatures between 800-1400 °C in the presence of steam. The inorganic matter is then converted into ash (Keiko et al., 2001).

The sewage sludge ash can then be treated by acid or alkali treatment in order to extract the ash (Yousuke et al., 2018). Acid treatment of the ash is however not desirable since it results in high amounts of aluminium unlike when alkali extraction is used (Kunihiko et al., 2004). This work focused on determining the optimal conditions for the recovery of phosphate from sewage sludge as a waste management technique through first the gasification process and then alkali treatment of the ash.

2. Materials and Methods
2.1 Sewage sludge sample collection
A sample consisting of 5kg sewage sludge was collected from a local wastewater treatment plan. The wastewater treatment plant uses the biological treatment of wastewater technique. The sewage sludge was then characterized for both moisture content and phosphorous content.

2.2 Proximate analysis of the sewage sludge

Moisture content determination, fixed content, volatile matter and the ash content of the sewage sludge were determined using proximate analysis.

The moisture content (MC) is defined as the total weight-loss of a sample after it has been dried at 105 °C. A 5g sample of dried sludge is measured using digital analytical balance before being dried by using oven at 110 ⁰C for 4 hours. The weight of the sludge after drying is then determined using an analytical balance. The moisture content of the sludge is then calculated as indicated in Equation 1.

$$\%MC = \frac{W_1 - W_2}{W_1} X\ 100\% \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (1)$$

Where, MC is meant the approximate equilibrium moisture content, W_1 is the mass of the wet sludge and W_2 is the mass of the dried sludge.

The volatile substance (VM) (loss on ignition) is the amount of mass of dry residue which has been gasified at 550 °C. A 1g sample of dried sludge was placed in a pre-weighted crucible and heated in a muffle furnace at 925 °C for 7 minutes, then cooled in a desiccator and weighted. The percentage of the volatile matter was determined using Equation 2.

$$\%VM = \frac{A - C}{A - B} X\ 100\% \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (2)$$

Where A is the weight of the dried sample and crucible, B is the weight of crucible and C is the weight of residue and crucible after ignition.

For ash content (AC) analysis, 1g of dried sample was placed in a preweighted crucible in order to determine the ash content of the sewage sludge. The sample and the crucible were weighted together before heated in a furnace at 750 °C for an hour. The crucible and the ash were weighted after cooling in a desiccator. The ash content is calculated in accordance to Equation 3.

$$\%AC = \frac{W_2 - W_C}{W_1 - W_C} X\ 100\% \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (3)$$

Where Wc is the weight of the crucible, W_1 is the weight of the sample and crucible, W_2 is the weight of the crucible and the ash.

The fixed carbon content (FC) value is determined in accordance to Equation 4.

$$\%FC = [100 - \mathrm{MC} - \mathrm{AC} - \mathrm{VM}]\% \dots\dots\dots\dots\dots\dots\dots\dots\dots (4)$$

Where MC is the percentage of moisture contents of the sample, AC is the percentage of ash content of the sample and VM is the percentage of the volatile matter content of the sample.

2.3 Ultimate analysis of the sewage sludge

The carbon, hydrogen, nitrogen, oxygen and sulphur of the dried sewage sludge were determined. The ultimate analysis of the sewage sludge is done by using Chemical Element Analyser (CHNS/O).

2.4 Sewage sludge calorific value analysis

Calorific value is the amount of energy that stored in a substance which is sewage sludge (Nzihou et al., 2014). Calorific value of the sewage sludge was obtained using a ParrTM Model 1108 Oxygen Combustion Vessel® at standard conditions.

2.5 Gasification of the sewage sludge

The sewage sludge was first dried and then passed through an ECN lab scale 2L gasifier in accordance to a procedure described in detail by (Hansen et al., 2015). The gasification was carried out at temperatures between 400 - 800 °C and partial oxygen conditions of 0.1-0.3 wt.%. The optimal gasification and partial oxygen conditions were then determined.

2.6 Analysis of the gasified sewage sludge ash

The composition of the sewage sludge ash was determined on a percentage wet basis (wt. %). The analysis of the oxides in the sewage sludge was done using a Perkin Elmer AAS Model 460 atomic adsorption with flame atomisation after dissolution in acid.

2.7 Extraction of phosphorous using NaOH

For the extraction process, 500g of 1M NaOH was added to 500 g of sewage sludge ash to form an alkali metal phosphate and heat treated at temperatures 750-900 °C. The mixture was then dried at 105 °C for 1 hour in the Memmert Oven Model 105. Afterwards, 5000 mL of water was then added to the mixture (ratio 1:10), after which it was dried at 105 °C. The optimal heat treatment temperature was chosen at the highest yield of phosphate.

3. Results and Discussion

3.1 Sewage sludge characteristics and proximate analysis

The dried sewage sludge had a moisture content of 60 wt.% and ash content of 37 wt.% as indicated in proximate analysis in Table 1.

Table 1. Proximate analysis of the sewage sludge

Parameter	**Value (wt.%)**
Moisture content (MC)	60.02
Volatile matter (VM)	56.19
Ash content (AC)	36.72
Fixed carbon (FC)	7.09

The sewage sludge had a carbon content of 33 wt.% and a calorific value of 15 MJ/kg (Table 2). These properties allowed the sewage sludge to be burnt to ashes during gasification.

Table 2. Sewage sludge ultimate analysis

Parameter	**Value**
Carbon	32.79 wt.%
Hydrogen	4.38 wt.%
Nitrogen	4.31 wt.%
Oxygen	22.37 wt.%
Sulphur	0.82 wt.%
Calorific value	14.9 MJ/Kg

3.2 Effect of process conditions on sewage sludge gasification ash quality

3.2.1 Effect of gasification temperature on ash quality

The P_2O_5 composition increased with increase in gasification time from 30 minutes to 90 minutes and increase in the gasification temperatures from 400 - 800 °C at constant oxygen availability of 10 wt.% (Figure 1). After that the composition became constant at all the gasification temperatures with 800 °C (Figure 1). The behaviour can be attributed to the potential of the sewage sludge to be completely burnt to ashes at high gasification temperatures and time. The 800 °C can then be chosen as the optimal gasification temperature at 120 minutes to give a P_2O_5 yield of around 14 wt.% composition.

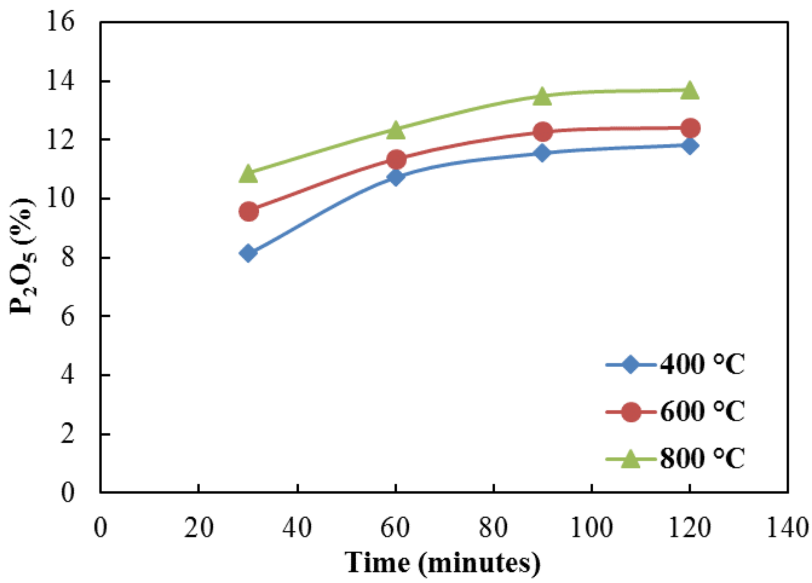

Figure 1. Effect of gasification temperature and time on P_2O_5 composition at 10 wt.% oxygen content

3.2.2 Effect of oxygen content during gasification

Gasification of sewage sludge takes place under partial oxygen conditions. From this study, complete gasification of the sewage sludge was achieved at partial oxygen concentration of 0.1 wt.% (Figure 2). In all cases with increase in gasification time and the oxygen concentration changing from 0.1 wt.% to 0.3 wt.% at a gasification temperature of 800 °C, the P_2O_5 composition steadily increased (Figure 2).

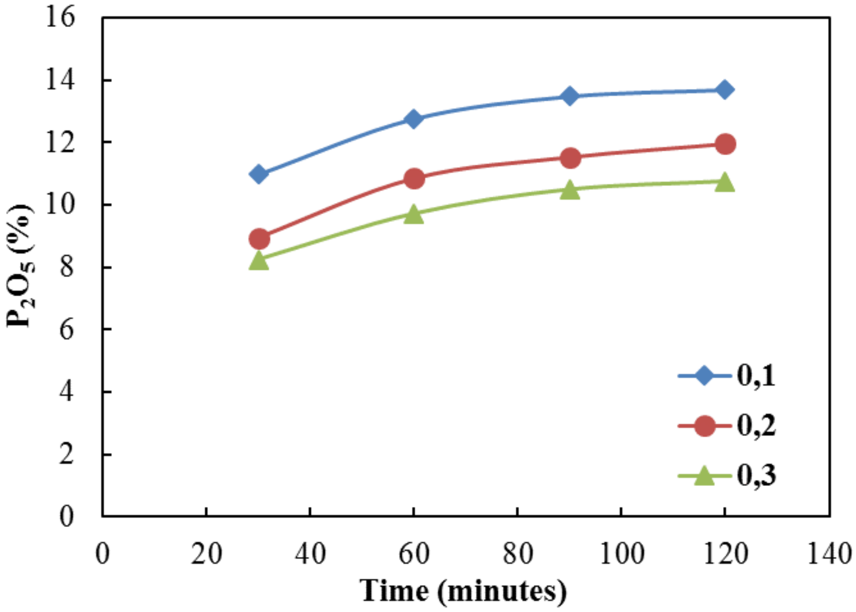

Figure 2. Effect of gasification time and oxygen concentration on P_2O_5 composition at 800 °C gasification temperature

3.3 Process description for sewage sludge through gasification under optimal conditions

The sewage sludge is first dewatered and dried to a moisture content of 60 wt.%; this is done in order to increase the calorific value of the sewage sludge so that it could fully be gasified. The dried sewage sludge is then gasified at 800 °C as the optimal temperature under partial oxygen conditions of 0.1 wt.% oxygen. The gas in the process is recovered as syngas for use as an energy source in the plant, whilst the ashes that are rich in various oxides are sent for further nutrient extraction. In this study, phosphorous oxide was the target oxide for phosphate extraction. The schematic diagram of the process is shown in Figure 3.

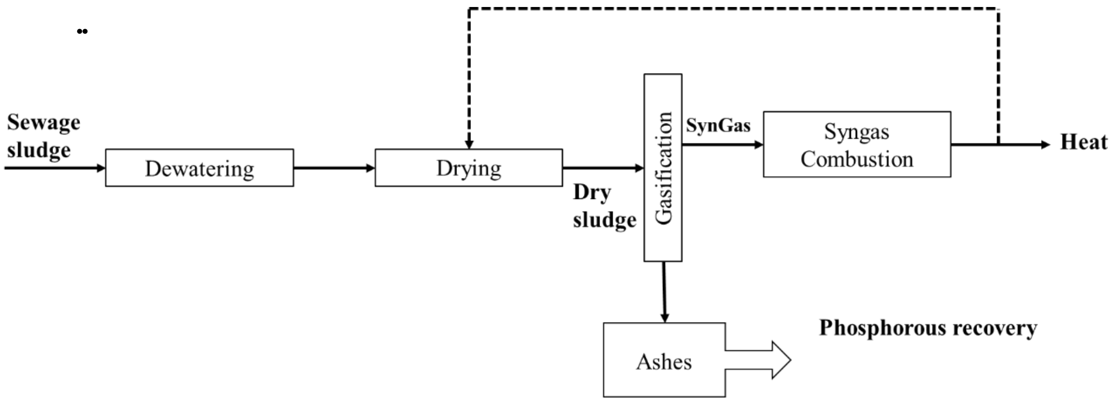

Figure 3. Process description for sewage sludge gasification for phosphorous recovery

A summary of the sewage sludge ash characteristics at optimum conditions of 800 °C, partial oxygen conditions of 0.1 wt.% and gasification time of 120 minutes is shown in Table 3.

Table 3. Sewage sludge ash characteristics at 0.1 wt.% partial oxygen conditions, 800 °C and 120 minutes gasification conditions

Oxide	**Composition (wt. %)**
Silicone oxide (SiO_2)	33.18
Aluminium oxide (Al_2O_3)	15.52
Iron oxide (Fe_2O_3)	7.62
Calcium oxide (CaO)	10.74
Phosphorous oxide (P_2O_5)	13.69
Sulphur trioxide (SO_3)	2.87
Sodium oxide (Na_2O)	1.44
Potassium oxide (K_2O)	0.82
Titanium oxide (TiO_2)	2.15
Magnesium oxide (MgO)	3.28
Manganese oxide (MnO)	0.55
Loss on ignition (LOI)	8.14
Total	100.00

3.4 Phosphorous recovery from sewage sludge ash at various heating temperatures

The amount of phosphorous recovered increased with the heating time applied per sample as well as increase in heating temperature from 750 °C to 850 °C (Figure 4). Applying longer heating times enables the sewage sludge ash and the NaOH to react completely for formation of the alkali metal phosphate. After 900 °C, the P recovered thereafter was 10 wt.% low and this can be attributed to probably disintegration of the sewage sludge ash at higher temperatures.

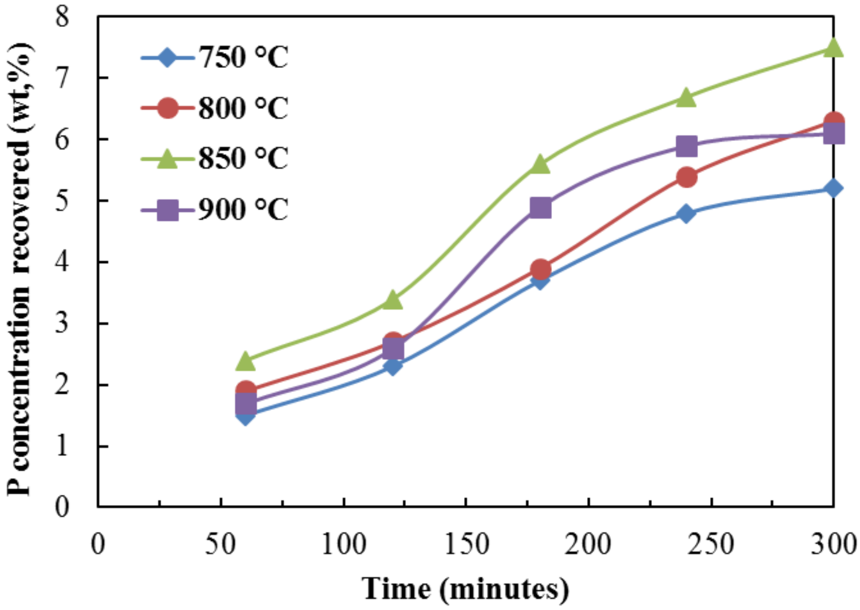

Figure 4. Effect of heating time and temperature on P recovery

3.5 Process description for phosphorous recovery from phosphorous oxide using NaOH

The sewage sludge ash was first mixed with NaOH and then heat treated at the optimal temperature of temperatures of 850 °C after the treatment the mixture was dried at 105 °C for 1 hour. An alkali metal phosphate was formed in the process. Water was then added in the mixture in the ratio 1: 10 to the solid material. The phosphorous was then filtered from the mixture through drying at 105 °C in the oven. The process description is schematically shown Figure 5.

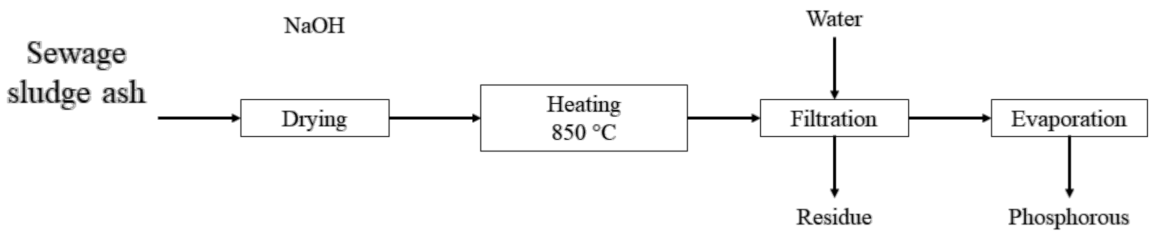

Figure 5. Alkali treatment process for recovery of phosphorous from sewage sludge ash

4. Conclusion
Gasification of sewage sludge is a viable technology for phosphorous recovery in municipal sewage treatment plants. Gasification of sewage sludge at 800 °C results in 13.69 wt. % of P_2O_5 in the ash of which the phosphate content of 6.5-7.5 wt. % is recoverable through alkali treatment. Optimum heat treatment under the alkali treatment method is 850 °C.

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Conflict of interest
The authors declare that there are no competing interests.

References
Hansen, V., Müller-Stöver, D., Ahrenfeldt, J., Holm, J. K., Henriksen, U. B. and Hauggaard- Keiko, Y., Takahashi, Y. and Hatano, M., 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, pp. 280-281, 2001.

Nielsen, H., Gasification Bio char as a Valuable By-product for Carbon Sequestration and Soil Amendment. Biomass and Bioenergy, vol. 72, pp. 300-308, 2015.

Nzihou, J. F., Hamidou, S., Bouda, M., Koulidiati, J. and Segda, B. G., Using Dulong and Vandralek Formulas to Estimate the Calorific Heating Value of a Household Waste Model. International Journal of Scientific and Engineering Research, vol. 5, no.1, pp. 1878 – 1883, 2014.

Parés Viader, R., Jensen, P. E., Ottosen, L. M., Thomsen, T. P., Ahrenfeldt, J. and Hauggaard-Nielsen, H., Comparison of Phosphorus Recovery from Incineration and Gasification Sewage Sludge Ash. Water Science and Technology, vol. 75, no. 5, pp. 1251-1260, 2017.

Samolada, M. C. and Zabaniotou, A. A., Comparative Assessment of Municipal Sewage Sludge Incineration, Gasification and Pyrolysis for a Sustainable Sludge-to-Energy Management in Greece. Waste Management, vol. 34, no. 2, pp. 411-420, 2014.

Standard Methods for Water and Wastewater Examination, 17th Ed. American Public Health Association, Washington, D. C., 1995.

Stendahl, K. and Jäfverström, S., Phosphat Recovery from Sewage Sludge in Combination with Supercritical Water Oxidation. In: Proceedings International Conference from Nutrient Removal to Recovery, Amsterdam, 02. - 04.10.2002.

Yousuke, N., Msanori, O., Sinsuke, H. and Daisuke O., Chemical Composition of Night Soil Sludge Incineration Ash and the Elution of Phosphate. Journal of Environmental Chemistry, vol. 28, no. 4, pp. 127-39, 2018.

Biographies

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