Recovery of Phosphate from Municipal Sewage Sludge

M. M. Manyuchi*.and N. Sukdeo
Department of Operations and Quality Management,
Faculty of Engineering and the Built Environment,
University of Johannesburg, South Africa
*mercy.manyuchi@gmail.com

Abstract

Huge amounts of municipal sewage sludge, a form of solid waste, are being generated daily 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. The current study evaluates the potential to recover phosphorous from municipal sewage sludge using gasification as a thermal method followed by alkali treatment using NaOH. The sewage sludge was first dewatered to 60% moisture content before it was gasified using hot air to convert it into ash for 120 minutes. Gasification at 800 °C was considered as the waste management option for the sewage sludge due to its flexibility and energy efficiency. In this study, 50% of the phosphate was recovered from the sewage sludge ash with a phosphorous nutrient composition of 6.5-7.5 wt. %. Gasification of sewage sludge to recover phosphate oxide and its subsequent alkali treatment to recover phosphate promotes environmental preservation at the same time creating a resource for fertilizer production.

Keywords:
Alkali treatment, gasification, phosphorous, sewage sludge, waste valorisation

1. Introduction

Phosphorous is a key element for fertilizers production and the mineral resources for phosphorous are depleting hence the need for alternative sources (Danesh et al. 2018). Phosphorus is an essential nutrient in living organisms. Phosphorous can be recovered and used as an alternative to the in fertilizer production or applied directly to crops (Zhipeng Zhang et al., 2023). Previous studies have indicated that potential phosphorous shortage are imminent hence the urgent need to have alternative sources of phosphorous (Zhenquan Fang et al.2023).
Alternative sources of phosphorous such as municipal sewage sludge must then be considered for sustainable availability of this resource where it is available in large quantities (Stendahl et al. 2002).

On the other hand, if this phosphorous is not recovered in municipal sewage sludge, it has potential to cause environmental problems such as eutrophication (Yimin Huang et al. 2022). It is estimated that approximately 68 000 m^3/day of activated sewage sludge is generated in municipal wastewater treatment plants in Zimbabwe (Onu et al. 2023). In addition, if this sewage sludge is left unmanaged, it will result in methane generation in overall negative climate change effects (Agoro et al. 2020). Municipal 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. However, unprocessed municipal sewage sludge if applied directly as a bio fertilizer has a potential to introduce unwanted contaminants to the soil including heavy metals such as mercury and lead (Borah et al.2020).

Municipal sewage sludge has been valorised to various products using various technologies such as incineration, pyrolysis, carbonization and gasification (Callegari et al. 2018). Although traditionally, municipal sewage sludge has been managed through incineration to recover phosphorous, gasification has become a topical alternative technology (Pareset al. 2017).

Gasification however, becomes an attractive potential method sewage sludge valorisation to phosphorous due to its flexibility and energy efficiency through potential utilization of gas produced through heat integration (Samolada and Zabaniotou 2014). In addition, gasification also allows for destruction of available pathogens in the municipal sewage sludge making it safer to use (Keiko et al. 2001). 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 sewage sludge ash (Keiko et al. 2001).

The sewage sludge ash can then be treated by acid or alkali treatment in order to extract the heavy metals present in the ash (Yousuke et al. 2018). Acid treatment of the sewage sludge 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 municipal sewage sludge as a waste management technique through first the gasification process and then alkali treatment of the sewage sludge ash.

2. Materials and Methods

2.1 Municipal sewage sludge sample collection

A sample consisting of 5kg municipal sewage sludge was collected from a local wastewater treatment plant at the Chitungwiza sewage treatment plant, Zimbabwe. The wastewater treatment plant uses the biological treatment of wastewater technique and this has potential to retain high phosphorous and nitrogen quantities. The municipal sewage sludge was then characterized for both moisture content, phosphorous content and other elements.

2.2 Proximate analysis of the sewage sludge

The moisture content determination, fixed content, volatile matter and the ash content of the sewage sludge were determined using proximate analyses methods (Shuioa et al. 2023).

2.2.1 Moisture content analysis

The moisture content (MC) is defined as the total weight loss of a sample after it has been dried at 105 °C (Agoro et al., 2020). A 5g sample of dried municipal sewage sludge was measured using digital analytical balance before being dried by using oven at 110 ºC for 4 hours. The weight of the sludge after drying is then determined using an analytical balance. The moisture content of the sludge was then calculated as indicated in Equation 1.

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

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.

2.2.2 Volatile matter analysis

The volatile matter (VM) also known as the loss on ignition is the amount of mass of dry residue which has been gasified at 925 °C (Borah et al., 2020). 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.

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

Where: VM is the volatile matter, 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.

2.2.3 Ash Content Analysis

The ash content in a substance refers to the inorganic material residue that remains after complete oxidation of the organic material in the municipal sewage sludge (Callegari and Capodaglio, 2018). For the ash content (AC) analysis, 1g of dried municipal sewage sludge 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.

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

Where: AC is the ash content, 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.

2.2.3 Fixed Carbon

The fixed carbon content (FC) value is determined in accordance to Equation 4 (Hansen et al., 2001).

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

Where: FC is the fixed carbon content, 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) (Nzihou et al.2014).

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 municipal sewage sludge to recover sewage sludge ash

The municipal 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) using hot air and oxygen air with various compositions. The gasification was carried out at temperatures between 400 - 800 °C and partial oxygen conditions of 0.1-0.3% for 120 minutes. The optimal gasification for sewage sludge ash generation and partial oxygen conditions were then determined. The sewage sludge ash rich in phosphorous oxide was then sent for alkali treatment for phosphorous recovery.

2.6 Extraction of phosphorous using alkali technique (NaOH) from gasified municipal sewage sludge ash

The phosphorous was extracted from municipal sewage sludge ash using the alkali treatment technique. For the alkali extraction of phosphorous from sewage sludge as 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 for one hour under aerobic conditions thereafter, 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. Water was used to separate phosphorous which dissolves in water with other elements such as magnesium oxide that do not dissolve in water (Pares et al., 2017). The optimal heat treatment temperature was chosen at the highest yield of phosphate through evaporation. The phosphate recovered (R) was then calculated as indicated in Equation 5.

$$R\ (\%) = \left(\frac{1-(P1\ X\ W1)}{P2\ X\ W2}\right) X\ 100 \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.. (5)$$

Where: P_1 is the phosphorus content in treated sewage sludge ash, P_2 is the phosphorous content in untreated sewage sludge ash, W_1 is the amount of the treated sewage sludge ash and W_1 is the amount of the raw sewage sludge ash

2.7 Analysis of the gasified municipal sewage sludge ash

The composition of the sewage sludge ash content was determined on a percentage (%). (Samolada and Zabaniotou. 2014). The analysis of the oxides in the municipal sewage sludge was done using a Perkin Elmer AAS Model 460 atomic adsorption with flame atomisation after dissolution in acid.

3. Results and Discussion

3.1 Municipal sewage sludge characteristics

The dried municipal sewage sludge had a moisture content of 60.0% and ash content of 36.7% as indicated in proximate analysis in Table 1. The sewage sludge also had a volatile matter content of 56.2% and fixed carbon matter context of 7.1%. High calorific values and fixed carbon content values are essential in heat recovery from the sewage sludge (Yousuke et al. 2018).

Table 1. Proximate analysis of the municipal sewage sludge

Parameter	**Value (%)**
Moisture content (MC)	60.0±12.0
Volatile matter (VM)	56.2±8.4
Ash content (AC)	36.7±7.3
Fixed carbon (FC)	7.1±1.4

3.1.2 Sewage sludge ultimate analysis

The municipal sewage sludge had a carbon content of 32.8%, hydrogen content of 4.4% and a calorific value of 14.9 MJ/kg (Table 2). These properties allowed the sewage sludge to be burnt to ashes during gasification. The high carbon content in the sewage sludge was ideal for gasification of the sewage sludge (Xinyue et al., 2023). The sewage sludge chemical composition was analysed using a Thermofischer Scientific X-ray analyser.

Table 2. Sewage sludge ultimate analysis

Parameter	**Value**
Carbon	32.8±6.5%
Hydrogen	4.4±%0.9
Nitrogen	4.3±0.5%
Oxygen	22.4±4.5%
Sulphur	0.8±0.2%
Phosphorous	16.5±3.2%
Calorific value	14.9 ±7.5 MJ/Kg

3.2 Effect of gasification process conditions on municipal sewage sludge ash quality

3.2.1 Effect of gasification temperature on sewage sludge ash phosphorous oxide composition

The phosphorous oxide (P_2O_5) composition increased with increase in gasification time from 30 minutes to 120 minutes and increase in the gasification temperatures from 400 - 800 °C at constant oxygen availability of 10% (Figure 1). After that the phosphorous oxide composition became constant at all the gasification temperatures with 800 °C (Figure 1). The behaviour was 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 14.1% composition. The same trend was observed by Zhenquan Fang (2023) when phosphorous recovery rates of 20-80% were noted for temperatures ranging from 750-1000 °C.

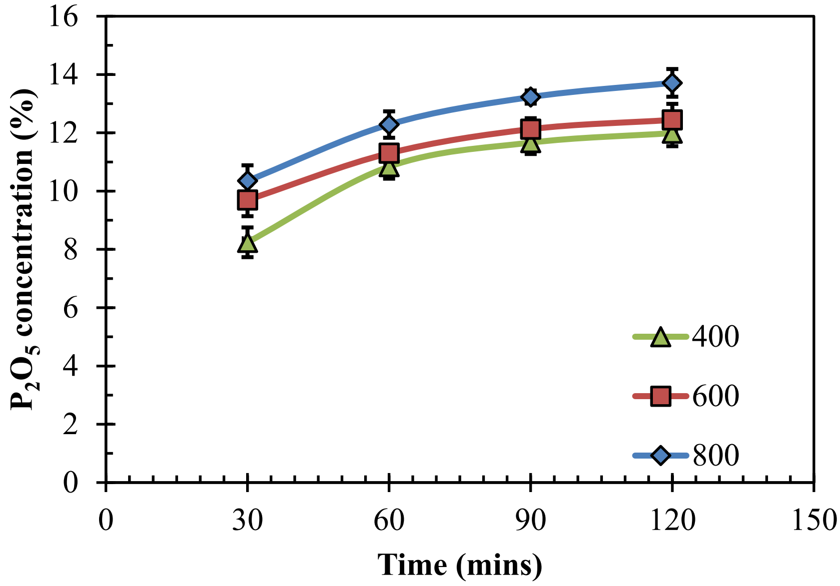

Figure 1. Effect of gasification temperatures (400-800 °C) and time on P_2O_5 composition at 10% oxygen content in sewage sludge ash

3.2.2 Effect of oxygen content during gasification

The gasification of municipal sewage sludge took place under partial oxygen conditions. From this study, complete gasification of the sewage sludge was achieved at partial oxygen concentration of 0.1% where the optimal phosphorous concentration of 14.1% was achieved (Figure 2). In all cases with increase in gasification time and the oxygen concentration changing from 0.1% to 0.3% at a gasification temperature of 800 °C steadily increased the P_2O_5 composition (Figure 2). The same trend was reported by Werle and Dudziak (2019) for oxygen concentrations ranging from 0.12-0.27%.

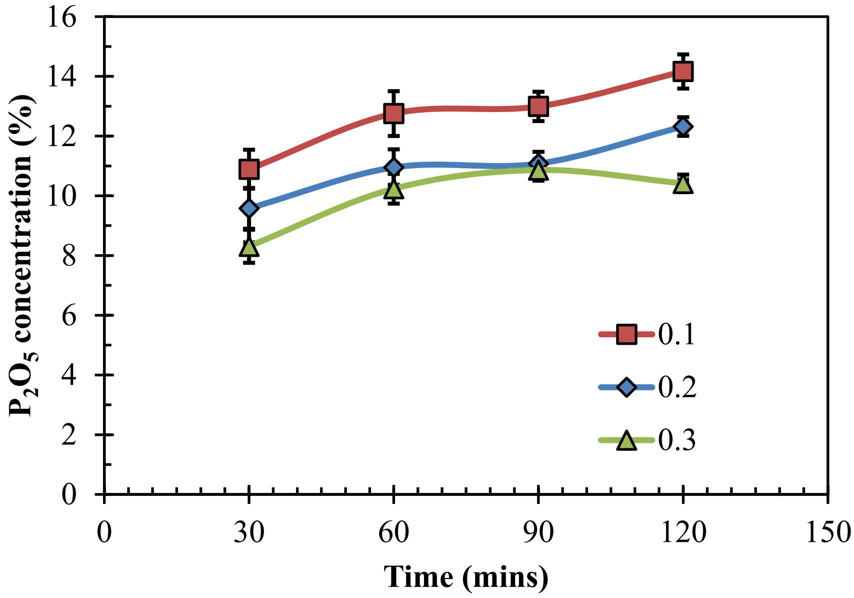

Figure 2. Effect of gasification time and oxygen concentration on P_2O_5 composition at 800 °C gasification temperature in sewage sludge ash

3.3 Process description for municipal sewage sludge gasification under optimal conditions for phosphorous oxide recovery

The municipal sewage sludge was first dewatered and dried to a moisture content of 60.0%; this was 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% oxygen. The hot gas in the process was recovered as syngas for re-use as an energy source in the plant which is an attraction for using the gasification technology. The sewage sludge ash that are rich in various metal oxides are sent for further nutrient extraction using various processes such as alkali and acidic treatment (Yimin et al., 2022). In this study, phosphorous oxide was the target oxide for phosphate extraction. The schematic diagram of the process for municipal sewage sludge gasification for further processing to recover phosphorous is shown in Figure 3.

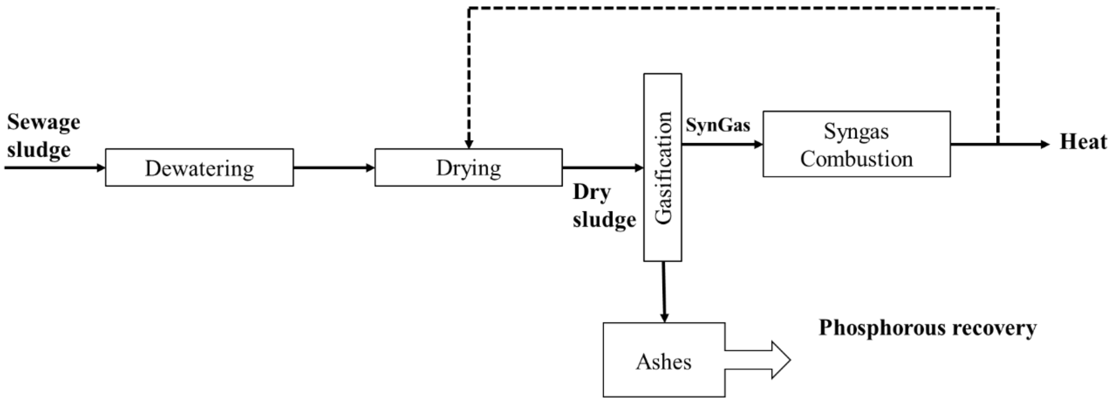

Figure 3. Process description for municipal sewage sludge gasification for phosphorous recovery with heat integration

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

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

Oxide	**Composition (%)**
Silicone oxide (SiO_2)	33.2± 6.6
Aluminium oxide (Al_2O_3)	15.5 ±3.1
Iron oxide (Fe_2O_3)	7.6±1.5
Calcium oxide (CaO)	10.7±2.1
Phosphorous oxide (P_2O_5)	13.7±2.7
Sulphur trioxide (SO_3)	2.9±0.6
Sodium oxide (Na_2O)	1.4±0.3
Potassium oxide (K_2O)	0.8±0.2
Titanium oxide (TiO_2)	2.2±0.4
Magnesium oxide (MgO)	3.3±0.7
Manganese oxide (MnO)	0.6±0.1
Loss on ignition (LOI)	8.1±1.7
Total	100.00

3.4 Phosphorous recovery from municipal sewage sludge ash at various heating temperatures

The amount of phosphorous recovered from sewage sludge ash using alkali treatment increased with the heating time applied per sample as well as increase in heating temperature from 750 °C to 850 °C (Figure 4) with about 50% of the phosphorous being recovered. The same trend was reported by Stendahl and Jäfverströ. (2002) with phosphorous recoveries of over 70% being recovered. Applying longer heating times enabled 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% low and this can be attributed to probably disintegration of the sewage sludge ash at higher temperatures.

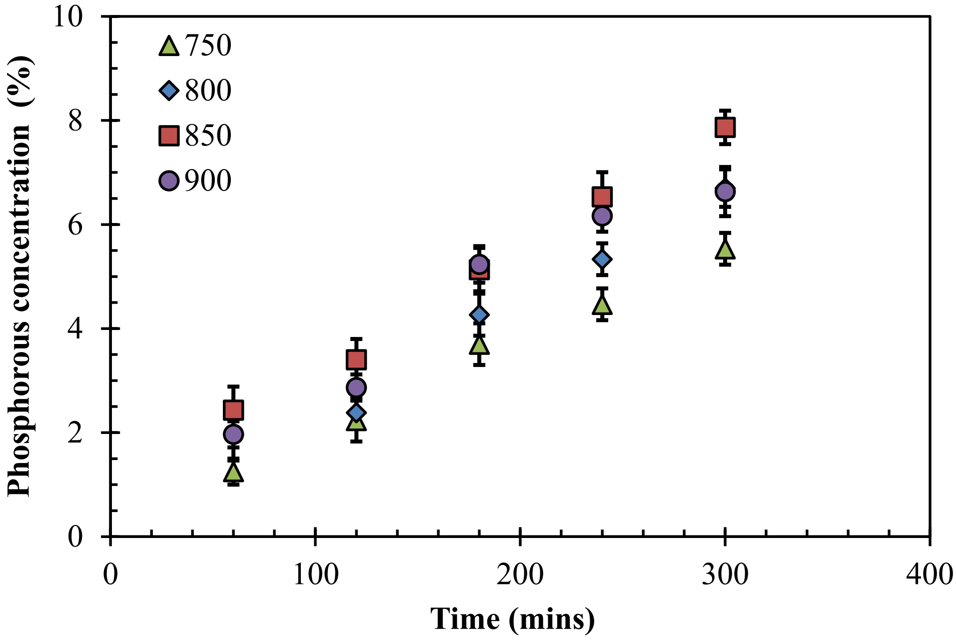

Figure 4. Effect of heating time and temperature on phosphorous recovery

3.5 Process description for phosphorous recovery from sewage sludge ash using NaOH treatment

The sewage sludge ash rich in phosphate oxide that was recovered from then gasification process underwent alkali treatment with NaOH to recover phosphorous from the ash (Figure 5). 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 to remove other salts that do not dissolve in water such as magnesium oxide. The phosphorous was then recovered as a liquid and then underwent evaporation at 105 °C in the oven. The process description for phosphorous recovery from sewage sludge ash is schematically shown Figure 5.

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

4. Conclusion

Municipal sewage sludge can be value added through adoption of various technologies for phosphorous recovery. Gasification of municipal sewage sludge is a viable technology for phosphorous oxide recovery in municipal sewage treatment plants as it also creates an option for heat integration in the process. Optimal gasification of sewage sludge at 800 °C results and partial oxygen concentration of 0.1% resulted in almost 100% recovery of P_2O_5. The phosphorous recovery through alkali (NaOH) treatment was done for sewage sludge ash of which the phosphate content of 6.5-7.5 % was recovered. This was achieved under optimum heat treatment under the alkali treatment method at 850 °C. Sewage sludge valorisation through gasification followed by alkali treatment results in high recovery of phosphorous. Further research in this area will include potential recovery of other high use minerals such as silica, magnesium and zinc.

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Conflict of interest

The authors declare that there are no competing interests.

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