

Nutrient Recycling from Septage Toward a Green Circular Bioeconomy: A Case Study in Salikneta Farm, Philippines

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Understanding the resources nexus such as food, energy, water, and land will play a crucial role in our progress towards sustainability and resilience. For example, food production is the main driver behind the excessive global use of mineral fertilisers. Though these fertilisers provide critical nutrients such as nitrogen and phosphorous, they also contribute significantly to the carbon footprint and embodied energy of the agri-food systems. In addition, these nutrients from agricultural runoff and sewage may also pollute and cause dead zones in our water bodies. On the one hand, phosphorous scarcity and food security could also pose an existential risk against the backdrop of feeding ten billion people by 2050. One emerging sustainability pathway is the green circular bioeconomy that encourages nutrient recycling from biowastes such as that agricultural waste, food waste, and domestic wastewater. Our work at Salikneta Farm demonstrates the potential of reimagining the agri-food system and transforming the sanitation system as a resource-oriented system to reduce the cost burden and losses of nutrients relative to the current existing systems. The main product from the resource recovery is a recovered P-fertiliser which can be used by farmers for crop cultivation or by social enterprises to promote a green circular bioeconomy. The pilot-scale demonstration of the proposed system at Salikneta farm indicates that processing 3.682 m³ of septage can produce 10,252 g of recovered phosphorous. The crops used to test the recovered P-fertiliser were tomato, eggplant, cabbage, and mustard, and the obtained yield was 2.06 kg/m², 2.65 kg/m², 1.39 kg/m², and 2.35 kg/m². Stakeholder engagements were also done as part of the study to raise the level of awareness and assess the acceptability of such resource-oriented sanitation systems.

1. Introduction

Nutrient management has been recently underscored as one of the emerging challenges and opportunities to feed our growing global population in the coming decades (Gerten et al., 2020). For example, a call to action for more efficient use of phosphorous (P) and its recycling is becoming widespread as the demand of phosphate-based fertilisers and their price is increasing. Phosphorous is extracted from non-renewable phosphate rocks found in a few countries such as Morocco, the United States, and China, among others (Jasinski, 2021), and about 95% of the phosphorous is utilised for food production (Daneshgar et al., 2018). In addition, the price of these fertilisers is also sensitive to geopolitical and economic risk, e.g., from the current situation brought by Ukraine-Russia conflict. The constant annual increase in the global demand for food production is taking a toll on the current global supply of such commodity fertilisers and phosphate rocks. It is estimated that the reserves

will be exhausted within 50 - 100 y, unable to meet the 70 % increase in the global food demand by 2050 (Cordell et al., 2009). There is a need to find sustainable alternative fertilisers to meet the future demands of food production.

Accordingly, this study explores nutrient recycling in the form of phosphate-based fertiliser from domestic wastewater. Recovering phosphates and other nutrients from wastewater could also alleviate the problem of “nutrient pollution” in the Philippines. For example, struvite, also known as magnesium ammonium phosphate ($MgNH_4PO_4 \cdot 6H_2O$), is a crystalline material initially discovered as nuisance deposits in wastewater treatment pipelines. It was then identified as a potential fertiliser alternative due to its nutrient content comparable to commercial fertilisers (Doyle and Parsons, 2002) and its slow-release property, which reduces the amount of phosphorous in land run-offs (Talboys et al., 2016). The recovery of struvite from wastewater treatment plants (WWTPs) can significantly reduce the demand on imported commercial fertiliser and address the food security issues of the country. Life cycle assessment showed that integrating nutrient recovery technologies in WWTPs in Metro Manila can significantly reduce the eutrophication impacts on water bodies (Pausta et al., 2020) and comply with the stringent effluent standards. An initial laboratory experiment in the Philippines conducted by Nochefranca et al. (2020) proved the potential of nutrient recovery from septage and sewage sludge. Further investigation is needed to scale-up the phosphate-based fertiliser production and to determine the impact of the recovered P-fertiliser in crop yield. This study thus aims to demonstrate the pilot-scale production of recycled P-fertiliser from septage wastewater and its farm applications. Septage is the wastewater collected from septic tanks and is more concentrated with nutrients in comparison to sewage wastewater. The results from our study were also presented in an online training with farmers organized by a civil society organization amid the COVID-19 pandemic situation in the country.

2. Methodology

The pilot-scale fertiliser production and farm applications were conducted at Salikneta Farm, San Jose del Monte, Bulacan, Philippines. The Salikneta Farm is a research farm school owned by De La Salle Araneta University. The process overview is shown in Figure 1.

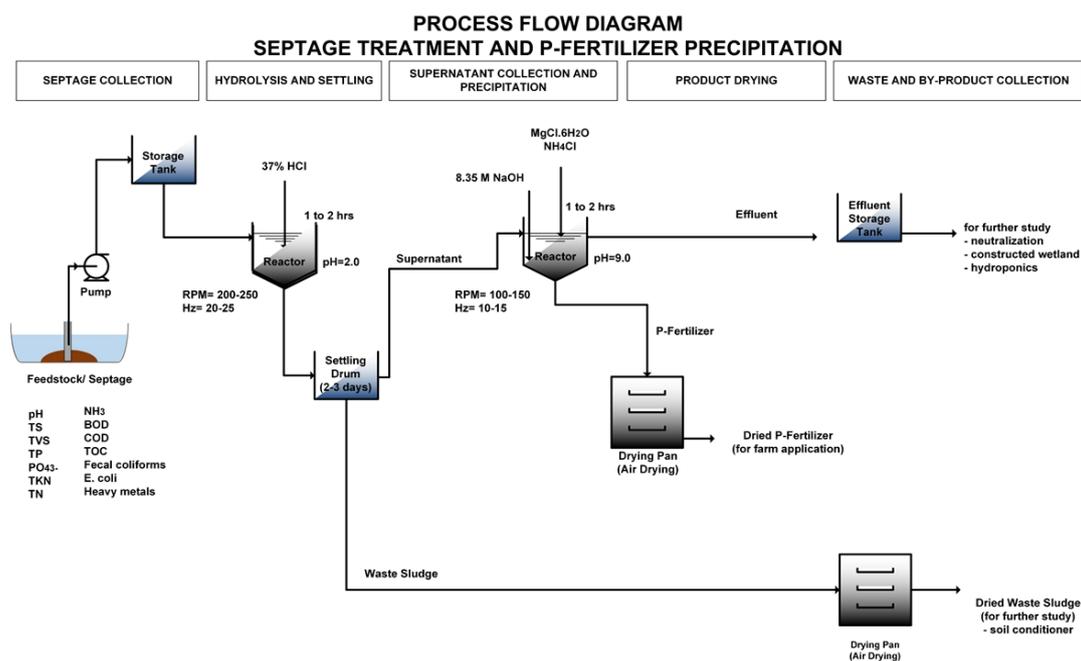


Figure 1: Process overview for recycled P-fertiliser production

2.1 Reactor design

A 0.10 m³ stainless steel batch reactor was fabricated with overall dimensions of 0.762 m (L) x 0.762 m (W) x 1.859 m (H). Its conical tank with a diameter of 0.610 m and depth of 0.644 m is supported by a stand and frame. The reactor consists of a motor drive (0.37 kW, 220/440V, 60Hz, 3-phase) and a variable frequency drive to adjust the mixer speed between 20.94 – 41.89 rad/s (0 - 60 Hz).

2.2 Sludge collection and analysis

Raw septage samples from septic tanks in Salikneta farm and from a septage treatment plant (SpTP) in Metro Manila were collected and stored in a collection drum at ambient temperature. The raw samples were analysed for orthophosphate, nitrate, and ammonium concentrations.

2.3 Sludge hydrolysis

A 0.10 m³ of raw septage is manually transferred to the pilot-scale reactor. The septage is treated with 37% w/v hydrochloric acid (HCl) and agitated at 20.94 – 26.18 rad/s for homogeneous mixing. After every 10 minutes, a small sample is collected, and its pH is measured using a benchtop pH metre (Sper Scientific, SP-860031) until pH 2.0 is achieved. The sample is continuously agitated for 1 - 2 h before collecting and storing the hydrolysed sample in a settling drum for 2 - 3 d to allow the settling of solids. The orthophosphate (PO₄³⁻), nitrate (NO₃), and ammonia (NH₃-N) concentrations of hydrolysed samples were measured using HACH DR1900 Portable Spectrophotometer.

2.4 Precipitation and drying of samples

The supernatant from the settling drum was manually collected and transferred to the reactor. Initial pH was measured to calculate the amount of 8.35M sodium chloride (NaOH) solution to be added, while 2:1 magnesium: phosphorous (Mg:P) and 4:1 nitrogen:phosphorous (N:P) ratios were used to calculate the required amount of magnesium chloride hexahydrate (MgCl₂.6H₂O) and ammonium chloride (NH₄Cl) for the precipitation process. The NaOH solution was added to the hydrolysed sample every 10 min until the pH reached 9.0 before adding the MgCl₂.6H₂O and NH₄Cl solutions. Mixing was carried out 1 - 2 h at 10.47 – 15.71 rad/s to allow the crystallisation process to occur. After overnight settling, the settled samples at the conical bottom of the reactor were transferred to a drying pan for air-drying.

2.5 Analysis

The ammonia-nitrogen (NH₃-N) and orthophosphate (PO₄-P) after the hydrolysis process were analysed using a portable spectrophotometer (Digital Reactor 1900, HACH). The raw septage, effluent, and recovered phosphate were outsourced to third-party laboratories for analysis of various parameters (wet chemistry, heavy metals, and microbiology). X-ray diffraction (XRD) and X-ray fluorescence (XRF) analysis were conducted to identify the elemental composition and mineralogical phases of the recovered P-fertiliser.

2.6 Farm application

The recovered P-fertiliser was tested on tomato, eggplant, cabbage, and mustard. The amount of fertilisers applied on the crops were based on the results of the soil analysis presented in Table 1. Recovered phosphate was applied 14 d after transplanting (DAT) for tomato and eggplant using ring and side dress method. Cabbage and mustard were applied with fertiliser during transplanting thru spot application.

Table 1: Chemical analysis of soil and fertiliser recommendation for tomato, eggplant, cabbage and mustard per 10,000 m²

Element	Unit	Soil Chemical Test	Interpretation	Fertiliser recommendation rate (kg/10,000 m ²)		
				Tomato	Eggplant	Cabbage and Mustard
Nitrogen	%	0.14	Medium	80	80	90
Phosphorous	mg/kg	1.58	Low	120	80	40
Potassium	cmol/kg	0.98	Sufficient ++	30	0	0

The fertiliser trials were laid using Randomised Complete Block Design (RCBD). The following are the treatments used in the experiment, namely Treatment 1 or no fertiliser application; Treatment 2 plants applied with Ammonium Phosphate alone, and Treatment 3 (Recycled Phosphate). The data recorded for the yield parameter were analysed using Analysis of Variance (ANOVA), and the difference among the means obtained was evaluated using Tukey's Honest Significant Difference (Tukey's HSD).

3. Results and discussion

The section discusses the wastewater and product characteristics, as well as the yield comparison between the recovered P-fertiliser, commercial fertiliser, and control (no fertiliser added). Batches 2 - 34 utilised a 90:10 ratio

of raw septage from SpTP and Salikneta, while batches 35 - 39 utilised 0.10 m³ of raw septage from SpTP since the raw septage from the Salikneta farm was fully exhausted. Initial challenges encountered were on the procurement of equipment and chemical supplies, and process optimisation to maximise P-fertiliser production.

3.1 Raw septage and effluent characteristics

The characteristics of the raw septage and effluent wastewater from the precipitation process are presented in Table 2. Due to high calcium (365.5 – 723 mg/L) and iron (107 – 307 mg/L) concentrations in the raw septage, the addition of a magnesium source is required during the precipitation process to promote the struvite formation. Low effluent PO₄-P content indicates high phosphorous recovery during the precipitation process. Lastly, based on the Department of Environment and Natural Resources (DENR) Administrative Order (AO) 2016-08 and 2021-19, all parameters except NH₃-N are compliant with the Class C General Effluent Standards (GES) (DENR, 2016; DENR, 2021).

Table 2: Characteristics of raw septage wastewater and effluent from the precipitation process

Parameter	Unit	Raw Septage Source		Effluent	
		Salikneta	Septage Treatment Plant	Process Effluent	DENR AO Class C GES
BOD	mg/L	1,325	1,459	47	50 ^a
COD	mg/L	30,750	2,648	78	100 ^a
NH ₃ -N	mg/L	77	129	21.93	4 ^b
PO ₄ -P	mg/L	7.70	7.90	< 0.20	4 ^b
Faecal coliform	MPN/100 mL	1.25 x 10 ⁶	9.20 x 10 ⁵	33	400 ^a

MPN/100 mL – Most Probable Number per 100 mL; N/A - Not available; ND - Not detected; ^aDAO 2016-08 (DENR, 2016) ^bAmended Class C effluent standards (DENR, 2021)

3.2 Fertiliser production and characterisation

The batch reactor utilised 3.682 m³ raw septage to produce a total of 10,252 g of recovered P-fertiliser. The product has 8.03% total nitrogen-phosphorous-potassium (NPK) content. The calcium:phosphorous (Ca:P), iron:phosphorous (Fe:P), and magnesium: phosphorous (Mg:P) values are calculated to be 0.89, 0.25 and 0.30, respectively. The ratios indicate higher concentrations of calcium and iron precipitates over magnesium precipitates. Heavy metal analysis showed low cadmium (3.6 mg/L) and lead (26 mg/L) contents and high zinc content (501 mg/L). Arsenic and mercury contents were below the detection limits. The microbiological analysis showed low concentrations of faecal coliform (2.5x10⁻¹ MPN/g) and Escherichia coli (2.5x10⁻¹ MPN/g). Comparing the results with the Philippine National Standards for Organic Fertilizer 40:2016 (BAFS, 2016) and the Association of American Plant and Food Control Officials (AAPFCO, 2019) concluded that the product is safe to use for farm applications. The XRF analysis showed that magnesium has the highest weight composition, followed by calcium and phosphorous. Other ions such as aluminium and iron were detected by the equipment, which imply that the recovered product contain calcium, iron, and aluminium precipitates.

Table 3: Elemental composition of the Recycled P-fertiliser using XRF analysis

Element	Composition (% weight)
Magnesium	27.04 ± 0.01
Calcium	21.26 ± 0.36
Phosphorous	16.67 ± 0.18
Aluminium	15.98 ± 0.23
Iron	9.03 ± 0.23
Silicon	5.95 ± 0.28
Sulphur	2.14 ± 0.18
Manganese	1.34 ± 0.02
Others	1.95 ± 0.06

The XRD plot in Figure 2 below detected the presence of struvite, halite (NaCl) and quartz (SiO₂) in the samples. Samples 1 and 2 are the recycled P-fertilisers from the recent batches. Sodium chloride has the highest percent weight for samples 1 (58.9%) and 2 (66.5%). Struvite has the least amount with percent weights of 27.8% in sample 1 and 18.6% in sample 2. Aside from these minerals, it is likely that amorphous matter containing calcium, iron and aluminium ions are precipitated based on the XRF analysis and the solids characterisation of the recycled P-fertiliser sample. The studies of Le Corre et al. (2005) and Yan and Shih (2016) observed that

the struvite purity and its distinct peaks in the XRD are reduced with the presence of calcium and iron concentrations. Acelas et al. (2014) demonstrated that the calcium and aluminium ions at different molar ratios inhibited the formation of struvite crystals.

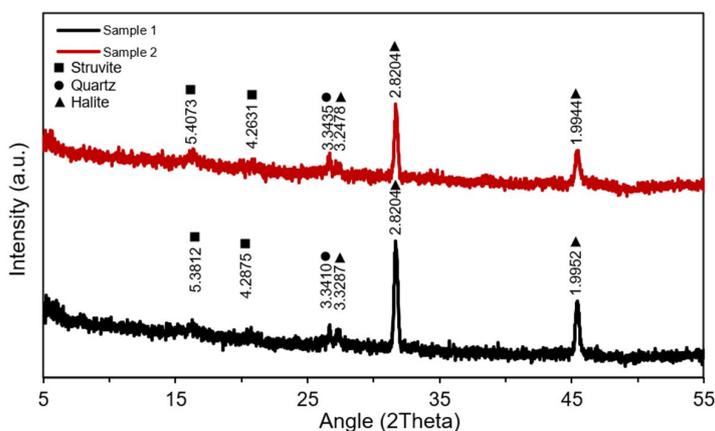


Figure 2: X-ray diffractogram of recycled P-fertiliser containing struvite

3.3 Crop yields using different fertiliser treatment

Table 4 shows that the highest yields were obtained from crops under Treatment 2 or crops fertilised with ammonium phosphate, followed by plants under Treatment 3 or crops applied with recovered phosphate. Statistical analysis shows that the means from Treatment 2 and Treatment 3 are comparable.

Table 4: Effects of different fertilisers on the yield components of tomato, eggplant, cabbage, and mustard

Treatment	Yield per plot in kg			
	Tomato	Eggplant	Cabbage	Mustard
Treatment 1 (No fertiliser)	3.26 ^b	8.99 ^b	0.89 ^b	1.43 ^b
Treatment 2 (Ammonium phosphate)	5.12 ^a	13.13 ^a	1.84 ^a	2.68 ^a
Treatment 3 (Recycled P-fertiliser)	5.18 ^a	11.69 ^a	1.53 ^a	2.58 ^a

^{a,b}Means with different letters differ significantly at the 5 % level using Tukey HSD

3.4 Engagement with the farmers

An online training which was organized by the Society for the Conservation of Philippine Wetland was held with the Filipino farmers from Arayat, Pampanga and Victoria, Laguna to increase the level of awareness on the impact of agriculture on the environment and the interrelationship of sanitation and nutrient pollution to water bodies. The training also includes the demonstration of how recovered P-fertiliser from septage can be used to grow crops such as tomatoes and eggplants. At the end of the training, a focus group discussion (FGD) was carried out to know the perception of the farmers on the recycled P-fertiliser from septage wastewater. The farmers have a positive view of the product due to its positive environmental impacts and are willing to test the said phosphate-based fertiliser on their crops. The main concern raised during the FGD is their technical capacity to produce the recycled P-fertiliser (NexCities, 2021). After the training, the research team has been continuously in contact with the farmers and are producing another batch of recovered P-fertiliser for the farmers to test on their crops.

4. Conclusions

The pilot-scale production at the Salikneta farm produced a total of 10,252 g of recycled P-fertiliser that were recovered from 3.682 m³ of septage. The analysis showed that the process recovered more than 90 % of the phosphorous from the hydrolysed septage samples. The results on the crop yield showed the comparable performance of the recycled P-fertiliser with commercial fertilisers. The positive impact of the recovered product on the farm applications in Salikneta Farm contributed to the willingness of the farmers to try the product on their crops. However, the results from the XRF and XRD analysis showed that further investigation and process optimisation are needed to improve the product quality in terms of struvite recovery. The use of alternative chemicals may be considered in order to reduce the costs of the P-fertiliser production. The preliminary cost

analysis showed that P-fertilisers from the pilot-scale production are still more expensive than the commercial fertilisers. Future works will also explore circular business models that will encourage social enterprise from such resource-oriented decentralized domestic wastewater management systems. This study thus provides an impetus for further cross-disciplinary research to map out potential resource recovery routes and business models to pave a sustainable pathway toward a green circular bioeconomy in the agri-food system.

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References

- Acelas N. Y., Flórez E., López D., 2015, Phosphorus recovery through struvite precipitation from wastewater: effect of the competitive ions, *Desalination and Water Treatment*, 54(9), 2468-2479.
- Association of American Plant Food Control Official, 2019, Statement of uniform interpretation and policy (SUIP) #25 - the 'heavy metal rule' <www.aapfco.org/rules.html> accessed 03.04.2022.
- Bureau of Agriculture and Fisheries Standards (BAFS), 2016, Philippine national standards for organic soil amendments (PNS/BAFS 40:2016) <spsissuances.da.gov.ph/attachments/article/1106/PNS-BAFS40-2014-OrganicFertilizer.pdf> accessed 30.03.2022.
- Daneshgar S., Callegari A., Capodaglio A. G., Vaccari D., 2018, The potential phosphorus crisis: resource conservation and possible escape technologies: a review, *Resources*, 7(2), 37.
- Department of Environment and Natural Resources (DENR), 2016, DENR administrative order no. 2016-08: water quality guidelines and general effluent standards <hemb.gov.ph/wp-content/uploads/2019/04/DAO-2016-08_WATER-QUALITY-GUIDELINES-AND-GENERAL-EFFLUENT-STANDARDS.pdf> accessed 30.03.2022.
- Department of Environment and Natural Resources (DENR), 2021, DENR administrative order no. 2021-19: updated water quality guidelines and general effluent standards for selected parameters <emb.gov.ph/wp-content/uploads/2021/07/DAO-2021-19-UPDATED-WQG-AND-GES-FOR-SELECTED-PARAM.pdf> accessed 30.03.2022.
- Doyle J. D., Parsons S. A., 2002, Struvite formation, control and recovery, *Water Research*, 36(16), 3925-3940.
- Gerten, D., Heck, V., Jägermeyr, J., Bodirsky, B. L., Fetzer, I., Jalava, M., Kummu, M., Lucht, W., Rockström, J., Schaphoff, S., Schellnhuber, H. J., 2020, Feeding ten billion people is possible within four terrestrial planetary boundaries. *Nature Sustainability*, 3, 200-208.
- Jasinski S. M., 2021, Mineral commodities summaries: phosphate rocks. United States Geological Survey. <pubs.usgs.gov/periodicals/mcs2021/mcs2021-phosphate.pdf> accessed 12.03.2022
- Le Corre K. S., Valsami-Jones E., Hobbs P., Parsons S. A., 2005, Impact of calcium on struvite crystal size, shape and purity, *Journal of Crystal Growth*, 283(3-4), 514-522.
- NexCities, 2021, Project Nexcities conducts virtual training for farmers <www.nexcities.org/2021/10/19/virtual-training-of-farmers/> accessed 12.03.2022.
- Nochefranca C. K. L., Oyson Z. K. C., San Pedro A. B., Pausta C. M., Damalerio R., Promentilla, M. B., Beltran A.B., Orbecido A. H., 2020, Phosphorus recovery from septage treatment plant sludge by struvite formation with alkaline hydrolysis as pre-treatment. In IOP Conference Series: Materials Science and Engineering (Vol. 778, No. 1, p. 012141). IOP Publishing.
- Pausta C. M. J., Razon L. F. Promentilla, M. A. B. Saroj, D. P., 2018, Life cycle assessment of a retrofit wastewater nutrient recovery system in Metro Manila, *Chemical Engineering Transactions*, 70, 337-342.
- Talboys P. J., Heppell J., Roose T., Healey J. R., Jones D. L., Withers P. J., 2016, Struvite: a slow-release fertiliser for sustainable phosphorus management?, *Plant and Soil*, 401(1), 109-123.
- Yan H., Shih K., 2016, Effects of calcium and ferric ions on struvite precipitation: A new assessment based on quantitative X-ray diffraction analysis, *Water Research*, 95, 310-318.