Analysis of Different Methods for Reclamation of Nitrogen and Phosphorus from Sewage Lagoons

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Abstract

The need for better nutrient management has spurred efforts towards more comprehensive recycling of nutrients contained in sewage lagoons to agriculture. Research in this direction has intensified throughout the past years, continuously unfolding new knowledge and technologies. The present review aspires to provide a systematic synthesis of the field by providing an accessible overview of terminology, recovery pathways and treatment options, and products rendered by treatment. Our synthesis suggests that, rather than focusing on a specific recovery pathway or product and on a limited set of nutrients, there is scope for exploring how to maximize nutrient recovery by combining individual pathways and products and including a broader range of nutrients. The research aimed at reclaiming two useful plant nutrients from sewage sludge, which were nitrogen and phosphorus. Nitrogen was reclaimed from sewage sludge through the Kjeldahl method and phosphorus through chemical precipitation and digestion methods. The two were then analyzed for trace metals using X-ray fluorescence spectroscopy (XRF) and their functional groups using Fourier transform infrared spectroscopy (FTIR). The results found the nitrogen sample to contain 57.91% of ammonia which was greater compared to the phosphorus sample which contained 27.44% of phosphoric acid. All the samples were found to contain a lot of impurities as they had high concentration content of up to more than 80% of compounds such as sulfur trioxide and magnesium oxide. In conclusion, reclamation of both nitrogen and phosphorous from sewage sludge was no pure as expected due to presence of impurities.

Key words: *wastewater, nitrogen, phosphorus, reclamation*

1. Introduction

The rise in the world population and the increasing use of natural resources which have occurred during the last century have caused strong alterations to the environment (Rawat et al., 2011). As it is known, the world overpopulation is gradually increasing the demand for freshwater, especially in cities, also leading to a consequent increase in wastewater production. Urban wastewater treatment plants (WW-TP) play a central role in domestic and industrial wastewater management for safe water reuse or disposal (Nayak et al., 2016).

Commonly, conventional municipal plants treat wastewaters biologically through the activated sludge process, which in turn produces a waste sludge needing further treatments (thickening, digestion and dewatering). During the entire process, water, derived from treatment, is usually still enriched in nutrients, especially nitrogen (N) and phosphorus (P); thus, it cannot be directly released into natural environments or reused. As a consequence, it is normally recirculated in the WW-TP for N and P abatement before discharge (Nayak et al., 2016). In fact, N and P, once including comprehensive recycling of nutrients contained in human excreta to agriculture (Elser & Bennett, 2009). Human excreta have a long history of being used as fertilizer and organic soil amendment. However, urbanization, the introduction of water closets and sewer networks, and the growing and nowadays widespread use of synthetic fertilizers has contributed to a significant departure from this practice (Rockefeller, 1998; Ferguson, 2014).

Treatment renders a treated effluent, gaseous emissions, and a solid residual referred to as sewage sludge (in European regulations) or biosolids (in North American regulations). Land accumulated in natural water bodies (lakes, rivers, lagoons, seas), cause eutrophication, with undesirable disturbance of the balance among living organisms and, then, a reduction in water quality (Nayak et al., 2016). However, the availability of large amounts of nutrient-rich waters presents an opportunity to couple nutrient removal and biomass production; indeed, in the last few decades, wastewaters, because of their composition not only in macro but also in micro-nutrients, have been widely proposed as alternative low-cost culture media for photosynthetic microorganisms. (Rawat et al., 2011).

Typically, the recovery of N and P from wastewater has been considered in independent processes due to their chemical nature, and their simultaneous removal by sorption processes has rarely been reported. The merit of using one single material for the simultaneous removal of both ammonium and phosphate species is obvious; however, this has not yet been achieved.

Growing concern about future fertilizer availability has re-emphasized the need for better nutrient management,

application of sewage sludge is a common practice in many countries and allows for partial recycling of nutrients to agriculture. The practice has been heavily debated for a long time, due to concerns about contaminants such as pathogens, organic pollutants, and heavy metals in the sludge (Petrik, 1954; Renner, 2000; McBride, 2003; Bengtsson & Tillman, 2004)

As a result, there is a trend towards incineration of a larger portion of the sludge (Kelessidis & Stasinakis, 2012; Kirchmann et al., 2017). The adequacy and long-term sustainability of conventional urban water and sanitation systems have increasingly been called into question. For low-income countries, the high infrastructure costs are prohibitive for widespread adoption (Larsen et al., 2016). In the context of high- income countries, issues of concern include high energy and water demand, sludge disposal problems, and limited nutrient recycling (Brands, 2014). Some scholars hold on to the idea of municipal sewers and call for more comprehensive resource recovery at municipal sewage treatment plants (STPs) (Peccia & Westerhoff, 2015; Verstraete et al., 2016; Puyol et al., 2017). Other scholars hold that source separation and control provide greater opportunities for resource recovery, as it minimizes dilution and contamination of human excreta (Larsen & Gujer, 1997; Otterpohl et al., 1997; Maurer et al., 2006; Larsen et al., 2009). Approaches based on source separation and control are commonly referred to as new, ecological, resource-oriented, source- separating, or decentralized sanitation or wastewater management.

2. RESEARCH METHODOLOGY

2.1. Experimental Design

The type of experiment design used was the true-experimental research design. The experimental design took about three weeks to complete. The experiment was carried out in two phases. First, there was reclamation of phosphorus and then nitrogen where raw sewage was obtained from Father Ntheiya, Narok County for recovery of both nitrogen and phosphorus. The raw sewage was kept in clean test tubes and tightly sealed. It was then kept in Maasai Mara University Chinese laboratory, Narok, Kenya.

2.1.1. Apparatus

The apparatus to be used in the experiment were washed with water and soap to do away with dust, oil and other contaminants. They were then rinsed using distilled deionized water and dried using a clean cloth.

2.2. Sampling

Samples of wastewater sludge were taken from the father Ntheiya wastewater treatment plant, Narok North, Narok County, Kenya (*see Figure 1*) and put into clean test bottles and tightly sealed. They were then safely transported to Maasai Mara University Chinese Laboratory, Narok, Kenya and stored in the oven at room temperatures.

Figure 1: A map of the Sampling Area

2.3. Materials used

2.3.1. Materials

Sewage sludge was collected from Father Ntheiya wastewater treatment plant, Narok North, Narok County, Kenya and was used as a source of P. The plant from which the sewage sludge was collected treats wastewater from domestic and industrial premises. The wastewater goes through primary treatment, which involves both screening and passing the waste stream through large settling tanks. It is at this point that about 70% of the materials in the water tank sink to the bottom, becoming sewage sludge. The sludge is pulled from the tank, thickened by dewatering and further treated by anaerobic digestion. The digested sludge is pumped from the digester and air dried in sludge drying beds. The dried sewage sludge was then used as a source of P in this study. All chemical reagents such as sulfuric acid, hydrochloric acid and nitric acid were supplied by Rochelle Chemicals.

2.4. Equipment

A reflux apparatus was used to leach out phosphorus from sewage sludge. FTIR (Fourier Transform Infrared Spectroscopy) Thermoscientific Nicket IS10 (USA) was used to characterize the sewage sludge before and after precipitation.

2.5. Methods used

2.5.1. Total Kjehdahls Nitrogen (TKN)

Digestion

3.00g of ground sample was put into a digestion flask (W). A reagent blank and concentrated HCl added. A second subsample for lab dry matter determination was also done. 15g K_2SO_4, 0.04g anhydrous $CuSO_4$, 1.0g alundum granules were added followed by 20ml H_2S0_4. A flask was placed on pre-heated burning (adjusted to take 250mL standard water at 25^0C to boil in 5 min). It was warmed until white fumes cleared the flask, swirling gently. The sample was then cooled off and 250mL water gradually involved to cool it (Jaber et al., 2009).

Distillation

85ml of 20% HCl was put into a titration flask followed by 1.0g of alundum chips. Slowly down side of flask, 80ml 0f 45% NaOH is added. (Do not mix until after flask is linked to distillation equipment or ammonia will be lost.) Instantly the flask is linked to distillation equipment and distilled until at least 150mL distillate gathered in titrating flask.

Titration

The excess acid was titrated against 1N NaOH to orange endpoint (color consist of red to lemon to yellow). A reagent blank (B) is also titrated.

2.5.2. Total Phosphorous determination by chemical precipitation

A filter paper was weighed and stored in a desiccator. 3.000 g of sample was dissolved in 40.0 ml of distilled water and a different filter paper used to filter the mixture. 45 ml of 10% $MgSO_4.7H_2O$ was then added to the filtrate followed by 150 ml of 2M NH_3 slowly while stirring. A white precipitate was expected and the mixture allowed to stand at room temperature for 15 minutes. The precipitate was then quantitatively transferred to the pre-weighed filter paper and washed with two 5 ml portions of distilled water and two 10 ml portions of 95% ethanol. The precipitate was then spread on a watch glass for 8 hours and dried in the oven at 100^0C for 1 hour. The precipitate was again cooled for 15 minutes before reweighing.

2.5.3. Total Phosphorus Determination by Digestion Method

50 ml of sample was pipetted into a dry digestion flask. 1 ml of conc. H_2SO_4 and 5 ml of conc. HNO_3 were added. The mixture was heated slowly until there was approximately 1 ml of solution left. Digestion was to be continued carefully until the solution became clear. The solution was then cooled to room temperature. 1 drop of phenolphthalein was added. One drop of 1N NaOH was added at a time until the solution developed a pink color. The solution was diluted to 100ml with distilled water.

2.5.4. Total Ammoniacal Nitrogen (TAN) by Formaldehyde Method

3.0 g of crude sample was weighed into a clean beaker and 100.0 ml of 3M HCl acid added. The mixture was swirled to completely mix and effervescence. 4.0 ml of 24% aluminium chloride solution followed by 3 drops of methyl red indicator were added turning the mixture color to pink. After filtration, 100 ml of saturated potassium hydroxide solution (17%) was added until the color changes to yellow. 100.0 ml of 1M HCl acid was then added followed by 10.0ml of 50% formaldehyde solution. The mixture was then standardized using 0.1N NaOH solution. For the blank determination, 100 ml of 1M HCl acid and 10.0 ml of formaldehyde acid was used. The concentration of NH_3 was determined as indicated in ***Equation 1***;

$$\% \; NH_3 = (V_S - V_B) \times C \times f \times V_1/V_2 \times 14.007/W \times 100/1000$$

Equation 1

Where; V_S- sample volume, V_B- blank volume, c- concentration of titrand in mol/liter, f- ammonia factor, V_1- volume of sample solution, V_2- volume of sample solution transferred after filtration, W is weight of sample in grams.

2.5.5. Total Phosphoric Acid (TPA) by Quinoline Gravimetric Analysis

1.0 g of sample was weighed into a round bottomed flask. Catalysts 10.0 g potassium sulphate and 1.0 g of hydrated copper sulfate was added followed by 20.0 ml of concentrated sulphuric acid together with alundum boiling chips. The mixture was allowed to digest until white fumes clear the flask. Thereafter, 100 ml of distilled water was added until the color of the mixture starts to change. The total volume was recorded as V_1. 10.0 ml of this solution (V_2) was mixed with 10.0 ml of conc sulphuric acid/nitric acid mixture (1:1) before adding into 50.0 ml of Quimosiac solution (prepared using quinoline and sodium molybdate). The mixture was then filtered onto a pre-weighed filter paper. The filter paper was then dried at 220^0C for 30 minutes and reweighed again. The concentration of $T\text{-}P_2O_5$ was determined as indicated in ***Equation 2***;

$$(T\text{-}P_2O_5) \; \% = A \times 32.07 \times 100/1000 \times V_1/V_2 \times 1/W$$

Equation 2

Where A is mass of precipitate and W is weight of sample in grams.

2.6. Data Analysis

The data obtained was analyzed by statistical packages of 2019 and Microsoft office excel 2021.

2.7. RESULTS AND DISCUSSION

2.7.1. Functional Group Analysis

From the FTIR results the sample 1 showed intense peaks at 3410.13 cm^{-1}, 1632.22 cm^{-1}, 1156.61 cm^{-1}, 1031.35 cm^{-1} and other peaks at less than 1000 cm^{-1}. The peak at 3410.13 cm^{-1} is associated to an N-H stretch of an aliphatic primary amine whose appearance is medium and had an absorption range is between 3400 $cm^{-1}$to 3300 cm^{-1}. The peak at 1632.22 cm^{-1} is associated to an N-H bending of amine which occurred at an absorption range of between 1600 cm^{-1} -1300 $cm^{-1.}$ The other peaks at 1156.61 cm^{-1} and 1031.35 cm^{-1} are associated to a C-N stretch of amine with an appearance that was medium with absorption range of between 1250 cm^{-1} -1020 cm^{-1}.

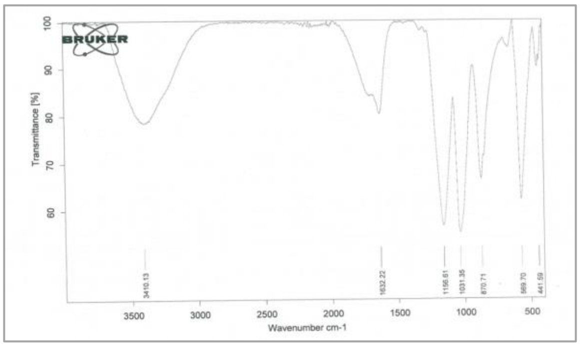

Figure 2: FTIR analysis of Nitrogen (TKN Method)

The FTIR spectrum shows an N-H stretch at 3410.13cm^{-1} showing that it could be an aromatic primary amine. There is another N-H bend of a primary amine at 1632.22 cm^{-1}. The C-N stretch appears at 1156.61 cm^{-1} and 1031.35 cm^{-1} rather than lower numbers. Therefore, the compound would be aniline which is an aromatic compound.

FTIR results from the sample 2 showed a weak peak at 2937.80 cm^{-1}, a strong peak at 1049.89 cm^{-1}, weak peaks at 993.24 cm^{-1} and 950.36 cm^{-1}, and a few peaks below 700 cm^{-1}. The weak peak at 2937.80 cm^{-1} is associated to an N-H stretch whose absorption range is between 3200 cm^{-1} to 2900 cm^{-1}. The strong peak at 1049.89 cm^{-1} is associated to a P-N-C asymmetric stretch whose absorption range is between 1110 cm^{-1} to 930 cm^{-1}. The weak peaks at 993.24 cm^{-1} and 950.36 cm^{-1} are associated to a P-N-C asymmetric stretch whose absorption range is between 1110 cm^{-1} to 930 cm^{-1}.

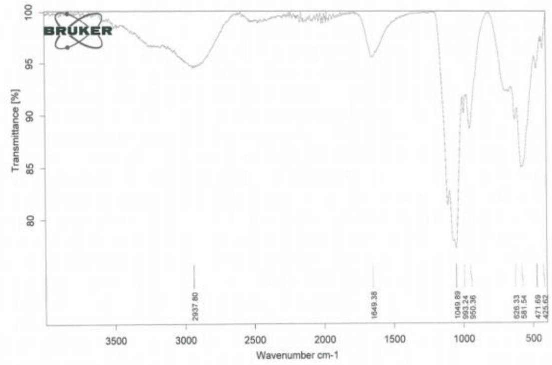

Figure 3: FTIR analysis of Phosphorus (Chemical Precipitation Method)

The peak at 2937.80 cm^{-1} is associated to an N-H stretch whose functional group might be P-NH. The other peaks at 1049.89 cm^{-1}, 993.24 cm^{-1} and 950.36 cm^{-1} are associated with an asymmetrical P-N-C stretch indicating that a P-N functional group is present.

FTIR results from sample 3 showed very strong peak at 3696.75 cm^{-}1 and a weak peak at 3642.96 cm^{-}1. There were very weak peaks at 2145.12 cm^{-1}, 2038.42 cm^{-1}, 2011.03 cm^{-1} and 1963.86 cm^{-1}. There was a strong peak at 1414.00 cm^{-1}, weaker peaks at 1189.80 cm^{-1}, 1000.58 cm^{-1}, 864.02 cm^{-1} and a few peaks below 700 cm^{-1}. The peaks at 3696.75 cm^{-1} and 3642.96 cm^{-1} were outside the absorption regions for any phosphorus compounds. The weak peaks at 2145.12 cm^{-1}, 2038.42 cm^{-1}, 2011.03 cm^{-1} and 1963.86 cm^{-1} were also outside the FTIR absorption ranges for phosphorus compounds. The peaks at 1414.00 cm^{-1} and 1189.80 cm^{-1} is associated with a P=N stretch whose absorption range is between 1440 cm^{-1} to 1100 cm^{-1}. The peaks at 1000.58 cm^{-1} and 864.02 cm^{-1} are associated with a P-N-C asymmetric stretch whose absorption is between 1110 cm^{-1} to 930 cm^{-1}.

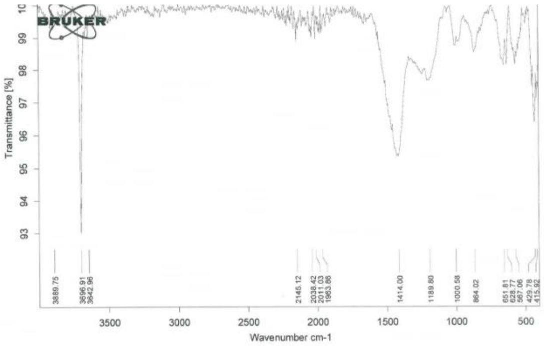

Figure 4: FTIR analysis of phosphorous (Digestion Method)

The peak at 3696.75 cm^{-1} had a sharp peak with a functional group of O-H stretch which indicated presence of an alcohol. The weak peaks *at* 2145.12 cm^{-1}, 2038.42 cm^{-1}, 2011.03 cm^{-1} and 1963.86 cm^{-1} seemed to contain noisy signals and could be as a result of another compound or the sample was contaminated. The peaks at 1414.00 cm^{-1} and 1189.80 cm^{-1} are associated with a P=N stretch showing that there is a P=N functional group of an acyclic compound. The peaks at 1000.58 cm^{-1} and 864.02 cm^{-1} are associated with a P-N-C asymmetric stretch with functional group of a P-NH.

2.7.2. X-RAY FLUORESCENCE SPECTROSCOPY

XRF results from sample 1 showed percentage concentration of three compounds. Sulphur trioxide (SO_3), potassium oxide (K_2O) and ferric oxide (Fe_2O_3). Sulphur trioxide (SO_3), was predominant with a concentration unit of 87% followed by potassium oxide (K_2O) with a concentration unit of 11.30% and ferric oxide (Fe_2O_3) with a very small concentration of 0.70%.

Table 1: Percentage concentrations of compounds present in Nitrogen (TKN Method)

Compound	**Concentration Unit**
SO_3	87.09%
K_2O	11.21%
Fe_2O_3	0.70%

Presence of SO_3 in sample 1 would be attributed to various factors. Sulfate ions could be naturally be present in the environment due to weathering of rocks and minerals containing sulfur compounds therefore finding their way to sewage sludge ponds. Also, sulfate ions could be found in domestic wastewater as a result of use of sulfate-containing products like detergents and cleaning agents.

Presence of K_2O in sample 1 would be due to naturally occurring or originate from various sources such as: food waste and residues from cooking which would contribute to their presence in sewage sludge. Small amounts of potassium compounds could enter sewage from household products such as soaps, detergents and cleaning agents containing potassium-based ingredients. Inflow and infiltration of ground water into sewage systems would also carry trace amounts of dissolved potassium compounds.

Presence of Fe_2O_3 in sample 1 would be attributed to discarding of items containing iron, such as broken appliances. Also, iron pipes, fittings and other iron-based components in sewage systems could corrode over time, releasing small amounts of iron compounds into the sewage systems.

XRF results from sample 2 showed percentage concentration of five compounds. The most abundant compound was magnesium oxide (MgO), followed by Sulphur trioxide (SO_3), then ferric oxide (Fe_2O_3), potassium oxide (K_2O) and calcium oxide (CaO). Magnesium oxide (MgO) was leading a concentration unit of 89%, Sulphur trioxide (SO_3) followed with a concentration of 9.5%. ferric oxide (Fe_2O_3), potassium oxide (K_2O) and calcium oxide (CaO) had a concentration unit of less than 1% each of 0.70%, 0.53% and 0.20% respectfully.

Table 2: Percentage concentrations of compounds present in Phosphorus (Digestion Method)

Compound	**Concentration Unit**
MgO	89.00%
SO_3	9.50%
K_2O	0.53%
CaO	0.20%
Fe_2O_3	0.70%

MgO is commonly be associated with industrial processes, construction materials and various applications. However, in some cases, magnesium compounds could find their way into sewage systems through ways such as: through usage of household cleaning products, detergents and personal care items that contain magnesium-based ingredients. If sewage sludge is used as a fertilizer in agriculture, it's possible that some magnesium compounds from sludge could make their way into the soil and finally to the sewage systems.

Presence of SO_3 in sample 2 would be attributed to various factors. Sulfate ions could be naturally be present in the environment due to weathering of rocks and minerals containing sulfur compounds therefore finding their way to sewage sludge ponds. Also, sulfate ions could be found in domestic wastewater as a result of use of sulfate-containing products like detergents and cleaning agents.

Presence of K_2O in sample 2 would be due to naturally occurring or originate from various sources such as: food waste and residues from cooking which would contribute to their presence in sewage sludge. Small amounts of potassium compounds could enter sewage from household products such as soaps, detergents and cleaning agents containing potassium-based ingredients. Inflow and infiltration of ground water into sewage systems would also carry trace amounts of dissolved potassium compounds.

Presence of Fe_2O_3 in sample 2 would be attributed to discarding of items containing iron, such as broken appliances. Also, iron pipes, fittings and other iron-based components in sewage systems could corrode over time, releasing small amounts of iron compounds into the sewage systems.

Presence of CaO would be due to its abundance in dried sewage sludge. Food residues from cooking can contribute to its presence in sewage. Calcium compounds are naturally present in soils, and some amount of calcium could be carried into sewage through stormwater runoff or other means. Discarded items containing calcium such as egg shells or some construction materials can contribute to the presence of calcium compounds in sewage sludge.

XRF results from sample 3 showed percentage concentration of five compounds. The most abundant compound was magnesium oxide (MgO), followed by Sulphur trioxide (SO_3), then ferric oxide (Fe_2O_3), potassium oxide (K_2O) and calcium oxide (CaO) tied. Magnesium oxide (MgO) was leading a concentration unit of 88.00%, Sulphur trioxide (SO_3) followed with a concentration of 11.00%. ferric oxide (Fe_2O_3), potassium oxide (K_2O) and calcium oxide (CaO) had a concentration unit of less than 1.00% each of 0.74%, 0.20% and 0.20% respectively.

Table 3: Percentage concentrations of compounds present in Phosphorus (Chemical Precipitation Method)

Compound	Concentration Unit
MgO	88.00%
SO_3	11.00%
K_2O	0.20%
CaO	0.20%
Fe_2O_3	0.74%

Presence of MgO in sample 3 could be as a result of several factors. Such as; magnesium compounds could find their way into sewage systems through ways such as usage of household cleaning products, detergents and personal care items that contain magnesium-based ingredients. If sewage sludge is used as a fertilizer in agriculture, it's possible that some magnesium compounds from sludge could make their way into the soil and finally to the sewage systems.

Presence of SO_3 in sample 3 would be attributed to various factors. Sulfate ions could be naturally be present in the environment due to weathering of rocks and minerals containing sulfur compounds therefore finding their way to sewage sludge ponds. Also, sulfate ions could be found in domestic wastewater as a result of use of sulfate-containing products like detergents and cleaning agents.

Presence of K_2O in sample 3 would be due to naturally occurring or originate from various sources such as: food waste and residues from cooking which would contribute to their presence in sewage sludge. Small amounts of potassium compounds could enter sewage from household products such as soaps, detergents and cleaning agents containing potassium-based ingredients. Inflow and infiltration of ground water into sewage systems would also carry trace amounts of dissolved potassium compounds.

Presence of Fe_2O_3 in sample 3 would be attributed to discarding of items containing iron, such as broken appliances. Also, iron pipes, fittings and other iron-based components in sewage systems could corrode over time, releasing small amounts of iron compounds into the sewage systems.

Presence of CaO would be due to its abundance in dried sewage sludge. Food residues from cooking can contribute to its presence in sewage. Calcium compounds are naturally present in soils, and some amount of calcium could be carried into sewage through stormwater runoff or other means. Discarded items containing calcium such as egg shells or some construction materials can contribute to the presence of calcium compounds in sewage sludge.

2.7.3. Total Ammoniacal Nitrogen (TAN) by Formaldehyde Method

From the results obtained, it was seen that the amount of nitrogen compounds present in sewage sludge is more than 50% i.e. 57.91 % of the compounds present in sludge. This figure is significant as the nitrogen can be of great use as a raw material for manufacture of fertilizers or can be used directly in agricultural activities.

However, the nitrogen content in sewage sludge was not close to 100% due to presence of several impurities predominant in sewage sludge. Compounds such as sulfur trioxide were found to be in greater quantities in sewage sludge. These compounds when exposed to the environment in excess quantities would pose great danger to the environment. Due to their existence in the sewage, it is therefore impossible to use the sewage sludge directly as a fertilizer in agricultural activities as the compounds present would cause environmental concerns.

2.7.4. Total Phosphoric Acid (TPA) by Quinoline Gravimetric Analysis

The value obtained from the formula is below the expected results. The % phosphoric acid found was 27.444, which was below at least 50%. This indicates that there are higher concentrations of other compounds such as trace metals in sewage sludge compared to the useful compounds like nitrogen, phosphorous and potassium. These compounds include; sulfur trioxide, ferric oxide magnesium oxide and many more. If exposed to the environment in large quantities, they would lead to environmental pollution. Therefore, use of sewage sludge directly as a fertilizer remains to be a challenge due to the presence of these impurities.

2.8. CONCLUSIONS

Reclamation of both nitrogen and phosphorus from sewage sludge were not pure as expected. They contained a lot of impurities. Therefore, the management of sewage sludge is becoming increasingly difficult due to the presence of heavy metals. Sewage sludge cannot directly be used as fertilizer due to the presence of these heavy metals which will lead to the pollution of the environment. Improving the fixation effect of heavy metals and ensuring their long-term stability are the key problems that need to be solved.

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2.11. CONFLICT OF INTEREST

The author declares(s) that there is no conflict of interest regarding the publication of this paper.

2.12. DATA AVAILABILITY STATEMENT

The author confirms that the data supporting the findings of this study are available within the article.

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