

Extraction and precipitation of phosphorous from sewage sludge: A feasibility study.

Freeman Ntuli, Thabo Falayi, Mbalenhle Mhlungu, and Portia Thifhelimbilu

Abstract—Sewage sludge from ERWAT (East Rand Water Care Company) was investigated to determine its potential as a source of P. The raw sewage sludge was found to be a potential source of P. At least 84% P could be leached from the raw sewage sludge after 60 min using either HCl or H₂SO₄. Selective precipitation of P as magnesium tetraphosphate could be achieved using ammonia and magnesium oxide to give yield a 92.2% pure magnesium tetraphosphate and 9.8% P.

Keywords—Leachate, phosphorous, precipitation.

I. INTRODUCTION

Phosphorus is an essential and irreplaceable, yet limited resource [1]. It is the limiting factor for plant growth. As early as 2035 it is calculated that the demand for phosphorus fertilizers will outpace the supply, therefore there is a need to find an alternative source of phosphorus [1]. Reserves of phosphate rock, the main source of phosphorus used in fertilizers, are running out [2]. It is therefore important to develop a sustainable extraction technique of P from any P rich resource. Sewage sludge has been widely applied to agricultural land, but due to pollutants in the sewage sludge its use has been restricted. Direct utilization of sludge as a fertilizer is hindered because of its pathogen content and high metal content [3]. Heavy metals are non-biodegradable, toxic and persistent [4]. Ashing of sewage sludge consumes energy and results in green house emissions. This project focused on P recovery from raw sewage sludge. Effects of acid type and concentration were investigated. Selective precipitation of P was also investigated.

Freeman Ntuli is with the University of Johannesburg, Chemical Engineering Technology Department (corresponding author's phone:0027 11 559 6003; e-mail: fntuli@uj.ac.za).

Thabo Falayi is with the University of Johannesburg, Chemical Engineering Technology Department (e-mail:tfalayi@gmail.com).

Mbalenhle Mhlungu was with the University of Johannesburg, Chemical Engineering Technology

Portia Thifhelimbilu was with the University of Johannesburg, Chemical Engineering Technology

II. PROCEDURES

A. Material

Sewage sludge was supplied by ERWAT and was used as a source of P. H₂SO₄ and HCl were supplied by Rochelle Chemicals.

B. Equipment

The reflux apparatus was used to leach the sludge. An ICP (GBC quantima sequential) was used to analyze the composition of the leachate. XRF (Rigaku ZSX primus II) was used for analyzing elemental composition of sewage sludge. Metal analysis was achieved using an Atomic Absorption Spectrometer (Thermo scientific ICE 3000 Series).

C. Effect of leaching time

400 ml of 1M HCl and H₂SO₄ was mixed with 10 g of ground sewage sludge (SS) and the solution was refluxed for 15, 30, 45, and 60 min.

D. Effect of Acid Concentration

The procedure in C was repeated at with 2M and 5M HCl and H₂SO₄ with the reflux time fixed at 60 min.

E. Removal of metals from leachate

Batch adsorption tests were carried out using activated attapulgit (calcined at 700°C), attapulgit and activated carbon. 5 g of each adsorbent was added to 3 different 250 ml volumetric flasks. To each volumetric flask 50 ml of leachate was added. The resulting solutions were agitated in a thermostatic shaker at 200 rpm for 3 h. After 3 h, agitation was stopped and the solution was filtered. The filtrate was then analysed for heavy metals using an AAS.

III. RESULTS AND DISCUSSION

A. Sewage sludge XRF analysis

Table 1 shows the XRF analysis of the sewage sludge

TABLE 1
THE CHEMICAL COMPOSITION OF ORIGINAL SEWAGE SLUDGE
AND ASH

Constituent	Concentration (% m/m)
P ₂ O ₅	14.6
SiO ₂	38.4
CaO	11.0
Fe ₂ O ₃	10.3
SO ₃	5.5
Al ₂ O ₃	11.9
MgO	1.7
K ₂ O	1.7
MnO	0.6
TiO ₂	1.1

The main components of sewage sludge were phosphorous, silica, iron oxide, calcium oxide and alumina (Table 1). The P₂O₅ content of sewage sludge was 14.6% making sewage sludge a potential source for phosphorous [5].

B. Effect of leaching time

Figure 1 shows the effect of refluxing time on the recovery of P from raw sewage sludge.

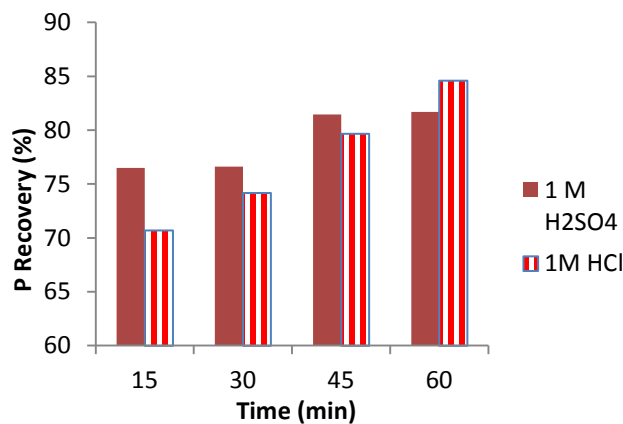


Fig.1 Variation in P recovery with time using 1M H₂SO₄ and HCl

There was a gradual increase in P recovery with time with the maximum leaching at 60 min for both HCl and H₂SO₄. The maximum recovery with HCl was slightly higher than that of H₂SO₄ (84.6 against 81.7% respectively). Using a χ^2 test at 95% confidence interval ($P_{critic}=7.81$ at 3 degrees of freedom). P_{calc} was found to be 0.88, therefore there was no significant difference in P extraction efficiency of HCl and H₂SO₄. The leaching time of 60 min was chosen for the rest of the experiments.

C. Effect of acid concentration

Fig. 2 shows the variation in P recovery with acid concentration

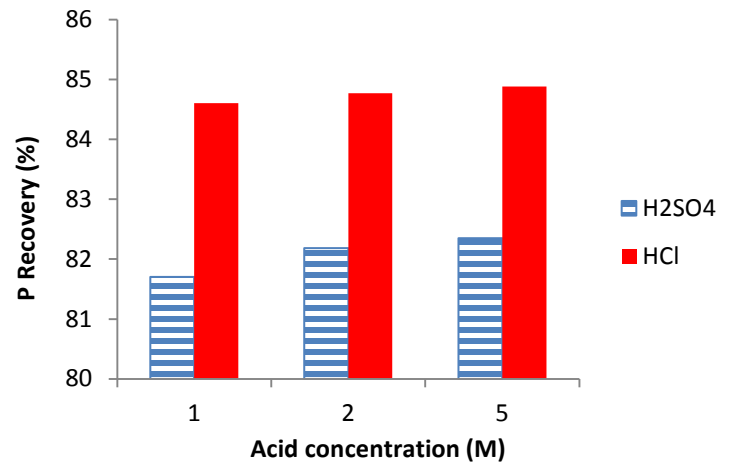


Fig. 2 Variation in P recovery with acid concentration

Fig. 2 shows no significant change in P recoveries with an increase in acid concentration. The limitation on percentage recovery (less than 85%) was due to the fact metals present acted as nucleation particles in the center matrix of the sludge [6, 7], therefore P was attached to the metals reducing the amount of P which could be extracted.

D. Removal of Metal sand Precipitation of P

P recovery from raw sewage sludge was accompanied by a corresponding leaching of metals as shown in Fig.3

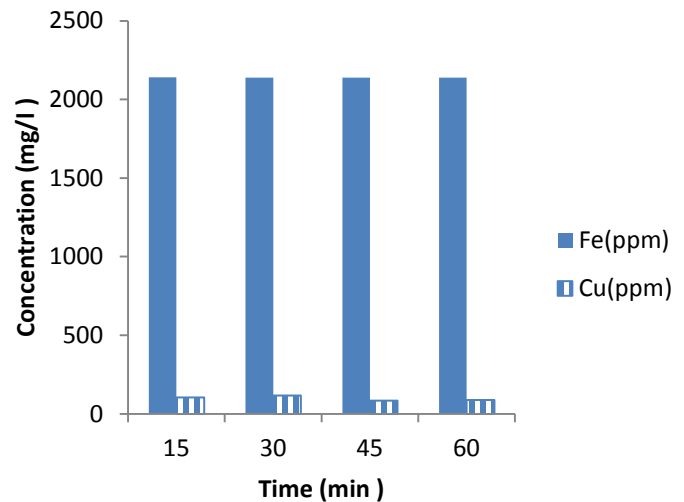


Fig.3 Co-leaching of metals with time

An average of 2100 ppm Fe and 88.9 Cu was leached from raw sewage sludge using both acids which partially exceed the limits of fertilizer ordinances [8]. Heavy metal removal using attapulgite, activated attapulgite and activated carbon was not successful as the leachate leached metals from the adsorbents due to the low pH, thus selective precipitation was the done. Ammonia was added to leachate to increase the pH from 0.5 to 2.5. The brown precipitate formed at a pH of 2.5 was

filtered and it was mainly composed of Fe^{3+} . Magnesium oxide was then added to the filtrate to selectively precipitate P at a pH of 4.5. The precipitate was then filtered and analysed using XRD. Fig. 4 shows the XRD qualitative analysis of the precipitate.

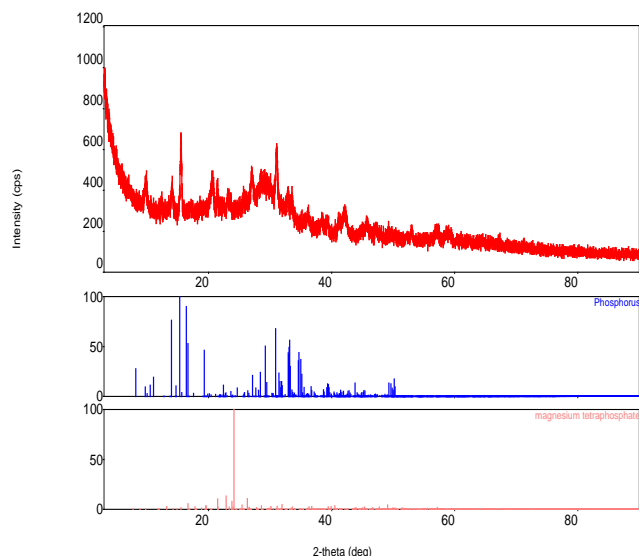


Table 2 shows the quantitative XRD analysis of the precipitate

TABLE 2
QUANTITY ANALYSIS OF P PRECIPITATE

Constituent	% m/m
Phosphorous	9.8
Magnesium tetraphosphate	92.2

The precipitate from both HCl and H_2SO_4 leachate was 92.2 % Magnesium tetraphosphate and 9.8 % Phosphorous

IV. CONCLUSION

Sewage sludge was a suitable secondary raw material for phosphorus fertilizer production, but also contained considerable amounts of heavy metals. The leaching process of phosphorus from sewage sludge was optimum at 60 min using 1M acid concentration (HCl and H_2SO_4). Increasing of the acid concentration showed no significant effect in P recovery. Selective precipitation of P resulted in a precipitate that was 9.8% phosphorus and 92.2% magnesium tetraphosphate.

V. RECOMMENDATIONS

Further test work should be done on the effect of L/S ratio on the recovery of P from raw sewage sludge. Pathogen tests should also be done on the precipitate and the bioavailability of the P precipitate should also be established. There is also a need to optimize the precipitation of P.

ACKNOWLEDGMENT

The authors acknowledge financial support from the University of Johannesburg Research Committee (URC) and ERWAT for providing raw sewage.

REFERENCES

- [1] D. Cordell, J.F. Drangert and S. White, "The Story of Phosphorus: Global Food Security and Food For Thought," *Global Environmental Change*, vol. 19, pp. 292–305, 2009.
- [2] T. Mórrigan, Peak Phosphorous, A potential Food crisis Global Climate Change, Human Security, and Democracy 2011.
- [3] C. Adam, "Techniques for P-recovery from wastewater, sewage sludge and sewage sludge ashes an overview," Presentation in BALTIC 21 Phosphorus Recycling and Good Agricultural Management Practice, September 2830, 2009.
- [4] M.R. Chaves, E. R. Dockal, R.C. Souza and P.M. Buchler, "Biogenic modified silica as a sorbent of cadmium ions: Preparation and characterization," *Environmental Technology*, vol 30, pp. 663–671, 2009.
- [5] C. Adam, B. Peplinski, M. Michaelis, G. Kley and F. G. Simon, "Thermochemical treatment of sewage sludge ashes for phosphorus recovery," *Waste Management*, vol 29, pp. 1122–1128, 2009.
- [6] B.K. Biswas, K. Inoue, H. Harada, K. Ohto, and H. Kawakita, "Leaching of phosphorus from incinerated sewage sludge ash by means of acid extraction followed by adsorption on orange waste gel," *Journal of Environmental Sciences*, vol 21, pp 1753–1760, 2009.
- [7] K. Stark, E. Plaza and B. Hultman, "Phosphorus release from ash, dried sludge and sludge residue from supercritical water oxidation by acid or base," *Chemosphere*, vol 62, pp. 827–832, 2006.
- [8] H. Xu, P. He, W. Gu, G. Wang and L Shao, "Recovery of phosphorus as struvite from sewage sludge ash," *Journal of Environmental Sciences*, DOI: 10.1016/S1001-0742(11)60969-8