SUSTAINABLE MANURE MANAGEMENT VIA ANAEROBIC DIGESTION INTEGRATED WITH STRUVITE PRECIPITATION

BY

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ABSTRACT

The amount of livestock waste produced is monumental due to the increase in intensive agriculture. The manure management stage is one of the largest contributors to the carbon footprint of the Canadian livestock sector. To mitigate these harmful effects, various manure management strategies have been undertaken. Anaerobic digestion (AD) is one of the methods used to treat manure sustainably, producing biogas (which has multiple applications) and digestate, a valuable by-product. Historically, digestate was applied to farmlands as a source of fertilizer. However, excessive application of digestates on agricultural lands leads to environmental problems: eutrophication, greenhouse gas (GHG) emissions, heavy metal, and pathogen contamination.

To diminish the mentioned problems, the focus has shifted to finding alternate methods to treat these digestates that can reduce their impact but also give value-added products. Struvite precipitation is one such method wherein the produced crystals can be applied as a fertilizer. The struvite crystals contain good levels of magnesium, nitrogen, and phosphorus (elements crucial for a crop's growth), with other benefits such as relatively low solubility, compact storage, easy transportation, lower GHG emissions, lower soil and water contamination due to reduced leaching while giving the same effect as conventional fertilizers in crop yield. Furthermore, struvite crystal production has been underutilized using agricultural waste streams which contain a considerable amount of nutrients that can be recovered and recycled, especially anaerobic digestates of nitrogen-rich livestock waste streams.

This thesis aims to ascertain struvite production in an agricultural scenario from anaerobic digestates of varying compositions to encourage valorizing wastes for the recovery of nutrients and to implement sustainable circular economic principles. This is done by first devising a protocol to validate the conditions: pH 9, 1.5 Mg/P, and 240 rpm. Digestates obtained from the co-digestion

of different wastes and mono-digestion of poultry manure were initially tested, where 3 sets of trials were done to see the efficacy of different phosphorus salts on nitrogen recovery and mass production. Nitrogen (N) recovery of ≥95% was achieved for all the digestates, also causing a substantial volatile fatty acids reduction. While the recovery was on par, the mass of crystals produced varied with the digestates, with a range of 0.33g/10 ml to 1g/10 ml being produced.

To advance the idea of bio-economic sustainability, integration of AD with struvite precipitation is proposed. To determine its feasibility, the efficiency of high solids anaerobic digester using co-digested wastes was assessed for one representative cycle of operation of 77 days with more emphasis on the struvite precipitation, which was conducted at the end of the cycle to maximize recovery of nutrients and energy. The AD presented good results, with a maximum specific methane yield of 1.26 L CH_4/g $COD_{s\ fed}$ for D1 (poultry and dairy manures and corn silage) and 1.49 L CH_4/ $COD_{s\ fed}$ for D2 (poultry, dairy, swine manures and corn silage), which was achieved at the end of the respective operation cycle. The N recovery was very high (around 98-99%), producing 0.67 g/10 ml for all the phosphorus salts. The overall results of this study show that high N recovery is possible in co-digested wastes when struvite precipitation is used, with further opportunities to scale up and integrate this with AD to create a closed nutrient loop cycle.

The goal is to show that struvite has immense potential in the agricultural sector, promoting a circular economy as it maximizes energy and nutrient production. The advantage of the struvite compound outweighs the direct usage of digestate as a fertilizer. Detailed research can be pursued to scale up the integration process and assess its capabilities as a fertilizer.

RÉSUMÉ

La quantité de déchets d'élevage produite est monumentale en raison de l'augmentation de l'agriculture intensive. L'étape de gestion du fumier est l'une des plus importantes contributions à l'empreinte carbone du secteur de l'élevage Canadien. Pour atténuer les effets néfastes de ces déchets, diverses stratégies de gestion du fumier ont été entreprises. La digestion anaérobie (DA) est l'une des méthodes utilisées pour traiter le fumier. Cette méthode durable produit du biogaz (qui a de multiples fonctions) et le sous-produit qu'est le digestat. Historiquement, le digestat était appliqué sur les terres agricoles comme source d'engrais. Cependant, une application excessive des digestats entraîne des problèmes environnementaux : eutrophisation, émissions de gaz à effet de serre (GES), contamination par des métaux lourds et des agents pathogènes.

Afin de réduire les problèmes mentionnés, on a commencé à rechercher des méthodes de traitement de ces digestats qui peuvent non seulement réduire leur impact, mais aussi donner des produits utiles. La précipitation de la struvite est l'une de ces méthodes, les cristaux produits pouvant être utilisés comme engrais. Les cristaux de struvite contiennent de bons niveaux de magnésium, d'azote et de phosphore (éléments cruciaux pour la croissance d'une culture) ; avec d'autres avantages tels qu'une relativement faible solubilité, des cristaux compacts pour un stockage et un transport facile, une réduction des émissions de GES, une réduction de la contamination du sol et de l'eau en raison de la diminution du lessivage, tout en ayant le même effet que les engrais conventionnels sur le rendement des cultures. En outre, la production des cristaux de struvite a été sous-utilisée dans l'emploi des flux de déchets agricoles qui contiennent une quantité considérable de nutriments qui peuvent être récupérés et recyclés, en particulier les digestats anaérobies des flux de déchets d'élevage riches en azote.

Ce mémoire vise à déterminer la production de struvite dans un scénario agricole à partir de digestats anaérobies de compositions variées afin d'encourager la valorisation des déchets pour la récupération des nutriments et de mettre en œuvre des principes économiques circulaires durables. Pour ce faire, il faut d'abord mettre au point un protocole permettant de valider les conditions : pH 9, 1,5 Mg/P et 240 rpm. Des déchets co-digérés de compositions variées et du mono-digestat de volaille ont été initialement testés - 3 séries d'essais ont été faites pour voir l'efficacité de différents sels de phosphore sur la récupération de l'azote et la masse produite. Une récupération de l'azote (N) de ≥95% a été obtenue pour tous les digestats entraînant aussi une réduction substantielle des acides gras volatils. Alors que la récupération était égale, la masse de cristaux produite variait avec les digestats ; une gamme de 0,33g/10 ml à 1g/10 ml de cristaux étant produite.

Pour faire avancer l'idée de la durabilité bioéconomique, l'intégration de la DA avec la précipitation de struvite est proposée. Pour déterminer sa faisabilité, l'efficacité d'un digesteur anaérobie à haute teneur en solides utilisant des déchets co-digérés a été évaluée sur une période de 77 jours, en mettant l'accent sur la précipitation de struvite qui a été effectuée à la fin du cycle pour maximiser la récupération des nutriments et de l'énergie. La DA a donné de bons résultats, avec des rendements spécifiques en méthane compris entre 1,26 L/g $DCO_{s\ alimenté}$ pour D1 (les fumiers de volaille et de bovin et ensilage de maïs) et entre 1,49 L/g $DCO_{s\ alimenté}$ pour D2 (les fumiers de volaille, de bovin, de porc et ensilage de maïs). La récupération d'azote a été très élevée (environ 98-99%), produisant 0,67 g/10 ml pour tous les sels de phosphore. Les résultats globaux de cette étude montrent qu'une récupération élevée d'azote est possible dans les déchets co-digérés lorsque la précipitation de struvite est utilisée ; avec d'autres possibilités de mise à l'échelle et d'intégration de la DA pour créer un cycle fermé de nutriments.

L'objectif final est de montrer que le struvite a un immense potentiel dans le secteur agricole, en favorisant l'économie circulaire, car il maximise la production d'énergie et de nutriments. Les avantages de la struvite l'emportent sur l'utilisation directe du digestat comme engrais. Des recherches approfondies pourront être menées à l'avenir afin de mettre à l'échelle le processus d'intégration et d'évaluer ses capacités en tant qu'engrais.

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THESIS FORMAT AND CONTRIBUTION OF AUTHORS

This thesis structure is in accordance with the manuscript-based thesis format approved by the Faculty of Graduate and Postdoctoral Studies, McGill University, and follows the conditions outlined in their Guidelines: Concerning Thesis Preparation.

As such, following this guideline, this thesis contains the following chapters:

Chapter I: Introduction, including the hypothesis and objectives of the work presented in this thesis.

Chapter II: Literature review, where the potential of struvite produced using livestock waste streams is discussed. Furthermore, factors pertaining to its production are also mentioned.

Chapter III: Sustainable Recovery of Nutrients from Anaerobic Digestates via Struvite Precipitation.

Chapter IV: Integration of Anaerobic Digestion with Struvite Precipitation to Circularity in Manure Management.

Chapter V: General Summary and Conclusion.

Below are the manuscripts which are published:

Nagarajan, A., Goyette B., Raghavan, V., Bhaskar, A., & Rajagopal, R. (2023). Nutrient recovery via struvite production from livestock manure-digestate streams: Towards closed loop bio-economy, Process Safety and Environmental Protection, Volume 171, Pages 273-288, ISSN 0957-5820, https://doi.org/10.1016/j.psep.2023.01.006.

The other 2 chapters (Chapter III and Chapter IV) are being prepared for publication.

The content comprising these manuscripts- review of literature, experimental work, analysis of results and preparation of manuscripts was carried out by Anita Nagarajan. Prof. G.S. Vijaya Raghavan (Supervisor), from the Department of Bioresource Engineering, Macdonald Campus of McGill University, and Dr. Rajinikanth Rajagopal (Co-Supervisor), Research Scientist, Agriculture and Agri-Food Canada (AAFC), Sherbrooke Research and Development Centre (RDC) supervised the work carried out primarily at AAFC, Sherbrooke RDC plus in editing the manuscripts. Manuscripts were reviewed by all the co-authors listed.

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ABBREVIATIONS

AD	Anaerobic Digestion
°C	Degree Celsius
Ca	Calcium
CH_4	Methane
CHP	Combined Heat and Power
COD_s	Soluble Chemical Oxygen Demand
COD_t	Total Chemical Oxygen Demand
CS	Continuous Stirred
CSTR	Continuous Stirred Tank Reactor
DM	Dry Matter
EDTA	Ethylenediaminetetraacetic acid
FBR	Fluidized Bed Reactor
FS	Free Solids
g	Grams
GHG	Greenhouse Gas
H_3PO_4	Phosphoric Acid
HCl	Hydrochloric Acid
HSAD	High Solids Anaerobic Digester
HRT	Hydraulic Retention Time
K	Potassium
kg	Kilogram
KH_2PO_4	Potassium Phosphate
MAP	Magnesium Ammonium Phosphate
Mg	Magnesium
$MgCl_2$	Magnesium Chloride
N	Nitrogen
N.A.	Not Available
$NaHPO_4$	Sodium Phosphate
NaOH	Sodium Hydroxide
OLR	Organic Loading Matter
OM	Organic Matter
P	Phosphorus
pH	Potential of Hydrogen
rpm	Revolution/Rotation per Minute
SEM	Scanning Electron Microscope
SMY	Specific Methane Yield
SRT	Solid Retention Time
TA	Total Alkalinity
TAN	Total Ammonical Nitrogen
TKN	Total Kjedahl Nitrogen
TS	Total Solids
TVFA	Total Volatile Fatty Acids
UASB	Upflow Anaerobic Sludge Blanket Reactor
VFA	Volatile Fatty Acids
VS	Volatile Solids
WW	Wastewater
WWTP’s	Wastewater Treatment Plants

CHAPTER I

INTRODUCTION

Based on 2014 estimations, there were approximately 19.6 billion poultry, 1.43 billion cattle, 0.98 billion swine and 1.87 billion sheep/goats (Robinson et al., 2014); this number projected to increase with demand caused by population growth. With these numbers, it is discernible the amount of manure that is produced per year. Manure management is thus essential to ameliorate environmental problems.

Anaerobic digestion (AD) is one of the sustainable manure management solutions, is a naturally occurring process, but when controlled and optimized, produces a high quantity of methane (Al Seadi & Lukehurst, 2012) which is used for various energy related purposes like heat, electricity, fuel, and fertilizer (Leiva et al.,2014; EPA,2014). Moreover, using manure for AD production in agriculture results in recycling nutrients, reducing GHG emissions and producing fossil-free biogas energy (Holm-Nielsen et al., 2009; Hou et al., 2017). AD is very proficient; about 20-95% of organic matter can be removed depending on the process used and waste characteristics (Moller and Müller, 2012). In AD, the upside of co-digestion of manures with other energy dense compounds is that not only does it increase the organic load, improve methane production and degradation efficiency, but it does not have any noticeable impact on hydraulic retention time (HRT) (Mata-Alvarez et al., 2014; Sondergaard et al., 2015; Ahlberg-Eliasson et al., 2017; Ma et al., 2020).

Post digestion, the digestate produced is around 90-95% of what was initially fed into the digester and is highly rich in nutrients (Lamolinara et al., 2022). The common way of disposing digestate is land application as a fertilizer in agriculture which minimizes environmental problems and encourages the economic sustainability of biogas (Iacovidou et al., 2013). However, if its application is misused (i.e., overapplied), it can impact the plant growth and soil ecosystem (Rigby & Smith, 2013) and also cause eutrophication due to leaching of N and P, which can create dead zones (Conley et al., 2009).

In an attempt towards sustainability, research is being focused on the recovery and recycling of nutrients, since the supply of resources for N fertilizer production and P rock reserves are finite and diminishing in nature (Dawson et al., 2011). It is important to note that recovery and reuse of 1 ton of both N and P results in many benefits, such as decrease in water and air pollution, GHG emissions, dependency on P reserves and natural gas for N production, environmental pollution because of reduced mining and related activities, plus reduced reliance on imported fertilizers due to nutrient supply diversification. (Buckwell et al., 2016).

As aforementioned, the benefits of nutrient recovery herald a pathway to sustainable agriculture. While the role of AD is indispensable, the digestate still has value that can be further valorized. Methods that are generally considered are physical adsorption, chemical precipitation, and biological uptake (Tran et al., 2014; Güiza et al., 2015). As part of chemical precipitation methods, struvite crystallization is one of the most promising methods as it recovers both N and P at the same time, supporting the agricultural nutrient loop (Muhmood et al., 2019). Struvite is an equimolar magnesium ammonium phosphate (MAP) compound with a molecular weight of 245.43 g/mol with decreasing solubility as it goes from acidic to alkalinity (Chirmuley, 1994).

While there are articles and review papers discussing the usage of agriculture wastewater for struvite production (Kumar & Pal, 2015; Darwish et al., 2016; Rahman et al., 2014; Kataki et al., 2016; Muhmood et al., 2019; Siciliano et al., 2020); there are few studies that concern themselves with using anaerobic digestates for struvite production. To be even more specific, there are not many studies that focus on the co-digestion of multiple agricultural wastes, including N-rich poultry manures.

As inferred from above, there is potential in doing studies on assessing struvite production from co-digested wastes and poultry manure digestate. The characteristics of manure and the digestate makes it less likely to find one set of conditions that is universally applicable. As such, optimal conditions stated in various studies give an idea of the possible ranges for best struvite production. These conditions not only affect crystal production, but also % N recovery and the mass produced as discussed in the upcoming chapter.

Hence, to gain a deeper understanding of struvite production as a nutrient recovery step after the anaerobic digestion process, this thesis has several objectives:

- To initially evaluate struvite production from different types of anaerobic digestates. The purpose is to see if the conditions applied (pH 9, 3:2 Mg:P and 240 rpm) to the process not only ensures struvite formation but also yields a good percentage of % N recovery.

- To develop a protocol for struvite production and test its efficacy using different phosphorus salts- to determine if there is any variation in the recovery of nitrogen that could help to refine the process.

- To determine the potential of a closed-loop bioeconomic system by integrating AD with struvite precipitation, with an emphasis on the struvite characteristics to show that maximum nutrient and biogas recovery is feasible.

CHAPTER I-II CONNECTING STATEMENT

An overall gist was given of the current need to search for alternative fertilizers and the potential of using agricultural wastes, mainly the anaerobic digestates of livestock manure as a source in Chapter I. With this overview, we will now elaborate on the potential of using livestock waste streams, factors, methods, and studies done in the production of struvite in Chapter II.

CHAPTER II

Nutrient Recovery via Struvite Production from Livestock Manure-Digestate Streams: Towards Closed Loop Bio-Economy

2.1 Abstract

Phosphorous and Nitrogen are key nutrients for plants growth, usually supplemented in the form of fertilizers. Therefore, management and utilization of these compounds in a sustainable manner for agriculture is of importance to serve the rising global demand of food. One way is to valorize agricultural wastes in many different ways, like anaerobic digestion, struvite production and so on to maximize nutrient recovery and reduce wastage. Struvite recovery is one of the green marketing tools in the fertilizer industry, given the high amount of agriculture and livestock wastes produced. While struvite can be produced from a wide range of wastewater, this article provides an overview about struvite with an emphasis about its production from anaerobic digestates, manure and livestock wastewater and its prospective as a source of fertilizer. Furthermore, discussions about integration with anaerobic digestion, cost benefits and post application plant yields are reviewed to show its practicality and commercial potential. Despite constraints, struvite production promotes circular bioeconomic, sustainable process with a high nutrient recovery and this review will aid to take decisions in implementing this method in the near future.

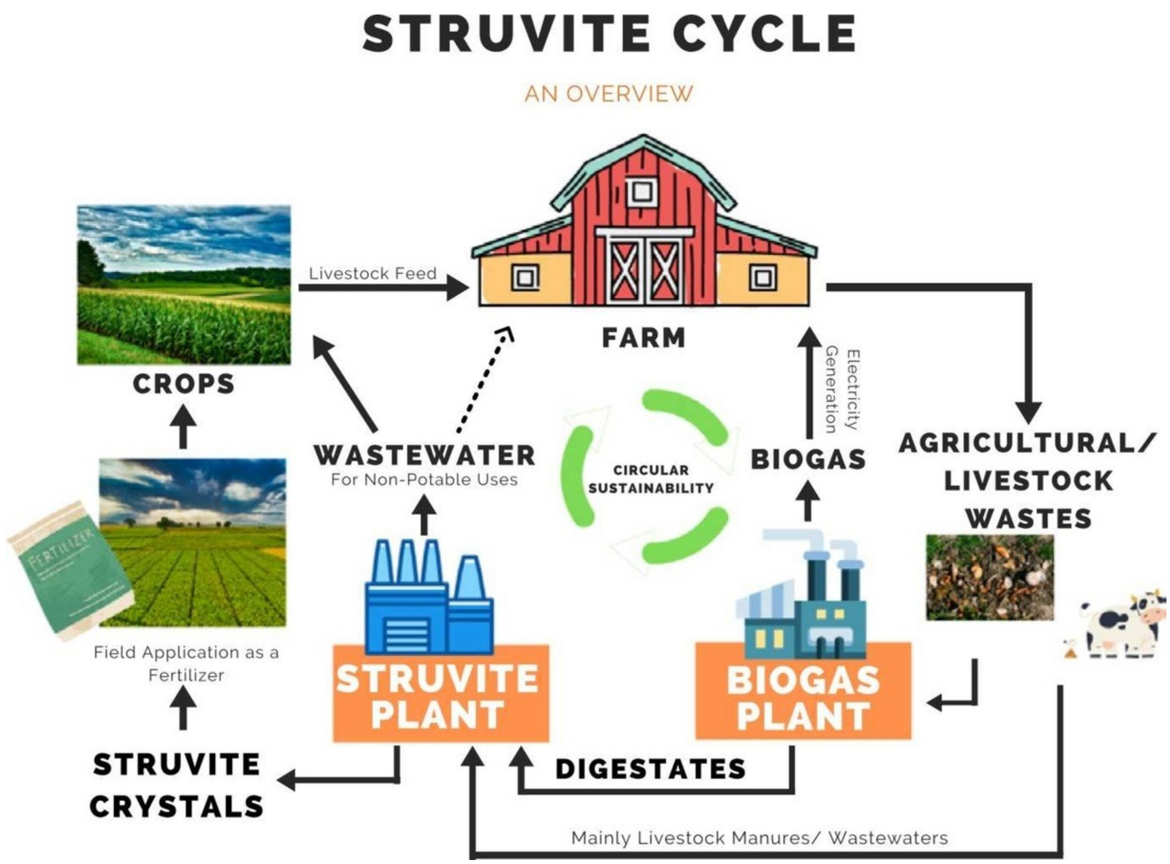

Figure 2.1 Graphical abstract of the struvite cycle

Keywords: Nutrient Recovery, Circular Bioeconomy, Struvite, Anaerobic Digestion, Waste Management, Fertilizer

2.2 Introduction

Animal manure is a versatile, readily available substance that can be used for various purposes ranging from biogas production to valorization producing valuable by-products. However, misuse and ineffective management can cause intensification of nutrients/toxins in the soil (Fijalkowski et al., 2017), greenhouse gas (GHG) emissions (Holm-Nielsen et al., 2009), leaching of nutrients (Lamastra et al., 2018) and other environmental problems in the long run. Especially in cold countries such as Canada, where unmanaged livestock manure contributes to a non-point source pollution (Duchemin & Hogue, 2009). For example, in 2019, about 7.9 Mt CO2 eq was produced from manure management in Canada (ECCC, 2021).

Animal manure is used directly as a fertilizer or as a substitute due to its rich content of nutrients. However, it is not the only source of fertilizers. Around 4946 k tonnes of NPK was used in Canada and global nutrient consumption was approximately 199,881 k tonnes of NPK in 2020/2021 (Nutrien, 2022). Unbalanced distribution of global phosphate rock reserves (Withers et al., 2015) coupled with projected increased usage due to population increase propels us to find new ways to recover and recycle P that can provide the same quality of current fertilizers.

One way to treat animal manure is to subject them to anaerobic digestion, a commonly used method which produces biogas which can be utilized (Romero-Güiza et al., 2014). But anaerobic digestion is only 13–65 % efficient while converting organic matter, which means the digestate is still concentrated with nutrients (Monlau et al., 2015). Digestate can be processed further to produce recyclables like fiber products, fertilizers, and clean water (Holm Nielsen et al., 2009). While many methods are present to recover the remaining nutrients, Struvite, $MgNH_4PO_4.6H_2O$ (MAP-Magnesium Ammonium Phosphate) whose removal by chemical precipitation from ammonia/phosphate rich wastewater has increasingly seen a demand. This has been mainly commercialized for municipal/industrial wastewater (Burns et al., 2003) as the formation of struvite in those cases affects the quality of the process (Ye et al., 2010). Attention has now shifted towards source recovery from livestock wastes, especially from livestocks: Swine, Dairy and Poultry (Balaman, 2019).

Recovery of struvite as a dry fertilizer is more suitable for its application, storage and transportation (Rech et al., 2020). Application of MAP (struvite) in the agriculture sector is a profitable investment (Hao et al., 2013). In fact, generating 1 kg of MAP per day is enough to fertilize 2.6 ha of arable land (Kumar & Pal, 2015). Studies have also shown that struvite is more effective than commercially used phosphorus fertilizers (González-Ponce et al., 2009; Szymanska et al., 2019a). Further, integration and implementation of struvite recovery with circular economic models encourages our transition towards sustainability and ensures sustainable production. This provides a multitude of benefits but also improves manure management thus reducing costs and amount of waste produced in the future.

The possibilities of struvite seem promising. Using this method can reduce the impact of animal manure by providing an alternative fertilizer and thus ensuring sustainable agriculture. On the other hand, not a lot of studies have focused on livestock wastewater and their potential to produce struvite. Even less studies have been done on pathogen and heavy metal contents, GHG reduction potential and overall cost- effectiveness of this process on a larger scale using animal manure/ digestates. In this light, this paper aims to provide a comprehensive review of (i) Nutrient recovery potential from animal manure (ii) The technologies and types of manure that are commonly used to produce struvite (iii) Various benefits of producing struvite from animal manure and the associated problems of implementing this technology on a larger scale.

2.3 Animal Manure's Nutrient Recovery Potential

Total global livestock in 2018 was around 965 million cattle, 242 million pigs and 237 million chickens (FAOSTAT Analytical Brief 14, 2020) with this number projected to increase slowly but steadily. For instance, livestock will produce waste estimating to about 55 billion tonnes of manure per year being generated (Girotto & Cossu, 2017). This clearly shows the potential of using animal manure for nutrient recovery purposes. Animal manure is rich in nutrients – containing carbon, metals, nitrogen, phosphorus and minerals (Brusseau & Artiola, 2019). In fact, livestock manure reached about 125 million tonnes of N in 2018; Out of which only 34 million were treated by manure management, the rest just left in pasture or directly applied to soil (FAOSTAT Analytical Brief 14, 2020). In the case of phosphorus, 12–14 Mt of P is produced by livestock manure worldwide (Smit et al., 2009). Hence, it is not surprising that animal manure is used to enrich the soil. But given the intensification over the years, manure today tends to have other substances like residual amounts of pesticides and antibiotics that could lead to further problems like leaching to groundwater and also uptake by the crops that are grown for human consumption. Continuous application of animal manure and other organic fertilizers is increasing the problem of antibiotic resistance (Cheng et al., 2013; Fahrenfeld et al., 2014). For instance, J. Li et al. (2017) found that long term manure application can increase the levels of residual antibiotics and antibiotic resistant genes (ARG's) presence in the soil and gave suggestions to reduce the same. Also spreading of manure directly on land is now regulated by policies and legislations to manage the odor, nutrient and waste management, disposal methods, soil and water contamination and pollution (Balaman, 2019). This means that new opportunities are sought out to recover nutrients in a sustainable manner.

Many methods exist to manage manure and extract nutrients from it. Struvite is one such option. Studies have focused mostly on wastewater and then digestates from swine, dairy, or livestock. One of the popular methods for biomass conversion process is anaerobic digestion to produce biogas. The digestate produced can later be further processed to make struvite. The liquid phase can be used directly for recovery of struvite while the solid phase is first subjected to phosphorous dissolution and then recovery processes are carried out (Yilmazel & Demirer, 2011). Tables 1 and 2 both show the potential of using manure for struvite production; swine is mostly favored as a source, followed by dairy and then poultry. While studies generally focus on liquid portion, there is also a possibility of recovering struvite even without digestion- like in conjunction with compost as seen in studies of Zhang & Lau (2007) and Fukumoto et al. (2011).

2.4 Factors that Affect Precipitation of Struvite

Like most chemical processes, the production of struvite is dependent on certain factors. These can affect the quality and quantity of the struvite produced and are discussed in the following subsections. On that note, this section aims to describe the provisions for struvite precipitation regardless of the type of animal manure/wastewater used. The range of values presented for each factor depicts the most suitable conditions necessary for the precipitation of struvite.

2.4.1. Effect of pH

It is important to maintain pH, as the formation of struvite is dependent on it (Uludag-Demirer et al., 2005). Kim et al. (2016) found from their analysis that the optimal pH was between 8 and 9 and from Visual MINTEQ the pH was 7–11 for struvite formation. Muhmood et al. (2021), on the other hand found that organic substances reduce the amount recovered at pH 8–9. Tünay et al. (1997) and Huang et al. (2017), both show that the optimal pH is around the ranges of 8.5–9.3. pH around 7–7.5 gave a struvite content of 95 % showing that pH close to neutral can also produce struvite (Hao et al., 2013). While optimal range of pH is taken to be 7–11, different studies used different pH according to their experimental design for their trials as shown in the Tables 1 and 2.

2.4.2. Effect of molar ratios

It is seen that an optimum ratio of 1:1:1 (Mg:N:P) (Warmadewanthi et al., 2020) is recommended as struvite is an equimolar compound of MAP. Depending on whether N or P is to be removed, optimization of the conditions for the maximum efficiency of removal is necessary (Zhou & Wu, 2012). This is shown in the tables of struvite production using digestates, manures and wastewater, where different ratios are utilized as per their requirement as seen in Tables 1 and 2.

2.4.3. Effect of temperature

Temperature plays a key role in determining their solubility, speed of formation and morphology of the crystals. Studies show that the optimal range for struvite formation is around 25–35 °C; with 30 °C being the temperature where struvite reaches a minimum solubility (Aage et al., 1997). However, steady rise of temperature from 14 °C− 35 °C, show an increase in both the ionic activity and Struvite coefficient causing a reduction of at least 30 % in the efficiency of crystal formation (Moussa et al., 2011). Studies have shown that as temperature increased from 25 °C to 37 °C the morphology of the crystals changed from prismatic to dendritic structure (Babic-Ivancic et al., 2002). A similar pattern is also noticed in the degree of supersaturation which is discussed below.

2.4.4 Degree of supersaturation

This factor determines the probability of crystal formation. The study done by Shaddel et al. (2019) shows that increasing the supersaturation showed a remarkable difference in the morphology of the crystals, which turned from a polyhedral to rough, dendrite like structure; while supersaturation plays a key role, the pH and concentration of ammonium and magnesium ions also contribute to the final structure.

Also from the same study, one can note that at lower supersaturation levels and pH there is stronger aggregation of struvite particles. This in turn can have a difference in the purity and quality of the crystals. This factor is important to regulate as it affects the crystal size and shape- aggregation of struvite particles preferring the granular round shape which is easier to transport and use in equipment (ESPP, 2016).

Table 2-1. List of struvite production using swine manure/wastewater.

Waste	Method	Volume of Digestate Used (L)	pH	pH Control	Salts added	Molar Ratio Mg:N:P	Ca Conc. (mg/L)	Initial Conc. (mg/L)		Removal %		Comments	References
								NH_4-N	PO_4-P	N	P		
Swine	Batch Reactor	0.100	9.0	NaOH and HCl	$MgSO_4$, fermented Super Phosphate	N.A.	N.A.	589-607	21-22	55	64	• Subjected to AD ○ HRT:>60 days • Reactor Capacity: 0.100L • Mixing Speed:200 rpm for 30 minutes • Resulting struvite used for Plant trials • Supernatant in the process used for biological treatment	Luo et al. (2019)
	Batch Reactor	1	8.8	NaOH	$MgCl_2.6H_2O$	1.5:0:1.0	N.A.	2974-3907	1120-1468	40	89	• Subjected to AD • Mixing Speed: 100 rpm • 10 minutes reaction time • Various other sets of trials were done	Lee at al. (2015)
	Air-Lift Reactor	5	9	NaOH HCl	$MgCl_2.6H_2O$ KH_2PO_4	1.2:1.0:1.0	N.A.	1725-1825	226-216	95	97	• Subjected to AD • Reactor ○ HRT: 10 minutes in the mixing zone ○ HRT overall: 3 hours	Kim et al. (2016)

Batch Reactor	1	9.5	NaOH	$MgCl_2.6H_2O$ Sewage sludge ash	1.2:1.0:1.0	N.A.	2511-3771	54-68	92	100	• Ultrasonic Pre-treatment for phosphate release • Subjected to AD • Sewage Sludge Ash was used in the experiments • Experiment done at room temperature • Mixing Speed: 400 rpm • 60 minutes reaction time	Kwon et al. (2018)
Batch Reactor	1.5	9	NaOH	$MgCl_2.6H_2O$ KH_2PO_4	1.0:1.0:1.0	61	234	42	71	97	• Various set of trials done • Subjected to AD • Temperature: 25℃ • Mixing Speed: 500 rpm • 60 minutes reaction	Perera et al. (2007)
Batch Reactor	3	9	NaOH	$MgCl_2.6H_2O$ KH_2PO_4	1.2:1.0:1.2	65	296	64	71	97	• Subjected to AD • Mixing Speed:500 rpm and accumulating device 50 rpm • 900 minutes reaction time	Perera et al. (2009)
Batch Reactor	0.150	9	NaOH	$MgSO_4$ $Na_2HPO_4.12H_2O$	1.3:1.0:0.8	279	1660	209	78	6	• Recover Copper and Zinc free struvite • Subjected to AD with Maize Silage and Crushed Corn Grain • Temperature:20℃ and 35℃ • Mixing Speed: 250 rpm and 150 rpm	Vidlarova et al. (2017)

Batch Reactor	0.100	9	NaOH	$MgSO_4$	N.A.	N.A.	800	10-30	N.A.	75	• Subjected to AD ○ HRT: 56 days • 24 hours reaction time	Wrigley et al. (1992)
Batch Reactor	0.200	10	NaOH	MgO H_3PO_4	1.0:1.0:1.0	N.A.	N.A.	N.A.	77	80	• Subjected to AD with industrial and municipal biodegradable wastes • Source: Raw Slurry • 30 minutes reaction time	Taddeo & Lepisto (2015)
Batch	1000	N.A.	CO_2 Stripping	N.A.	1.79 (Mg/P)	123.6	706	40.3	90	85	• Subjected to AD • Source: Swine WW • HRT for the reactor 2.5-5.5 hours • Another set of trials done with CSTR	Song et al. (2011)
Batch Reactor	0.400	10	NaOH	$MgCl_2$ Na_3PO_4	1.0:1.2:1.0	N.A.	1013-1426	55-139	87	96	• Source: Swine WW • Mixing Speed: 400 rpm for 10 minutes then 160 rpm for 30 minutes	Zhang et al. (2012)
Electrolytic Reactor	4.0	N.A.	CO_2 Stripping.	$MgCl_2$	2 M struvite disolved. 0.5 M $MgCl_2$	N.A.	2513-2707	32.48-29.19	53	79	• Source: Swine WW • Phosphorus Dissolution and Recycling • 1.5 hour reaction time	Liu et al. (2011)
Crystallization reactor	4000	8.1	Aeration	N.A.	N.A.	83	N.A.	68	N.A.	N.A.	• Source: Swine WW • HRT of reactor- 22.3 hours and of aeration column 3.6 hours • 8 Month operation of the reactor	Suzuki et al. (2005)
CSTR	2.72(working volume of the reaction zone)	N.A.	CO_2 Stripping	$MgCl_2$	1:1 (Mg/P)	N.A.	3809.83	60.01	31.47	93	• Source: Swine WW • HRT- 4 hours in the reaction zone	Rahman et al. (2011)

											• Resulting struvite used for Soil Test	
Crystallization Reactor	4000 or 5300	7.5-8.5	NaOH CO_2 Stripping	Bittern	N.A.	255	532	72	N.A.	73	• Source: Swine WW • HRT of reactor: 16.8/22.3 hours and 2.7/3.6 hours in the aeration column • Operated for 3.5 years	Suzuki et al. (2007)
Microbial Fuel Cell	0.070 (anode chamber)	N.A.	N.A.	N.A.	N.A.	190	N.A.	110	N.A.	82	• Source: Swine WW • 76 days of operation ○ Phase 1: 49 days ○ Phase 2: 27 days	Ichihashi & Hirooka (2012)
Batch Digestion	0.400	9.0	NaOH	$MgCl_2$	1.6:1 (Mg/P)	N.A.	N.A.	572	N.A.	91	• Source: Swine WW • 24 hour reaction time • Also did a field study in a storage pond containing 14,000L of liquid swine manure ○ agitated for 50 minutes after Mg addition	Burns et al. (2001)
Jar Test	0.800	N.A.	N.A.	MgO	2.25:1(Mg:Ca) 3:1(N:P)	645	N.A.	629	N.A.	>90	• Source: Synthetic biologically treated WW • Temperature: <20°C • Mixing Speed: 90 rpm for 30 seconds during Mg addition and 24 hours • Average mixing speed: 45-90 rpm	Capdevielle et al. (2013)

											• 4 hours and 24 hours reaction time	
Composting with forced aeration	60	N.A.	N.A.	MgO H_3PO_4	15% mol/mol of initial N	N.A.	3.8 g/kg DM	N.A.	N.A.	N.A.	• Subjected to composting with Corn Stalk • Production of struvite using various Additives • Composted for 5 weeks	Jiang et al. (2016b)
Composting with forced aeration	32 and 100	7.3	N.A.	$MgCl_2.H_2O$ H_3PO_4	N.A.	N.A.	1900 mg/kg DM	3000 mg/kg DM	N.A.	N.A.	• Source: Pig Manure and mix of Pig manure and Mature Swine Compost • 76-91 days of operation	Fukumoto et al. (2011)
Batch Reactor	2	10	HCl NaOH	$MgCl_2.6H_2O$ KH_2PO_4	1.4:1.0:1.0	N.A.	1807-2119	4.6-32.8	74	83	• Subject to AD with food waste • Biological Treatment of AD digestate • Mixing Speed: 144,000 rpm for 2 minutes • 30 minutes reaction time • High removal of Copper and Zinc	Ryu et al. (2020)
Jar Test	0.300	9	NaOH	MgO	3.2:1 (Mg:P)	N.A.	2360	1591.2	N.A.	98	• Subjected to AD ○ HRT-36 days • Temperature: 35℃ (for one set of trials) • Mixing for 15 minutes • 15 minutes reaction time	Moody et al. (2009)

Crystallization reactor	20	9	NaOH CO_2 Stripping	$MgCl_2.6H_2O$	N.A.	418	4342	87.8	>85	N.A.	• Subjected to AD • Temperature: 25-36℃ • 30 minutes reaction time • Resulting struvite was used for Soil Test	Cerrillo et al. (2015)
Semi-CSTR	2	N.A.	N.A.	$MgCl_2$ $Mg(OH)_2$ KH_2PO_4	1.0:1.0:1.0	0-400	400-2300	N.A.	>80	N.A.	• Pilot plant was built on a Dairy farm with a volume of 3100L • Subjected to AD • Use of Stabilizing agent • Temperature: 37℃ • Mixing Speed: 80 rpm • HRT of reactor: 20 days	Romero-Guiza et al. (2014)
Jar Test	N.A.	10	N.A.	$MgCl_2$ NH_4Cl	0.7-2.1:1.0:1.0	67.67-82.15	N.A.	40.5-50.5	N.A.	85	• Ultrasound/H_2O_2 Digestion pre-treatment • Temperature: 24-25℃ • Mixing Speed: 55-545 rpm for 2 hours	Zhang et al. (2018)
Intermittently Aerated Reactors	10 (of one reactor)	9	NaOH	$MgCl_2$ KH_2PO_4	N.A.	N.A.	732-931	30-56	90	97	• Source: Swine WW • SRT: 20 days • Done as a pre-treatment for AD • 2 reactors and a settler of volume 5L	Ryu & Lee, (2010)
Jar Test	0.500	8-8.5	NaOH	Magnesium Pyrolysate H_3PO_4	2.5:1.0:1.0	135	1013-1426	55-139	80	96	• Source: Swine WW • Intermittent agitation for 1 min at different intervals of 0.5-8 hours	Huang et al. (2011)

											• Did another trial with Struvite pyrolysate	
Batch Jar Test	N.A.	8-10	N.A.	MgO H_3PO_4	3.0 :1.0 :1.5	N.A.	1855-4500	45-80	N.A.	N.A.	• Source: Slurry Swine WW • Done as a pre-treatment for AD • Also produced struvite from stock ammonium solution	Kim et al. (2004)
Batch Reactor	100	8.5	CO_2 Stripping	$MgSO_4$ $Mg(OH)_2$	N.A.	N.A.	N.A.	N.A.	N.A.	83	• Subjected to AD with Vegetable residues, ○ HRT-150 days ○ Solid-Liquid Separation • 2.5-6 hours reaction time depending on the Mg salt	Pintucci et al. (2017)

- HRT – Hydraulic Retention Time
- SRT- Sludge Retention Time/ Solid Retention Time
- AD- Anaerobic Digestion
- WW- Wastewater
- N.A. – Not Available
- Rpm-Revolutions per minute
- CSTR- Continuous Stirred Tank Reactor
- DM- Dry Matter
- FBR- Fluidized Bed Reactor
- CS- Continuous Stirred

Table 2-2. List of struvite production using dairy/cattle, poultry and miscellaneous manures/ wastewaters.

Waste	Method	pH	pH Control	Salts added	Molar Ratio Mg:N:P	Ca Conc. (mg/L)	Initial Conc. (mg/L)		Removal %		Comments	References
							NH_4-N	PO_4-P	N	P		
Dairy/ Cattle	CS Batch Reactor	8.5-9.2	NaOH	$MgCl_2$ $Mg(OH)_2$ Na_2HPO_4	2.2:1.0:4.8	N.A.	225-519	N.A.	95	N.A.	• Subjected to AD ○ SRT/HRT: 20 days for 1 phase ○ SRT/HRT: 2,8,10 days for 2 phase • Temperature:21-22℃ • Volume of Digestate Used::0.050L • Mixing Speed:4000 rpm for 20 minutes	Uludag-Dermirer et al. (2005)
	Batch Jar Test	9.0	N.A.	Bittern Bone Meal	1.3:1.0:1.3	N.A.	1060	450	91	99	• Subjected to AD with Food Waste (Corn Silage and Olive Oil Waste), ○ SRT:20 days • Temperature:20-22℃, • Volume of Digestate Used::0.250L • Mixing Speed: 300 rpm, • 15 minutes reaction time	Siciliano & Rosa (2014)
	Crystallization Reactor	10.0	N.A.	$MgCl_2$ Na_2HPO_4	1.6:1.2:1.0	N.A.	100-700	10-60	>89	>99	• Subjected to AD, • Volume of Digestate Used::6.69L, • Resulting struvite used for Plant trials	Gong et al. (2018)
	CSTR	9.0	NaOH	$MgCl_2$	N.A.	N.A.	3000	183	N.A.	44	• Subjected to AD • Part of a bio-refinery • Liquid portion of digestate after Sanitation was used for struvite precipitation	Szymanska et al. (2019b)
	Batch Jar Test	8.2	NaOH	$MgCl_2.6H_2O$	1.5:0:1.0	268	1229	65	N.A	80	• Subjected to AD • Volume of Digestate Used::1L • Mixing for 1 hour	Brown et al. (2018)

Batch Reactor	9	KOH	Brine	1:0.5 (Urine/Brine)	N.A.	7732	N.A.	N.A	N.A.	• Treated with Oxalic Acid to remove Calcium • Source: Cow Urine • 120 minutes reaction time, • Resulting struvite used for Plant trials	Prabhu & Mutnuri (2014)
Batch Jar Tests	8.3-9.8	NaOH	$MgCl_2.6H_2O$	0.75-1.2:1.0 (Mg:P) 1.2: 1 (Mg:N)	N.A.	N.A.	N.A.	N.A.	70.8-92.7	• Subjected to AD via UASB of 1L volume o Solid and liquid Separation • Volume of Digestate Used::0.050 L • Mixing for 5 mintues • 10 minutes Reaction Time	Rico et al. (2011)
Cone Shaped-FB crystallization Reactor	7.8	NaOH	$MgCl_2$	0-155.4 mmol/L of salt	N.A.	N.A.	N.A.	65-82	N.A.	• Subjected to AD o Solid-Liquid Separation • Acidification and Addition of EDTA to remove Calcium • A pilot study was done using the crystallizer	Zhang et al. (2010)
Jar Test	7.2	NaOH	$MgCl_2$	N.A.	1735	1405	19	N.A.	69	• Acidification pre-treatment and addition of EDTA/ Oxalic Acid to remove Calcium, • Volume of Digestate Used::0.250 L • Mixing Speed:10,000 rpm • 15 minutes reaction time	Shen et al. (2011)
Jar Test	7-11	NaOH	N.A.	N.A.	373	23.6	489	N.A.	<90	• Microwave pre-treatment, • Volume of Digestate Used:: 0.25L • Mixing Speed:20-40 rpm • 24 hours reaction time	Qureshi et al. (2008)
Jar Test	9.0	N.A.	$MgCl_2$	2:1 (Mg:P)	N.A.	5.37 mg/g DM	0.48 mg/g DM	N.A.	<80	• Microwave pre-treatment, • Volume of Digestate Used::0.10L • Mixing Speed:200 rpm	Jin et al. (2009)

	Cone Shaped-FB crystallization Reactor	8.5	KOH/NH_3	$MgCl_2$	N.A.	80	N.A.	N.A.	N.A.	<82	• 20 minutes reaction time • Subjected to AD via Axial-Mixed Plug Flow reactor • Acidification and Addition of EDTA to remove Calcium	Zhao et al. (2010)
	Jar Test	8.5-11.5	NaOH	N.A.	1.3 Mg/P	N.A.	N.A.	N.A.	24.3	93.7	• Subjected to AD, • Temperature:20-22℃, • Mixing Speed:800-900 rpm • 25 minutes reaction time	Hidalgo et al. (2016)
	Jar test	9	NaOH HCl	$MgCl_2$	1.5:1.0:1.25	347	N.A.	N.A.	56	N.A.	• Subjected to AD o Residence time: 14 days • Volume of Digestate Used: 0.100L • Temperature:20-21℃ • Mixing Speed: 180 rpm, • 60 minutes reaction time	Zeng & Li (2006)
Poultry	Continuously Stirred Batch	8.5	N.A.	$MgCl_2.6H_2O$ 75% H_3PO_4	1.5:1.0:1.0	N.A.	4495-4729	163	97	32	• Subjected to AD o Solid-Liquid Separation o Solid phase was treated by phosphorus dissolution before struvite experimentation • Volume of Digestate Used:: 0.150L • Temperature:20-21℃ • Mixing Speed:250 rpm for 30 minutes • 60 minutes reaction time	Yilmazel & Dermirer (2011)
	Continuously Stirred Batch	9	N.A.	N.A.	1.0:1.0:1.0	N.A.	1318	N.A.	85.4	N.A.	• Subjected to AD via UASB • Source: Poultry WW • Volume of Digestate Used:: 0.400L • Temperature::25℃ • Mixing for 15 minutes • 30 minutes reaction time	Yetilmezsoy & Spaci-Zengin (2009)
	Jar Test	8.5	KOH	$MgCl_2$	N.A.	301.5	1940	153.6	N.A.	>90	• Manure subjected to various assays before struvite experimentation	Rech et al. (2020)

											• Volume of Digestate Used::1L • Temperature:27℃ • Mixing for 60 minutes • 240 minutes reaction time	
	Composting	N.A.	N.A.	$MgCl_2$	1.0:1.0 (Mg:P)	N.A.	N.A.	N.A.	N.A.	N.A.	• Source: Poultry manure mixed with Sawdust and Hog fuel • Volume of Digestate Used::6L • Forced Aeration • Manual agitation every 1-4 day • Each run lasted for about 10-13 days	Zhang & Lau, (2007)
	CSTR	8.5	NaOH	$MgCl_2.6H_2O$ $NaH_2PO_4.2H_2O$ 75% H_3PO_4	1.0:1.0:1.0 1.3:1.0:1.0 1.5:1.0:1.0	N.A.	N.A.	N.A.	72.1	95.1	• Subjected to AD with Maize Silage ○ HRT: 13 days ○ Overall HRT: 78 days ○ Solid-Liquid Separation ○ Solid phase was treated by phosphorus dissolution before struvite experimentation • Volume of Digestate Used::0.150L • Temperature: 21-22℃ • Mixing Speed:250 rpm for 30 minutes • 60 minutes reaction time	Yilmazel & Dermirer (2013)
Miscellaneous WW	Continuous U-Shape Reactor	N.A.	CO_2 Degasification	$MgCl_2$	N.A.	24.6	470	96	N.A.	80-86	• Subjected to AD ○ Solid-Liquid Separation • Source: Animal Manure WW • Volume of Digestate Used::2L • Various reaction time depending on the aeration flow rate	Zhang et al. (2014)
	Jar Test	9.0	N.A.	$MgCl_2.6H_2O$ $NaH_2PO_4.2H_2O$	1.2:1.0:1.0	N.A	240	N.A.	73	N.A.	• Source: Sheep Slaughterhouse WW ○ Blood Clot was Filtered • Volume of Digestate Used::0.400L • Temperature:25℃	Yetilmezsoy et al. (2022)

Airlift reactor	9.0	N.A.	$MgCl_2.6H_2O$ H_3PO_4	1.0:1.0:1.0	N.A.	N.A.	N.A.	97.5	97.7	• Mixing Speed: 120 rpm for 15 minutes • 30 minutes reaction time • Subjected to AD • Source: Livestock WW • Volume of Digestate Used::5L • HRT-180 minutes for the reactor • 10 minutes reaction time in the mixing zone	Min & Park (2021)

- HRT – Hydraulic Retention Time
- AD- Anaerobic Digestion
- WW- Wastewater
- N.A. – Not Available
- Rpm-Rotations per minute
- CSTR- Continuous Stirred Tank Reactor
- DM- Dry Matter
- FBR- Fluidized Bed Reactor
- CS- Continuous Stirred
- SRT- Solid Retention Time
- UASB: Upflow Anaerobic Sludge Blanket Reactor
- EDTA- Ethylenediaminetetraacetic acid

2.4.5. Effect of stirring speed

A study done by Morales et al. (2019) showed that stirring rate did affect the formation of struvite, establishing that as speed increases the particle size formed decreases. While this did not affect the removal rate of P; the rate of N removed also increased with speed. The study recommended a rate of 100–200 rpm to lower energy consumption, producing larger crystals. By raising the speed from 160 rpm to 240 rpm, Liu et al. (2014), showed that precipitation efficiency of struvite increased by 33 %. On the other hand, stirring at 500 rpm or more reduced the growth of the struvite crystals (Hanhoun et al., 2011).

2.4.6. Effect of co-existing/competitive ions

The substrate contains a multitude of ions apart from N and P. Ions commonly found are Ca^{2+}, Na^{+}, K^{+}, CO_3^{2-}, HCO_3^{-}, SO_4^{2-}. Various studies have evaluated and shown that these ions negatively affect the induction time, purity, shape, and size of the struvite crystals. To prevent the influence of these ions, different procedures are used to negate their effects. Regulating pH can prevent HCO_3^{-} and CO_3^{2-} forming stable products leading to less struvite (Huang et al., 2017). Increasing the molar ratios of magnesium and ammonium can prevent sodium and potassium from forming stable compounds (Siciliano et al., 2020). Ca^{2+}, is the main ion that in high quantities severely impacts struvite precipitation (Koutsoukos et al., 2003); to counteract this, chelating agents such as Ethylenediaminetetraacetic acid (EDTA) or oxalic acid are used to neutralize the ion (Shen et al., 2011; Brown et al., 2018).

2.4.7. Chemicals that can be added for struvite formation

As stated above, molar ratios are important when it comes to producing struvite. To increase the recovery rate, salts are added to ensure the reaction takes place. Below shows a few chemicals that are commonly added during the process:

• $MgSO_4$, $MgCl_2$, $Mg(OH)_2$, and MgO (Adnan et al., 2003; Fattah et al., 2010) and low-cost magnesium sources such as bittern (Huang et al.,2014).

• H_3PO_4 and phosphate salts such as Na_3PO_4, Na_2HPO_4, or NaH_2PO_4 (Kabdaslı and Tünay, 2018)

• NaOH (Ueno and Fujii, 2001) and HCl (Ryu et al., 2020) are generally used for pH correction

In a study done by Zeng and Li (2006) showed that P removal depended on the type of Mg salt used; the order of highest removal being $MgCl_2 > MgSO_4 > MgO > Mg(OH)_2 > MgCO_3$. To summarize, while not many studies have focused on speed of formation and morphology, it commonly depends on the factors as seen in Sections 3.3 to 3.6. In addition, while crystals formed are affected by the conditions, they are also influenced by the waste used and their characteristics, causing them to be different from each other even if other conditions for precipitation are kept the same. Tables 1 and 2 also show the main factors that are considered during struvite precipitation trials.

2.5 Methods of Struvite Recovery

While factors impact the quality and quantity based on the type of methods used, they also determine the efficiency of the process. There are various methods in which one can use to produce Struvite as seen in the table below (Tables 1 and 2.). The types are often chosen depending on the type of waste used. Reactors are commonly used due to its simplicity and their versatility towards majority of the wastes handled. Reactors can also be configured according to one's needs. Removal of around 95 % N and 98 % P was observed by using an airlift reactor on continuous mode (Kim et al., 2016). Whereas, using a fluidized bed reactor in batch, 80 % P removal was obtained (Le Corre et al., 2007). Usage of reactors, usually termed as crystallization reactors, is favored as it is more feasible and accessible as seen from the Tables 1 and 2. Due to a reactor's simplicity, it can be used and modified according to the type of waste used.

Electrochemical methods can also be used to recover P. In a microbial fuel cell using swine wastewater, the Struvite crystals are formed on the cathode and can be removed directly (Ichihashi & Hirooka, 2012). Both the following studies used anaerobic sludge/digestate from domestic wastewater plants. Around 90% of the phosphorous can be removed by using a microbial fuel cell (MFC), with struvite being obtained in a single chamber MFC (Tao et al., 2015). Using a microbial electrolysis cell, it reduces P around 70%− 85% and gave struvite as its main precipitate (Cusick et al., 2014), which is less compared to the other methods. This shows the potential of using electrochemical methods that utilize anaerobic digestate from animal manure and wastewaters.

Ion exchange and Membrane technologies are generally used with municipal wastewater and will not be discussed much in detail here. Bio- mineralization is a natural process of microorganisms to strengthen their structural tissues by mineral deposition (Da Silva et al., 2000). For example, using *Shenwanella oneidensis* MR-1, H. Li et al. (2017) found that this species can not only help grow Struvite it can also control the morphogenesis of the crystal. While studies have been done, there is scope to investigate the interactions and methodology that initiates the process within them. Also, more research should be done to scale-up the bio-mineralization process in order to produce Struvite from microorganisms.

While the method used for struvite production does help in increasing its production efficiency, the conditions used play a critical role in determining the recovery of nitrogen and phosphorus from the wastes (this also depends on the wastes and their characteristics). Other methods apart from using reactors were to show that there is a rising interest in producing struvite via these processes.

2.6 Future Research Directions

So far, we have discussed the capability of using animal manure to produce struvite by considering the key factors and methods available. These topics helped to get a basic idea of struvite and its functionality. In the next section, we are going to see the implications of using struvite to reduce pollution and achieve sustainability. The topics will cover from integration with other biological processes to economic prospects.

2.6.1. Integration of anaerobic digestion with struvite precipitation

To maximize the benefits, animal manure can be subjected to pre- treatment processes to make the nutrients more accessible for further processing. Pretreatments like acid leaching, microwave treatments, chelating agents and anaerobic digestion are generally used for farm wastes. Anaerobic digestion is a reliable technology used to manage waste (De Baere, 2000). However anaerobic digestion being a biological process that utilizes the energy contained in organic compounds to produce biogas (Manyi-Loh et al., 2013), it is a preferred method as it can produce biogas which can be used for other purposes around the farm.

Anaerobic digestion has various benefits- Reduction in GHG emissions (Styles et al., 2016; Tonini et al., 2016); Reductions in water depletion, acidification, and aquatic toxicity (Van Stappen et al., 2016); Reduction in the organic carbon introduced into the soil (Hamelin et al., 2011). Using compressed biogas produced from manure and by considering the avoidance of manure storage emission, a reduction of 257 % and a 700 % lower acidification and terrestrial eutrophication impact was found (van den Oever et al., 2021).

The digestate produced from the anaerobic digestion might have other uses, and one of the ways is to produce Struvite. The digestate contains a good amount of N and P that can be recovered instead of wastage. Moody et al. (2009) showed anaerobic digestion of swine manure to increase the amount of reactive phosphorus by 26 % and magnesium ions by 254 % leading to better removal and reduction efficiencies. The baseline to be considered is integrating the anaerobic digestion process with struvite production to extract the maximum amount of nutrients from the animal manure. The focus should be on making the integration of both processes a profitable and sustainable investment.

When it comes to scale-up it is generally done with municipal and industrial wastewater, and very rarely with livestock wastewater. There are around 80 commercial plants around the world producing struvite. Struvite produced at 24 plants in the European union were analyzed to assess their quality, some of which were digestates from livestock wastewater/agricultural waste streams. (Muys et al., 2021). However, there have been pilot plants studying the production of Struvite from animal manure and digestates. De Vrieze et al. (2019), while assessing the resource recovery from pig manure saw that using anaerobic digestion and/or nutrient recovery via struvite yielded in benefits but at the same time increased the operating costs and capital expenditure when compared to technologies like composting. Further research should be done to explore the various solutions to integrate both of them at a reasonable cost to maximize benefits.

In another study done by Song et al. (2011), they created a pilot scale crystallization reactor to produce struvite from anaerobically digested swine wastewater.; This case is interesting as struvite is produced without addition of any chemicals- which is generally done in almost all studies. Using these reactors, 85% P removal and recovery was achieved with a struvite purity of more than 90 %. (Romero-Güiza et al., 2015) coupled anaerobic digestion and struvite crystallization in the same reactor, showing a decrease in ammonia concentration in all cases of Mg salts used. At the same time, it also seemed that certain Mg salts seem to reduce the bio-methane potential of the pig manure. Kwon et al. (2018) added sewage sludge ash to the anaerobic digestate of swine wastewater to remove both ammonia and phosphorus leading to a removal of more than 90% of both N and P.

ManureEcoMine a pilot installation in a study by Pintucci et al. (2017), showed that digestion of swine manure and vegetable residues lead to increase in gas production due to recirculation of digestate but allowed for about 83 % phosphorus removal showing that this can provide for fertilizers that are refined and concentrated. Hidalgo et al. (2016) compared the usage of either ammonia stripping or struvite crystallization with anaerobic digestion; and found that not only struvite removes both N and P, but also it is slightly more economical than ammonia stripping.

Cerrillo et al. (2015) also studied the integration of anaerobic digestion with struvite production and found that P availability in struvite is similar to that of ammonium phosphate fertilizers, hence showing that struvite is a good way to recover nutrient of AD of manure. A plant in USA treats 50.82 m^3/day of swine waste (from approximately 40,000 pigs) for a period of 20 years by combining anaerobic digestion and struvite precipitation (Amini, 2014; Amini et al., 2017).

Another medium pilot plant in Spain treats 274 tons/day of pig and cow manure with the end products being struvite and activated sludge (Pedizzi et al., 2018). A LiveWaste treatment plant by Lijo et al. (2018), treats 1 ton of livestock waste which combines many processes such as anaerobic digestion, solid/liquid separation, CHP and struvite precipitation. Overall, seeing that there are many pilot plants operating that manage manure and extract maximum value-based products from them; the integration of AD with Struvite production is a process well worth for valorizing manure.

The Tables 1 and 2, the phrase “subjected to AD” shows that the manure has been used for anaerobic digestion and the resulting digestate was used for struvite crystallization. As part of it, other related data such as HRT/SRT, temperature have been included wherever it was available the relevant details pertain to the conditions used for struvite production. As seen from both the tables, many studies have used anaerobic digestion and struvite crystallization together to maximize nutrient recovery.

2.6.2. K-struvite studies

Potassium is another necessary element crucial to a crop growth and is considered as the most limited macronutrient in the soil (Fernández-Lozano et al., 1999; Tarrago et al., 2016). K-Struvite is the compound formed when ammonium ions are replaced with potassium ions (Tansel et al., 2018). This leads to both removal of K and P from the substrate. This presence of potassium in the struvite ensures growth of the crops. While struvite and K-struvite are similar in nature, K-struvite needs a higher pH (around 9–11) in order to be precipitated (Xu et al., 2015).

Most studies focus on recovering struvite and find that K-struvite is also formed as a byproduct as discussed in Bao et al. (2011), and Zeng & Li (2006). This is due to high K content in pig wastewater and dairy manure water; hence co-precipitation of struvite and K-struvite might occur (Huang et al., 2011). However, care should be taken on using the right amount of dosage of K ions, as overdosing would have an adverse effect on struvite crystallization and even promote the formation of $Ca_3(PO_4)_2$ (Hao et al., 2013).

Few studies focused on only producing K-struvite. For example, Tarrago et al. (2018), showed that the best operating conditions were at pH 10 and 38 °C giving about 80 % P recovery from digested manure. While a small percentage of K-struvite is formed naturally during the process, not much attention has been given to produce it exclusively. Doing this will prevent the usage of a separate fertilizer to provide potassium for the crops. However, only producing K-struvite means there is a lack of N in the product, which can be handled by optimizing the process to produce suitable amounts of struvite and K-struvite. Furthermore, since struvite is used more as a phosphorus fertilizer than nitrogen, external nitrogen is added with struvite to ensure the proper growth of the crop. Further research is recommended to check the feasibility of producing only K-struvite from agricultural waste streams.

2.6.3. Pathogen and heavy metal removal

Apart from the production, one has to take into consideration the quality of the struvite. One way to assess its suitability as a fertilizer is to check for pathogens and heavy metals. The presence of pathogen also determines the usability of the crystals. An absence of fecal coliforms (FC) and *Salmonella* sp, helminth eggs (HE) as well as reduced levels of heavy metal content was observed in the digestates implying that it is suitable for the production of struvite crystals (Parra-Orobio et al., 2021). In a study done by Muhmood et al. (2018), struvite crystals produced from chicken slurry showed a reduction in total coliforms and E.Coli. Furthermore, the solid product was tested and shown to have no pathogens, deeming it usable as a fertilizer. Jiang et al. (2016a) found that mixing both struvite and dicyandiamide resulted in a mixture which was phytotoxin free. Also, pH is more effective in removing heavy metals than magnesium (Huang et al., 2019).

Wang et al. (2022) found that antibiotic resistant genes (ARGs) and Class 1 Integron-Integrase gene (intI1 gene) exist in manure treated soils; where planting crops could decrease the quantity of certain gene groups and could increase the risk of them entering the human body. In a study done by Muys et al. (2021) showed that the heavy metal concentration is well below the standard limit sometimes even below the detection limit. The same study showed that pathogens and micro-organisms are present in low quantities. Chen et al. (2017) found that application of struvite increased both the abundance and diversity of ARG's in the soil, rhizosphere and phyllosphere. Cai et al. (2020) also found that tetracyclines and ARG's were higher in the swine wastewater than the synthetic wastewater.

Further, there have been studies concerning the presence of antibiotics and the effect struvite has on the soil microbial community. A study by Huang et al. (2021) shows that tetracyclines, and typical antibiotics were transported via adsorption of the struvite crystals from the swine wastewater. Bastida et al. (2019) found that actinobacterial populations and *Verrucomicrobia* in the soil were impacted by the struvite application. Latifian et al. (2012) found that the salt index and heavy metal content of struvite was lower than the commercial fertilizers.

Perera et al. (2009) showed that using a stainless-steel digester enhances the purity of struvite (96 % free of heavy metals) from swine waste digestate as copper or zinc ions present in the digestate did not precipitate during the process. These show that struvite is a good choice as a use of fertilizer due to low content of pathogens and heavy metals. Further research should be done for other agricultural wastes and optimize the process for maximum removal of pathogens and micro-organisms and to reduce the presence of antibiotics.

2.6.4. Emissions study

Another aspect that is detrimental to the environment and human health is the emission of harmful gases that occur when exposing animal manure to the open air or before/after a process. Fertilizers play their role in GHG emissions- the global average emission from a P fertilizer usage is 1.36 kg CO_2 eq. kg^{-1} of P_2O_5 (Kool et al., 2012). Huge quantities of P fertilizers are used to ensure high food production. Using struvite as an alternative can cut down these emissions (Rahman et al., 2011) and promote more sustainable use of fertilizers.

In a study by De Vries et al. (2016) shows a reduction of − 0.35 kg CO_2 eq. kg^{-1} of struvite. The value may not be as high, but nevertheless it cuts down the GHG emissions. Another study done by Jiang et al. (2016b), showed that there was a 50–82 % decrease in loss of ammonia gas by subjecting pig manure to struvite production. 45–53 % NH_3 loss was seen when producing struvite during composting of pig manure (Jiang et al., 2016a).

The study done by Zhang & Lau (2007) shows that by producing struvite from poultry manure a reduction of around 40–84 % of ammonia was achieved. According to that study the variation of molar ratios impacts the amount of ammonia removed. Struvite has the capability of reducing most nitrogen-based emissions except for N_2O and reduced around 51 % of the emissions (Fukumoto et al., 2011). A study by Wang et al. (2013) showed that N loss decreased from 40.8 % to 23.3 % when struvite was formed from food waste compost. Struvite clearly shows the potential of reducing GHG emissions, leaching and volatilization when applied as a fertilizer. Further research is recommended to ascertain the possibility and to optimize the struvite production process to reduce such emissions in other conditions and wastes.

2.6.5. Effect on crop production

The main application of struvite is its use as a fertilizer. To check its feasibility, struvite should be evaluated to see whether it as effective as the commercially used phosphate fertilizers for crop growth. Also using struvite has benefits and savings that are more sustainable than using conventional phosphate fertilizers (Talboys et al., 2016). However, being a slow-release fertilizer, care must be taken that the release meets the plants requirement of phosphorus to prevent insufficient supply that can slow the plant growth (Rech et al., 2018; Do Nascimento et al., 2018).

According to a study by Szymanska et al. (2020), applying struvite resulted in a higher crop yield when compared to ammonium phosphate over the course of 2 years and the study recommended an application of struvite every 2 years. Thiessen Martens et al. (2022) showed that when struvite is applied, maximum benefits in yield and phosphorus accumulation was the greatest in the second year due to its residual levels of struvite from the previous year.

Another study by Yetilmezsoy et al. (2013), grew 4 types of medicinal plants, adding struvite from poultry manure digestate as a fertilizer and found that the application increased the fresh weights, dry weights, and heights of the plants. In addition, it was learnt that when these were used as a feed material for the guppy fish, they did not cause any acute toxicity symptoms or mortality. Arcas-Pilz et al. (2022), used struvite in hydroponic production of lettuce and pepper and found that it sustained the growth of both short- and long-term crop and reduced environmental impacts when compared to conventional mineral fertilizers. Luo et al. (2019) treated corn with a mixture of fermented superphosphate/MAP and saw that it gave a higher fertilizer efficiency and biomass yield than that of superphosphate, concluding that fermented superphosphate/MAP was a high grade fertilizer. Yetilmezsoy et al. (2018) also grew 9 medicinal plants and saw increases in total fresh weights, total dry weights, and fresh heights of plants in soils treated with struvite. Higher yield of ley was seen in the soil treated with struvite having a low concentration of manure (Rittl et al., 2019). On the other hand, few studies have talked about the nitrogen dynamics in struvite.

For example, Robles-Aguilar et al. (2020), found that ammonium present in the struvite had an impact on the growth of Lupine as compared to that grown with no fertilizer. Overall, struvite can be used as a fertilizer for crops as it gives a similar output as commonly found with the use of mineral fertilizers. While studies have been done on the leaching of struvite in soils (Rahman et al., 2011; Ahmed et al., 2018; Gomez-Suarez et al., 2020), more research could be done to understand the dynamics of the reactions to better utilize struvite as a fertilizer.

2.6.6. Economic aspect of production

Cost benefit analysis and life cycle analysis (LCA) integration is a key part to get a quality product while ensuring sustainability. Various studies have been done either to demonstrate a cost-benefit analysis or a LCA to check the feasibility of producing struvite. One of the major costs is the use of pure reagents in the process; In order to cut down the costs various low cost conventional or otherwise sources of magnesium have been studied (Siciliano et al., 2020). Various studies have shown that using such alternative Mg sources have considerably cut down costs leading to more profit. In the experiments conducted by Uludag-Demirer et al. (2005), it was found that the initial pH adjustment was unnecessary as there was no significant differences in the percentage of removal of nutrients; therefore, usage of NaOH can be excluded which results in cost reduction.

A study done by Yetilmezsoy et al. (2017), showed that a struvite production facility with a recovery rate of 98.7 % $NH4^+$-N has 6-year payback period if the product is sold at 560 Euros/ton. This can come across as reasonable but is highly dependent on the market prices, type of substrate used, and quantity being produced. Since LCA and cost benefit analysis are variable, it is hard to come with an optimal solution that can be beneficial no matter what the type of waste used for struvite production.

Although struvite recovery reduces the impact of nutrients on the ecosystem, it has its own downsides. struvite has very unclear characteristics when compared to other fertilizers. In one of the studies on struvite recovered from dairy manure, it was found that for every 1 kg of struvite recovered, it had 0.156 kg of Nitrogen (nitrogen fertilizer) and 0.583 kg of Phosphate (phosphate fertilizer) (Temizel-Sekeryan et al., 2021).

Another downside is its high investment cost coupled with uncertain ROI (Return on Investment). Developing methods with reduced operational and maintenance costs will prove useful for investors to carry forward with the processes. Struvite is also subjected to various regulations as recovery from WWTP's come under "Waste Management" leading to enormous time consumption while regular fertilizers (e.g., phosphate rock) do not have to go through all these processes of getting approvals when it comes to trade (de Boer et al., 2018). However, a study done by Yetilmezsoy et al. (2022), showed that even though struvite sales form the lowest percentage in the revenue section, the process of producing struvite reduces the pollutants from the wastewater and can be sold at 1041.30 Euros/ton to gain a net profit.

A "Multi-Waste Plant" concept introduced by Hildago et al. (2019) describes a circular economic model where integrated co-digestion of manures and other wastes to produce valuable by products and at the same time make it economically feasible. Min & Park (2021) projected struvite sales at 103% of the operating cost when coupled with use and sale of zeolite. Saerens et al. (2021) did a LCA on struvite and found economic outcome to be positive in a realistic scenario.

The overall production cost in struvite recovery is through the cost of magnesium source as it constitutes almost 75 % of the total production cost (Dockhorn, 2009). Chimenos et al. (2006) found that using a low grade MgO has major economical and practical benefits, though it has to be supplied 3–4 times the normal amount. Having a source of seawater close to the treatment unit would bring in some savings, without which its feasibility would be questionable (Molinos-Senante et al., 2011). While studies are there to generate alternative methods to cut down costs, a general guideline can be helpful to commercialize this process.

2.7 Conclusion

The paper aims to give an understanding of struvite production from animal manure for its final use as an alternative fertilizer. Mainly commercialized for wastewater, further research should be done to encourage the scale-up of struvite production from manure, given its untapped potential.

In this way, one can implement the circular economic model on farms to reduce dependence of chemical fertilizers and be more sustainable in the long run. Apart from benefiting farms, the nutrient recovery process helps to mitigate environmental problems and also ensures the safety of the food produced. Even though it has its own set of barriers in implementation like cost, technical feasibility, regulations on trade and transport, questionable aspects of safety and composition – These problems can be dealt with through planning and a deeper understanding of struvite. On the whole, the advantage of struvite is commendable to be used as a fertilizer. However, further research has be done to encourage its use on a larger scale.

CHAPTER II-III CONNECTING STATEMENT

Chapter II elaborated on the underutilized potential of using livestock waste streams, including anaerobic digestates for struvite production. With this insight, Chapter III will comprise the first stage of struvite experiments that aim to develop the conditions for maximizing recovery. This chapter will also investigate the recovery potential of N using various anaerobic digestates and other aspects of interest in struvite production.

CHAPTER III

Sustainable Recovery of Nutrients from Anaerobic Digestates via Struvite Precipitation

3.1 Abstract

Environmental problems from excessive application of either manure or digestate are a key concern to tackle when it comes to agricultural sustainability. The main drawbacks include extended storage times, which leads to the release of greenhouse gases (GHG), contamination of soil health and groundwater quality that could eventually harm human health. To overcome these problems, struvite precipitation, a chemical precipitation method, is considered to further manage waste due to its characteristics such as high purity, ease of usage and production. As a compound that was thought to be a hindrance to wastewater treatment plants (WWTP's) operation, its efficacy as a fertilizer was just recently rediscovered. To face the problems caused by fertilizer usage in the agricultural sector, especially of N and P (e.g., eutrophication); struvite being a Magnesium Ammonium Phosphate (MAP) compound, can provide both nutrients in a sizable amount. While its precipitation has been studied for a given particular waste, not many have focused on digestates obtained from mono-digestion of poultry manure and co-digestion of multi-wastes. In this paper, we focused on creating a protocol and testing its efficiency in recovering nitrogen using 3 different phosphorus salts: phosphoric acid, sodium phosphate and potassium phosphate, namely 1.5 Mg/P ratio, pH 9, 240 rpm were found to be optimal for production, leading to ≥95% N recovery respectively for D1 co-digestion of dairy, poultry, swine manures), D2 (mono-digestion of poultry manure) and D3 (co-digestion of dairy, swine manure and corn silage). Furthermore, an overall average of 61.5% reduction in VFA was observed across the digestates plus producing struvite in the range 0.33-1 g/ 10 ml of digestate used. While the type of phosphorus salts used depends on

the circumstance, for ease of utilization either sodium or potassium phosphate is recommended to be used. This process is very suitable for anaerobic digestates of various characteristics, and future focus can be initiated on the integration of this process with AD and safety assessment of the crystals for crop growth to encourage circular economic practices.

Keywords: Anaerobic digestates, co-digestion, nutrient recovery, struvite precipitation,

3.2 Introduction

Presently, the heightened scrutiny on the status of the environment is encouraging development of sustainable methods to tackle various problems faced in the agricultural sector. One of the major goals of sustainable agriculture is to search for cost-effective methods of manure management- with a focus on production of stable green fertilizers from organic waste streams (McCrakin et al., 2018). This is because the dependence on the application of animal manures as fertilizer in farmlands has created an uneven distribution of nutrient accumulation and deficiency in the lands causing environmental problems (Buckwell et al., 2016). Digestates also follow the same pattern, where accumulation of digestate in one area would lead to deficiency in another requiring transport to places in need (Rehl & Muller, 2011). In addition, digestates do cause environmental concerns, such as GHG emissions during storage, pollutants (heavy metals, pathogens) causing soil contamination with subsequent human food contamination (Sambusiti et al., 2013; Bonetta et al., 2014; Monlau et al., 2015).

While many alternatives exist, struvite is one such green fertilizer that is being studied. Struvite is an equimolar Magnesium Ammonium Phosphate (MAP) compound. Originally, its deposit was considered as an inconvenience in pipes and tanks of wastewater treatment plants (WWTP's) which in turn decreased system efficiency while increasing maintenance costs (Jaffer et al., 2002). However, its potential as a fertilizer especially for phosphorus has significantly gained interest

(Muhmood et al., 2019) due to its composition as reported by 10% magnesium, 7% ammonium, 39% phosphate and 44% crystal water by mass (Gell et al., 2011). It should also be noted that struvite has a considerable amount of Mg and N, making it an effective source for both nutrients as well (Omidire et al., 2023). This is important as Mg, N and P are important nutrients for plant growth (Li & Zhao, 2003); agriculture depends on a steady supply of nutrients such as N and P (Dawson et al., 2011). As seen from this, struvite can replace fertilizers that depend on phosphate rock as its raw source (Rahman et al., 2014) thus furthering sustainable actions of recycling N and P. In addition, struvite crystals have many advantages- odorless, granular, easy to handle, concentrated (Bouropoulos & Koutsoukos, 2000), high purity of crystals and P content, plus ease of production (De-Bashan & Bashan, 2004).

Furthermore, struvite as nutrient recovery method was initially focused upon using waste waters (Munch et al., 2001). However, given its versatility, studies are being done to a variety of wastes such as industrial wastewater, manure, and livestock slurries (Huygens et al., 2019). Struvite in these instances has shown immense potential but remains underutilized in livestock and agricultural wastes, even less when its anaerobic digestates from co-digestion and high ammonia digestates as seen from the Tables 1 and 2 in Nagarajan et al. (2023). The process, type and amount of chemical used plus the waste source can affect the composition of struvite (Antonini et al., 2012). The nature of the digestates makes it perplexing to find a given set of conditions that would yield good production over a wide range of digestates.

Hence, the objective of this paper is to ascertain struvite production in various types of digestates by using a set of pre-determined conditions. In addition, to compare differences, if any, in using a variety of phosphorus salts to produce the crystals. The end goal being to show the high potential of recovery via struvite production from anaerobically digested livestock wastes to promote its viability as a suitable fertilizer source that aids in sustainability.

3.3 Methods and Experimental Set-Up

3.3.1 Digestate collection

The anaerobic digestates were collected from 3 different digesters (In continuous operation) at AAFC, Sherbrooke Research and Development Centre with different compositions and was stored at 4°C for further physiochemical characterization as shown in Table 3.1. The compositions of the digestates are D1 (co-digestion of dairy, poultry, swine manures), D2 (mono-digestion of poultry manure) and D3 (co-digestion of dairy, swine manure and corn silage). .

Table 3-1. Characteristics of the various digestates used for struvite precipitation.

Characteristics	**D1 (Dairy Manure+ Poultry Manure+ Swine Manure)**	**D2 (Poultry Manure)**	**D3 (Dairy Manure + Swine Manure+ Corn Silage)**
pH	7.37	7.72	7.15
Alkalinity (mg $CaCO_3$/L)	12336.0	19353.0	6763.0
TAN (mg/L)	2608.0	4168.0	1198.0
TKN (mg/L)	3373.0	4806.0	2467.0
COD Total (mg/L)	23862.0	15443.0	46393.0
COD Soluble (mg/L)	5897.0	11786.0	913.0
% Total Solids (TS)	2.3853	1.9605	3.7173
% Volatile Solids (VS)	1.3217	0.7790	2.7918
% Free Solids (FS)	1.0636	1.1816	0.9255
VFA (mg/L)	2474.0	2039.0	150.0
Acetic C2	1756.3	191.7	113.9
Propionic C3	614.9	1720.6	11.7
Isobutyric	21.2	12.0	11.8

Butyric C4	9.7	0.0	2.7
Isovaleric	57.4	104.6	0.0
Valeric C5	5.6	0.0	0.0
Caproic C6	8.7	9.8	9.5

3.3.2 Experimental set-up

3 sets of trials were done with each sample done in quadruplicate split as 2 experiments to ensure replicability. All the experiments were done at room temperature and used 30 mL of anaerobic digestate. To supplement the reaction, magnesium and phosphorus salts were added in a ratio of 3:2 (Mg:P) i.e., 1.5 Mg/P - where 1M $MgCl_2$ (constant throughout the trials) and 1M for KH_2PO_4, Na_2HPO_4 and H_3PO_4 were added respectively to the 30 mL of digestate. Furthermore, all experiments were stabilized at pH 9.0 and 1M of NaOH and 1M HCl were used as required to adjust the pH. Mixing was done using a magnetic stirrer at 240 rpm and pH was measured using a pH meter. Mixing was done for a further 3 minutes after pH stabilization to ensure complete mixing. After this, the samples were left overnight for the precipitate to settle completely. It was then vacuum filtered, and the resulting residue kept in the oven at 105°C overnight then taken out. Both the filtrate and the powder were then analyzed accordingly.

3.3.3 Methods for digestate characterization

Standard APHA protocols (Eaton et al., 2005) were used to analyze the Total and Volatile Solids. pH (measure of acidic/basic nature) and alkalinity (acid neutralization capacity) were measured using Mettler Toledo AG 8603 pH, SevenMulti (Schwerzenbach, Switzerland) and Titralab AT1000 Series (Hach Lagne Sarl,Hach, Switzerland). TAN (amount of ammonia/ammonium) and TKN (total amount of nitrogen present) were determined by macro-Kjeldahl methods (Eaton et al., 2005) using the 2460 Kjeltec Auto-Sampler System (FOSS, Sweden). VFA was prepared according to the protocol given by Massé et al. (2003) then analyzed using Perkin Elmer gas chromatograph (Model Clarus 580, Perkin Elmer, Shelton, CT, USA). Closed reflux colorimetric method was used to determine CODs (Eaton et al., 2005).

3.3.4 Validation of chosen parameters: optimal Mg/P ratio, pH and mixing rate for struvite production

While struvite can be produced in a 1:1:1 ratio but it is difficult to put them into practice due to the nature of the wastes (Zhang et al., 2009). For example, Perera et al. (2007) produced struvite using a 1:1:1 ration but only recovered 71% of nitrogen. Using the extensive review done by Nagarajan et al. (2023), the appropriate parameters with the optimal values of 1.5 Mg/P, pH 9, and 240 rpm were established and were found to be suitable for treating the digestates. As amounts of Mg and P are quite lower compared to N in manures (Muhmood et al., 2019); salts of Mg and P must be supplemented for formation of struvite and removal of N (Siciliano et al., 2020); which lowers the risk of eutrophication and N_2O formation. Discussion is still ongoing as to whether increase in Mg or P concentration (salt) increases the % N recovered (Zhou & Wu, 2012; Li et al., 2012; Siciliano et al., 2013) though it is recommended to dose a Mg/P ratio above stoichiometric values (Kim et al., 2016); as such a median ratio was taken, 1.5 Mg/P in this case to prevent overdosing. pH 9 is considered as the optimal pH for struvite precipitation which reduces ammonia production thus ensuring struvite formation (Doyle et al., 2002). A stirring rate of 240 rpm was chosen to not affect crystal growth and showed positive results in struvite formation (Liu et al., 2014). Hence, the parameters used for this study gave a good % N recovery as discussed in the sub-section below.

3.4 Results and Discussion

3.4.1 Nitrogen recovery from various phosphorus salts.

One of the key elements present in struvite and required by plants is nitrogen. Hence, its recovery is as important as phosphorus. As seen from Figure 3.1, there is a very high recovery of N, about ≥95% across all the 3 types of digestates, no matter the type of phosphorus salt used. However, there were very slight differences observed in the % N recovery when various phosphate salts were used.

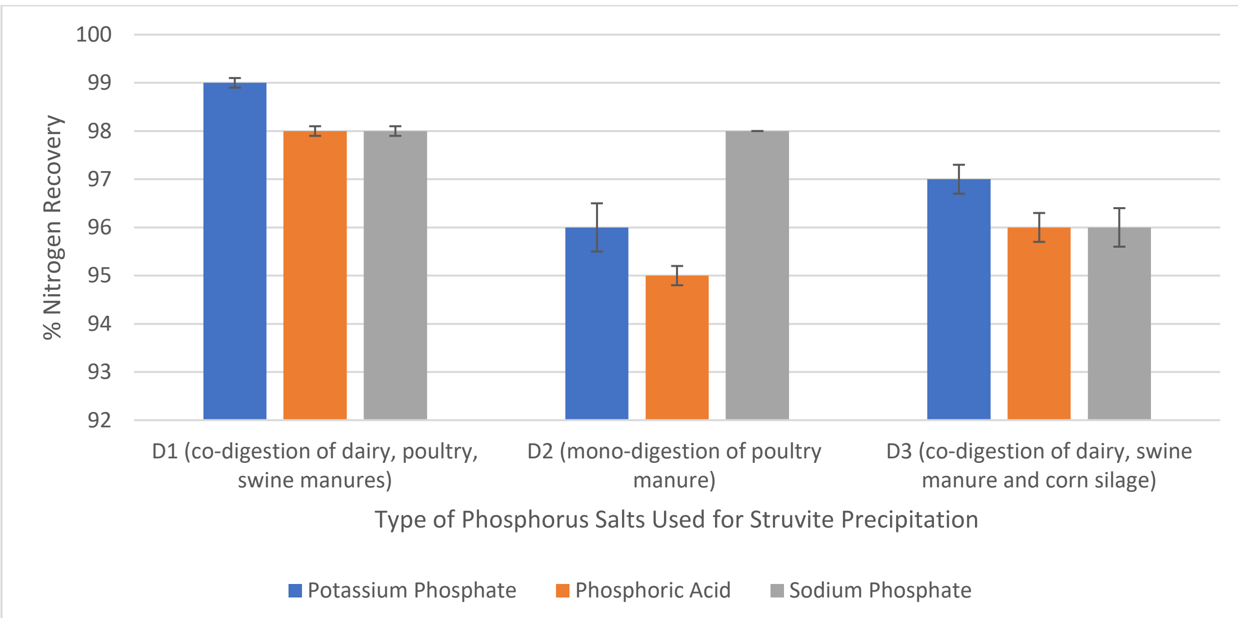

Figure 3.1. % Nitrogen recovery from digestates using various phosphorus salts

The recovery of nitrogen (N) across the salts for a digestate was slightly higher in D1 than D3 and D2 based on the assumption that all N was precipitated as struvite. Overall, it can be noticed that co-digested wastes (D1 and D3) have better % N recovery than D2 (mono-digestate). This can also be attributed to the high TAN value of D2 which could mean that the Mg/P ratio was not sufficient to recover all the N; however, this is not fully the case as there is 98% recovery using sodium phosphate. The average recovery of D2 using phosphoric acid is less than the 97% reported by Yilmazel & Demirer (2011) and occurs due to manure characteristics and operating conditions of the digesters. In potassium phosphate, D1 gave the highest recovery of 99% with the lowest being 96% of D2. However, with potassium phosphate there is a chance of K-struvite formation- whose physio-chemical characteristics are similar to struvite (Graeser et al., 2008), wherein the NH_4^+ ion is replaced by K^+ ion. Even though K^+ can elevate the thermodynamic driving force, overdosing can lead to K- struvite precipitation (Hao et al., 2013) which can be the reason for the marginally lower values for D2 and D3.

The % N recovery can probably be said to depend on the volume of base/acid required to raise it to pH 9. The strength of phosphoric acid required more volume of base to bring the mixture to pH 9, which perhaps led to its lower % N recovery; Nevertheless, there was still recovery of 95% and above in all 3 salts. Following this reasoning, sodium phosphate on the other hand already contains Na^+ ions that can aid in raising pH, thus requiring less volume of acid/base. This could explain the high recoveries seen in D1, D2 and D3, followed by potassium phosphate (as it also contains K^+ ions which are basic in nature). While the reasoning stands, it can be noted that in D3 both phosphoric acid and sodium phosphate gave a recovery of 96% which can be attributed to the process conditions employed. The more wastes are co-digested together, the higher the value of recovery as there is more nutrient availability. The lower recovery values in D3 which comprises of dairy manure can be said to be caused by Ca^{2+} inhibition reported in earlier studies (Koutsoukos et al., 2003; Le Corre et al., 2005; Tao et al., 2016) which must have been neutralized to a certain degree in the case of D1.

Moreover, studies prefer the use of either sodium phosphate or potassium phosphate (Nagarajan et al., 2023) and while phosphoric acid might seem to be convenient due to its lower cost (Yilmazel

& Demirer, 2013) from Figure 3.1 the % N recovery is higher in potassium and sodium phosphate making them more viable to be used than phosphoric acid. To reduce the usage of base to raise pH, it would be suitable to use sodium phosphate and potassium phosphate where the latter also gives the added plausible benefit of K-struvite production, which provides potassium, an element that is also important for crops (Hidayat & Harada, 2021). As Na is not a principal element for crop growth there is less focus on its formation, though there are studies that have studied its growth as seen in Chauhan & Joshi, (2014).

3.4.2 Mass of struvite produced

The weights of the produced struvite did have some variation across the digestates like % N recovery. It can be said that there is a slight positive co-relation between the %N recovery and the mass produced as seen in Figure 3.2.

In the case of D1, like its %N recovery, there is a very high stability of mass produced for all the 3 salts- producing approximately 0.67 g/10 ml (approx.1 g/10 ml). For both D2 and D3, the struvite mass produced was low using phosphoric acid, yielding 0.33 g/10 ml and 0.67 g/ml respectively. However, D2 gave the same value as D1, 0.67 g/mol for the other 2 salts. D3, despite having a lower % N recovery as compared to D1, produced the highest mass in both sodium and potassium phosphate, yielding about 1g /10 ml of digestate. The range of mass observed ranged from 0.33 g/10mL to 1 g/10 mL is within acceptable limits when substantiated with the fact that 0.32 g/10mL to 1.71g/10 mL was achieved despite using different operating conditions (Suzuki et al., 2007).

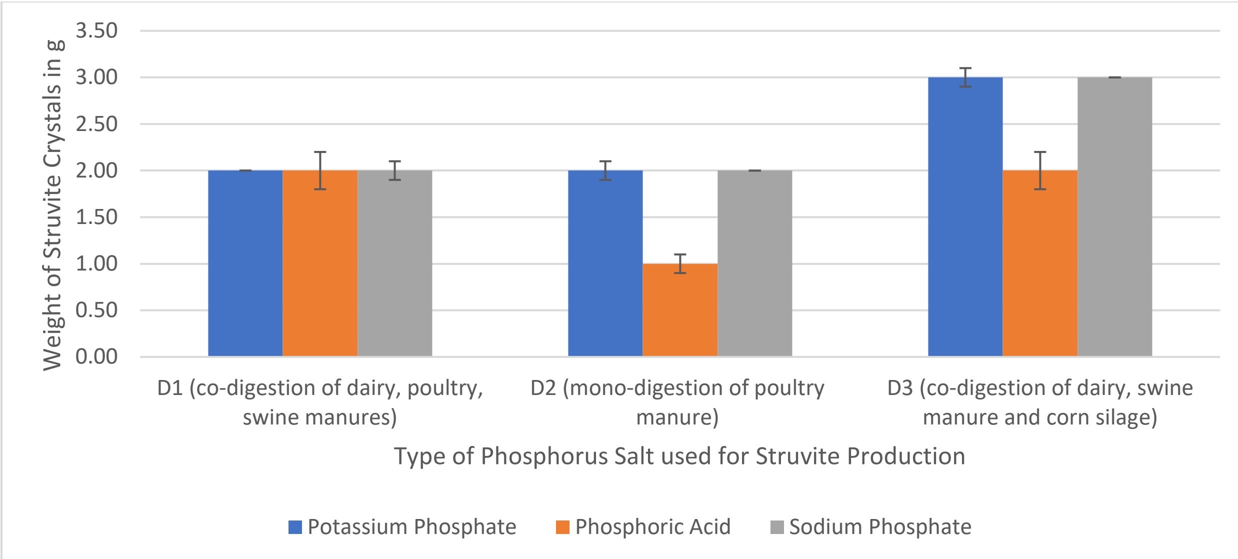

Figure 3.2. Weight of produced struvite crystals from digestates using various phosphorus salts

However, as organic matter (OM) also tends to precipitate along with the struvite crystals (the crystals agglomerates with OM), it is hard to ascertain the amount of either OM, struvite crystals, secondary compounds due to agglomeration of the crystals and as such, the mass produced is taken as the amount of struvite crystals precipitated. The high struvite mass of D3 could be assumed due to its high OM presence compared to the other 2 digestates. Studies report that OM has a negative effect on the struvite formation, but it plays a part in morphology of the crystals (Cerrillo et al., 2015; Capdevielle et al., 2016) and its attachment to the crystals can be verified under SEM (Wang et al., 2022). Moreover, like recovery of N, values are better when using either sodium or potassium phosphate. This further verifies their usage as the P source for struvite precipitation.

3.4.3 VFA reduction

It is also interesting to note that the precipitation process can cause a reduction in VFA's. The decrease depended on the characteristics of the waste and despite the complex chemical reactions (beyond current scope) inducing this reduction, it nevertheless follows the pattern as shown in

Figure 3.3. During AD process, VFA is consumed, but tends to form complexes with Ca and Mg at high values inhibiting P precipitation (Van Rensburg et al., 2003). The continuous operating nature of the digesters attributed to high VFA's of the raw digestate; but the high % N recovery suggests that if there was any inhibition due to other ions, if it was not discernable to affect the precipitation (as extra P was added to facilitate the reaction).

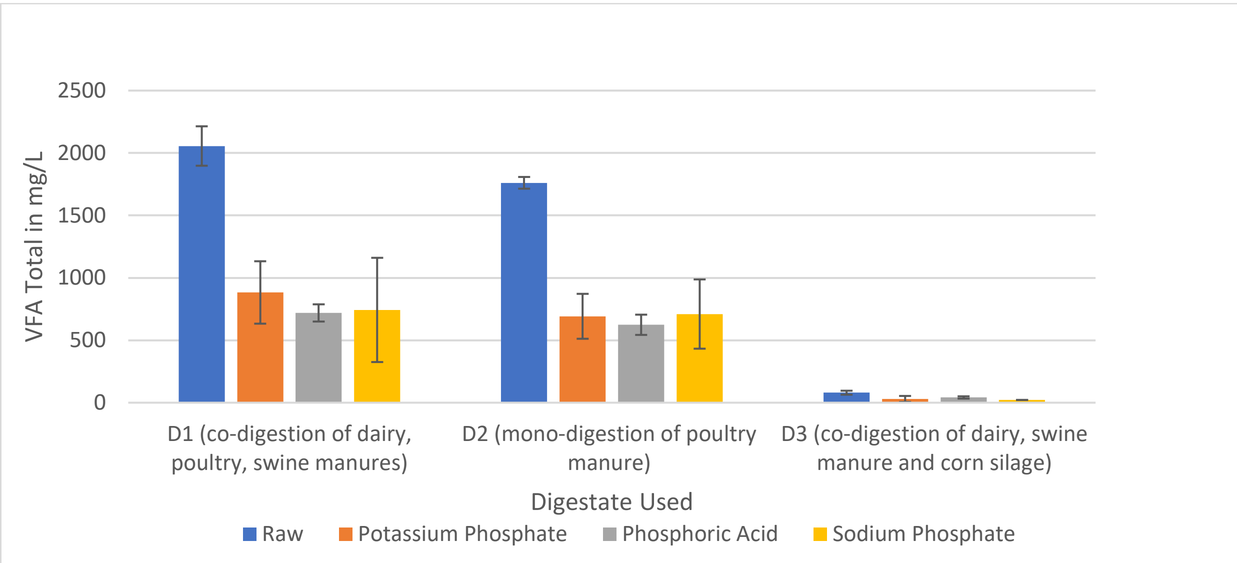

Figure 3.3. Total VFA reduction by precipitating struvite in digestates using various phosphorus salts

However, raw VFA's is approximately 2056 ±418 mg/L for D1 and 1761 ±278 mg/L for D2. As per Figure 3.3, one can infer that the precipitation process can lower the VFA's to <1000 mg/L for D1 and D2 regardless of the phosphorus compound used. Furthermore, the variations in the values could be due to the waste's inherent properties. The current operating conditions of the digester producing D3 is the reason behind its low raw VFA and hence its values show the recurring trend of reduction in VFA even if the values are negligible. It can be gleaned that there is no significant difference in the final VFA's value when using any phosphorus salts. For example, it has been shown that C2 does not affect the kinetics of struvite precipitation in synthetic wastewater, but it may just require more base to raise to the required pH (Zhang et al., 2021); while a similarity is seen in this case as per the % N recovery, it is difficult to deduce the reduction and kinetics of struvite in this instance due to convoluted nature of the reactions.

VFA's are known for their strong odor due to their fermentation (Reyhanitash et al., 2017) and the VFA reduction could prevent it from fermenting, which could be the reason for the lack of odor, even though OM is precipitated in it. There was a cumulative reduction of VFA by about 62% for D1 and D2 and by 61% for D3. These values are not high as compared to %N recovery but nevertheless substantial. In terms of the salts' efficacy, in this case, it can be concluded that phosphoric acid achieves a slightly better VFA reduction than either sodium phosphate or potassium phosphate.

3.5 Conclusion

From this study, mono-digestion of poultry waste and co-digestion of multiple agricultural wastes have great potential to be used in struvite precipitation. The chosen conditions of 1.5 Mg/P and pH 9, 240 rpm yielded a high recovery of nitrogen across the different P salts used. While the difference in %N recovery using the various phosphorus salts was marginal, there was a slight difference in the mass of crystals produced and a reduction of the VFA's. The highest N recovery of 99% was achieved with potassium phosphate for D1 while D3 produced the highest mass of 1 g/10 ml of digestate. The recommended P salts are sodium phosphate and potassium phosphate; wherein the latter case can give an added benefit of producing K-struvite. While the results of struvite precipitation highly depend on the waste characteristics and the conditions used, it can be said that it is one of the most sustainable nutrient recoveries methods available. However, a need to upscale the process for manures leads to further scope in assessing the produced struvite safety and practically by studying its pathogenic level, heavy metal quantities and life cycle analysis.

CHAPTER III-IV CONNECTING STATEMENT

Chapter III validated the ability of the process to further valorize the nutrients and matter present in anaerobic digestates. It also depicted its characteristics for N recovery and produced a substantial mass of crystals. The basis of chapter III sets the tone for the experimentation done in chapter IV to ensure the process replicability and to show the benefits of integrating AD alongside struvite precipitation.

CHAPTER IV

Integration of Anaerobic Digestion with Struvite Precipitation to Circularity in Manure Management.

4.1 Abstract

With the focus shifting towards circular principles, the same can be applied to manure management. One of the major ways to treat manure is through anaerobic digestion (AD). This method produces energy in the form of biogas and the resultant digestate is commonly applied as an organic fertilizer to farmlands. On the other hand, the misuse of the digestate application can lead to problems like greenhouse gas (GHG) emissions, soil contamination, eutrophication, which is detrimental to the environment. In fact, the digestate is still nutrient rich, further valorization can be done to recover nutrient in dry and easily transportable forms and also to maximize energy. In this regard, struvite is heralded as a potential green fertilizer as it is a magnesium ammonium phosphate (MAP) compound (containing 2 prime nutrients required for plant growth). Struvite provides significant control over land application, unlike the application of raw manure or digestate in terms of quality and quantity. This chemical precipitation process can be integrated with AD to increase benefits as well as give useful by-products with practical applications. As such, this study focuses on a representative cycle of high solids anaerobic digester (HSAD) of 2 mixtures of co-digested wastes, whose cycle length was 77 days, highest specific methane yield (SMY) being 1.26 L/g $COD_{s\ fed}$ for D1(Poultry Manure, Dairy Manure and Corn Silage) and 1.49 L/g $COD_{s\ fed}$ for D2 (Chicken Manure, Dairy Manure, Swine Manure and Corn Silage). The maximum cumulative biogas produced was 374 L and 369 L for D1, D2 respectively. The digesters were found to give favorable results and could adapt to the increase in feedstock. The struvite produced from D3 (Mixture of all D1 and D2 digestates at the end of the cycle) resulted in very

high recovery of N 98-99% with mass of 0.67 g/10 ml. It is recommended to use either potassium or sodium phosphate for this process. Overall, this integration yields very high advantages with practical application and studies can be furthered to scale-up the process to make it more viable.

Keywords: Anaerobic digestion, high solids anaerobic digester, integration, nutrient recovery, struvite precipitation.

4.2 Introduction

Part of sustainable development is to improve the viability of closed nutrient recovery systems in the agricultural sector -due to population growth leading to more wastage, fertilizers requirements due to rising food demands (Pigoli et al., 2021). This in turn is placing a burden on biogeochemical cycles, especially of N and P and by appropriate distribution (through assessment and requirement) of said nutrients can reduce the pressure on these cycles (Rockström et al., 2009; Steffen et al., 2015).

Anaerobic digestion efficiently converts organic wastes into valuable resources (Yu and Huang, 2009); reducing wastes going to the landfills, water pollution and GHG emissions while furthering economy (Dennehy et al., 2016). From manure, agricultural waste streams, AD is commonly employed to treat the wastes producing biogas, a renewable energy; and by integrating AD and nutrient recovery, both energy and nutrients can be recovered (Lorick, 2020). The digestate produced during the process has good fertilizer properties as it is made up of water, biomass, inert and undigested solids (Di Costanzo et al., 2021). Furthermore, the utilization of high solids anaerobic digester (HSAD) generates nutrient rich digestate that can be used for agricultural applications (Pigoli et al., 2021). The digestate will have amendable properties due to its relatively high biological stability -degree of OM degradability (Wojnowska-Baryła et al., 2018), neutralized phytotoxicity, and nutrient availability (Tambone et al., 2010).

However, digestate must have adequate storage and processing to mitigate release of air pollutants that results in nutrient loss (Lamolinara et al., 2022). Furthermore, disposal of digestate is problematic as it might contain compounds harmful to humans, organisms, and the environment (Jomova & Valko, 2011; Silkina et al., 2017). To prevent this, further valorization of the digestate from agricultural waste streams is required and struvite precipitation was one of the most viable technologies (Macura et al., 2019). Also, total ammonical nitrogen (TAN) and ortho-phosphate are more readily available due to biological degradation of organic matter during the AD process (Scaglia et al., 2018) making it a readily usable source for struvite production.

To develop a circular sustainable economy, AD of organic wastes is a major component as it addresses issues like waste, food production, energy, and nutrient cycling (Antoniou et al., 2019). Considering the high efficiency of chemical precipitation, it would be economical to integrate struvite precipitation with agricultural waste streams high in N and P (Muhmood et al., 2019). The combination of both mass and energy systems (struvite and biogas) in line with circular economic principles can diminish the use of fresh resources (raw N and P resources used to make fertilizers) and can even drastically reduce waste discharge (e.g., untreated digestate and raw manure); making the integrated system both economically and environmentally stable (Hidalgo et al., 2019).

As such, a preliminary study was done on the potential of integration by assessing the efficiency of a representative cycle of HSAD for multi substrate and then using the digestates at the end of the cycle for struvite precipitation. Moreover, the feasibility of using various phosphate salts and the mass produced were studied. Doing so, this paper aims to emphasize that maximum nutrient recovery is possible with struvite precipitation from co-digested wastes, highlighting the benefits of integrating the systems.

4.3 Methods and Experimental Set-Up

4.3.1 Raw feedstocks and digestates sampling

The raw feedstock (swine manure, poultry manure, dairy manure, and corn silage) used for the representative cycle of HSAD was collected in and around AAFC, Sherbrooke Research and Development Centre. The anaerobic digestates were collected from the HSAD digesters designed for co-digestion process at the AAFC Centre. To monitor the performance, samples were taken weekly from the liquid digester and thrice per week for biogas quality of both the solid and liquid digesters. They were all stored at 4°C for further physiochemical characterization and experimentation as shown in Table 4.1 and Table 4.2. The compositions of the raw manure used for anaerobic digestion for D1 are (co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage). For struvite characterization, all the liquid digestate of the digesters at the end of the cycle were mixed, which is D3 (Mixture of all D1 and D2 digestates at the end of the cycle).

Table 4-1. Characteristics of the raw feedstock used for the HSAD

Sample Type	%TS	%VS	TAN (mg/l)	TKN (mg/l)	TVFA (mg/L)	COD_s (mg/L)	Alkalinity ($mgCaCO_3/L$)	pH
Solid Inoculum	19.98	16.94	2759.00	6174.00	1033.00	12510.00	15131.00	9.01
Poultry manure	59.38	48.73	7105.00	20294.00	1263.00	81655.00	36406.00	8.99
Dairy Manure	16.53	15.26	433.00	4138.00	3287.00	20112.00	6696.00	7.30
Swine Manure	7.84	6.20	1680.00	3402.00	3766.00	10098.00	8812.00	7.10
Corn Silage	31.88	28.84	520.00	3468.00	5341.00	57156.00	0.00	3.86

Table 4-2. Characteristics of the wastes used for AD and of the digestate used for struvite precipitation

Characteristics	**D1 (Poultry Manure, Dairy Manure and Corn Silage)**		**D2 (Chicken Manure, Dairy Manure, Swine Manure and Corn Silage)**		**D3 (Mixture of D1 and D2 at end of cycle)**
	Initial	Final	Initial	Final	
pH	8.59	8.40	8.49	8.43	NA
Alkalinity (mg $CaCO_3$/L)	16501.00	20091.29	15515.15	19354.93	14605.00
TAN (mg/L)	2968.18	3641.37	2825.82	3509.83	2716.00
TKN (mg/L)	7733.99	6845.89	6876.45	6683.72	3234.00
COD Total (mg/L)	NA	NA	NA	NA	NA
COD Soluble (mg/L)	23132.99	12695.80	20036.44	9267.88	5494.00
% Total Solids (TS)	23.62	19.44	21.82	19.60	2.02
% Volatile Solids (VS)	20.23	16.23	18.79	16.39	0.91
% Free Solids (FS)	3.39	3.21	3.03	3.21	1.11
VFA (mg/L)	1274.07	2550.18	1543.00	957.00	325.50
Acetic, C2	677.16	1761.33	831.00	738.00	251.50
Propionic, C3	59.76	516.61	113.00	157.00	31.00
Isobutyric	0.00	50.90	53.00	0.00	1.90
Butyric, C4	425.54	0.00	331.00	0.00	9.40
Isovaleric	62.32	152.56	87.00	63.00	16.90
Valeric, C5	0.00	0.00	0.00	0.00	1.20
Caproic, C6	49.29	68.78	128.00	0.00	13.50

4.3.2. Experimental set-up

2 different kinds of feedstock mixtures D1 and D2 were digested in duplicate digesters for a representative cycle: percolation-recirculation digesters based on the design by Mahato et al. (2022) and adapted for use with mixed substrate as seen in Bele (2022). The below flowchart depicts the operation of the percolation-recirculation digester, also known as a solid-liquid digester.

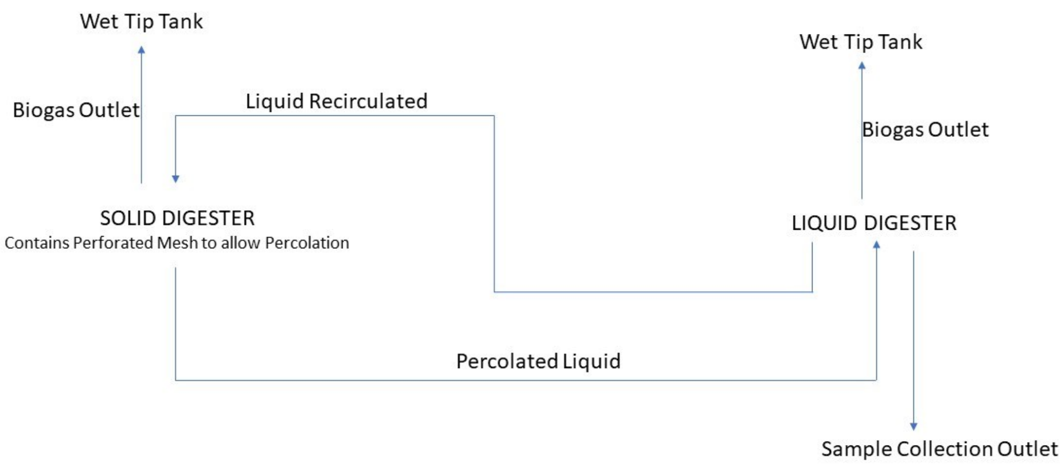

Figure 4.1. The flowchart depicts the working of the percolation-recirculation digester.

For struvite, 2 sets of trials were done using D3 with each sample done in duplicate. All the experiments were done at room temperature and 30 mL of anaerobic digestate was used. To supplement the reaction, magnesium and phosphorus salts were added in a ratio 3:2 (Mg:P) i.e., 1.5 Mg/P where 1M $MgCl_2$ (constant throughout the trials) and 1M for KH_2PO_4, Na_2HPO_4 and H_3PO_4 were used. Furthermore, all experiments were stabilized at pH 9.0 and 1M of NaOH and 1M HCl were used to adjust the pH. Mixing was done using a magnetic stirrer at 240 rpm and pH was measured using a pH meter. Mixing was done for a further 3 minutes after pH stabilization to ensure complete mixing. The samples were left overnight for the precipitate to settle completely. It was then vacuum filtered, and the resulting residue was kept in the oven at 105°C for 4 hours, then taken out. The filtrate and the powder were then analyzed appropriately.

4.3.3. Methods for sample characterization

APHA standard protocols (Eaton et al., 2005) were used to analyze the Total and Volatile Solids. pH and alkalinity were measured using Mettler Toledo AG 8603 pH, SevenMulti (Schwerzenbach, Switzerland) and Titralab AT1000 Series (Hach Lagne Sarl,Hach, Switzerland). VFA was prepared according to the protocol given by Masse et al. (2003) then analyzed using Perkin Elmer gas chromatograph (Model Clarus 580, Perkin Elmer, Shelton, CT, USA). While the biogas quality was quantified using 490 Micro GC Biogas Analyzer (Agilent Technologies, CA, USA). Closed reflux colorimetric method was used to determine COD_s (Eaton et al., 2005). TAN and TKN were determined by macro-Kjeldahl methods (Eaton et al., 2005) using the 2460 Kjeltec Auto-Sampler System (FOSS, Sweden). Biogas production in terms of volume was recorded by calibrated wet tip-tanks. Specific methane yield was calculated using the volume of methane produced per gram of $COD_{s\ fed}$.

4.4 Results and Discussion

4.4.1. Digester performance and relationship between SMY, biogas production and acid ratios (C3/C2, C4+C5/C2 and TVFA/TA)

For this representative cycle, the feedstock to inoculum ratio used was 1:7. Overall, it took 77 days to complete this cycle for both cases. The cycle length was determined based on the daily biogas production and VFA levels. The deteriorating VFA levels by the end of 77 days indicated that cycle had reached its end. The calculated OLR yielded 3.14 gVS/L.d for D1 and 3.22 gVS/L.d for D2. It should also be noted that from day 0-43, 20% of the inoculum was pumped into the HSAD and was increased by 10% from days 48-77 for both D1 and D2. The digester efficiency in terms of $COD_{s\ fed}$ is 45% for D1 and 54% for D2. From this, it can be inferred that the swine manure added in D2 had enriched the digester with nutrients that were utilized by the methanogens.

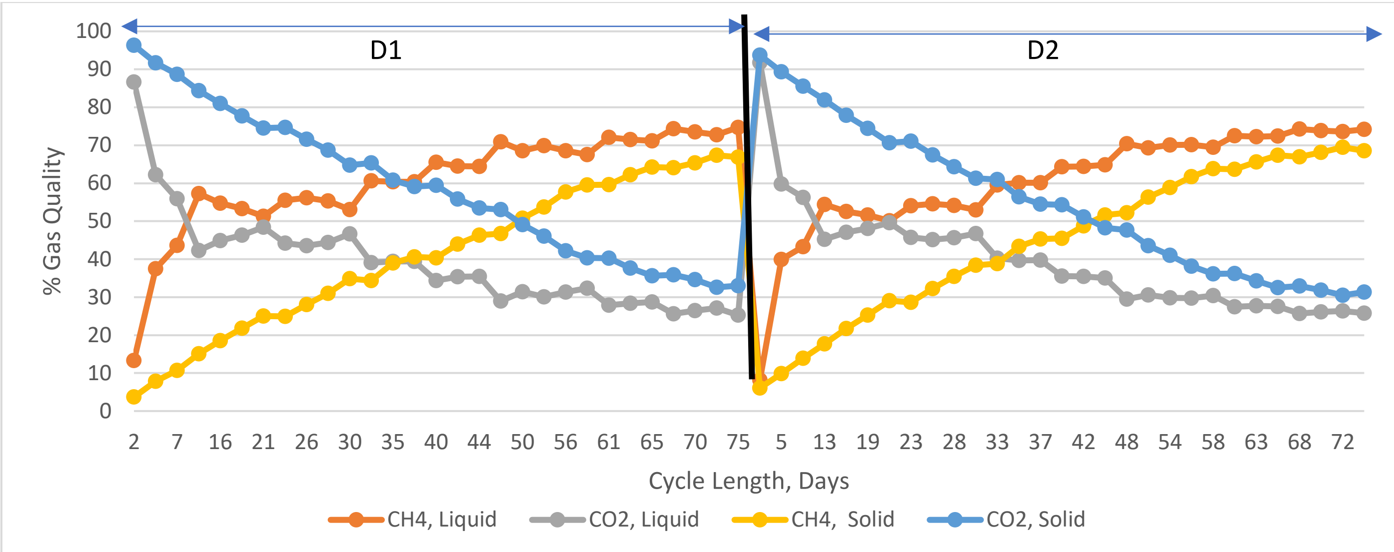

Figure 4.2. Relationship between %CH_4 and %CO_2 for both the solid and liquid digesters for the cycles of D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage)

The quality of methane is essential to know if the digester is functioning as intended. For both the solid and liquid digesters, initially there was a high level of CO_2 and low level of CH_4 whose values reversed by the end of the cycle (Figure 4.2). The remaining percentage can be taken as the % of H_2S as was detected by the Micro GC, the amount produced dependent on the feedstock (Wellinger et al., 2013). There is a strong negative correlation between %CO_2 and %CH_4 for both D1 and D2. The highest % CH_4 was 74.69% in liquid and 66.90% for D1 and 74.18% in liquid and 68.59% in solid for D2; showing that methane quality is better in the liquid digesters than the solid even if there was more fluctuation in the former. Even then, the methane quality is similar in D1 and D2. The common range for CH_4 is 50-75% and CO_2 are 25-45% with minor traces of H_2S (Wukovits & Schnitzhofer, 2009). Furthermore, our results indicated above, around 74% of CH_4 was produced in both D1 and D2 indicating stellar quality. The high quality produced can be attributed to the consumption of nutrients, VFA's due to degradation of organic matter (OM) over the period, and CO_2 being utilized to form methane.

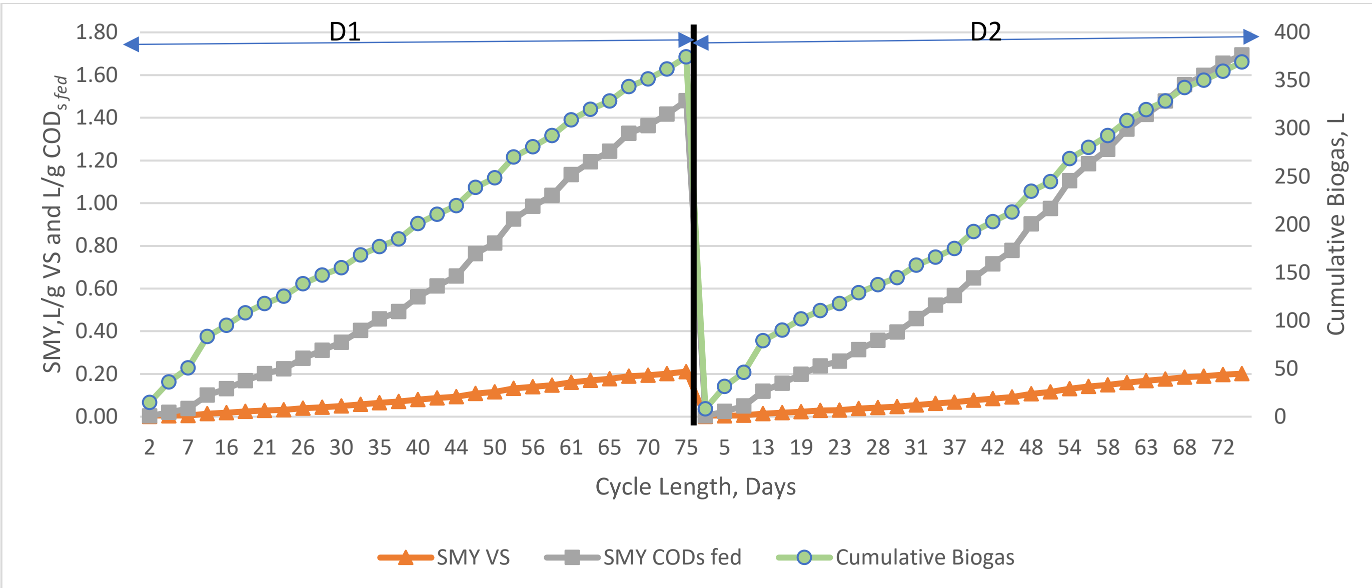

Figure 4.3. Relationship between cumulative biogas with SMY (COD$_{s\text{ fed}}$)/SMY(VS) for the whole cycle of D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage)

Since there is a strong positive correlation in both digesters for specific methane yield SMY (VS) and SMY (COD$_{s\text{ fed}}$), R=1; using either SMY (VS) or SMY (COD$_{s\text{ fed}}$) would have resulted in the same trend that is shown in Figure 4.2, so the following results are explained using SMY (COD$_{s\text{ fed}}$). In Figure 4.3, D1, cumulative value has steadily increased while SMY (COD$_{s\text{ fed}}$) has a gradual increase, giving a parallel curve. In D2 shows the same starting pattern as D1, with SMY (COD$_{s\text{ fed}}$) overlapping the cumulative biogas towards the end of the cycle. The reason for this could be the 30% inoculum recirculation from day 48 onwards which influenced D2 while having no effect on D1. The increment in both SMY (COD$_{s\text{ fed}}$) and cumulative biogas over the duration of the cycles of D1 and D2, shows that methanogenic activity was taking place at a good rate. This indicates that the adapted inoculum was able to handle the increased substrate load and produce a satisfactory amount of biogas over this period.

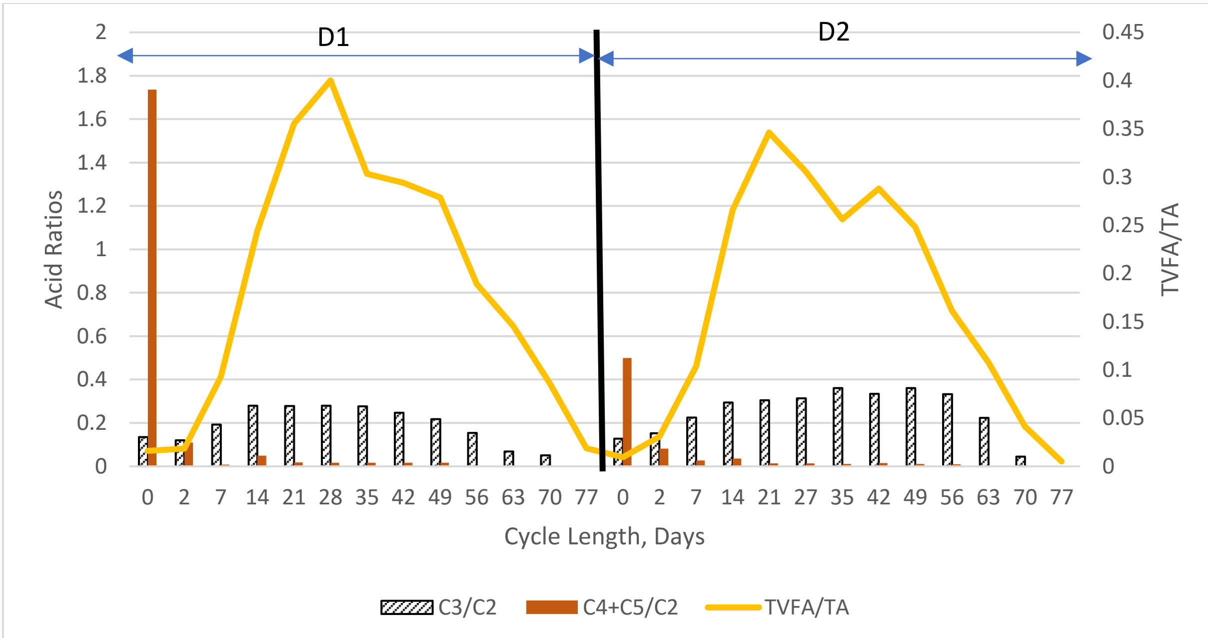

Figure 4.4. Acid ratios and TVFA/TA for D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage)

Volatile fatty acids (VFAs) are short-chain aliphatic mono-carboxylate compounds that are made up of 2 to 6 carbon atoms (Merrylin et al., 2020). Figure 4.4 highlights the 3 main VFA ratios used to assess a digester's stability. C3/C2 was below 0.4 for both D1 and D2, which is well below the recommended <1.4 (Kwietniewska & Tys, 2014). Whereas C4+C5/C2 values for both were within the range suggested of <0.3 (Mahato et al., 2020), but on day 0, it seemed that the start-up was unstable, leading to such high values. This could be an outlier and did not seem to have an impact on the stability afterwards. A value below 0.5 is advised for TVFA/TA (Rajinikanth et al., 2008) but might cause some instability around 0.4- 0.8(Callaghan et al., 2002); D2 having good stability, and D1 falling between the margins. Nevertheless, the rise in SMY ($COD_{s\ fed}$) and cumulative biogas plus the VFA consumption and CODs decrease verify that the HSAD system was, in fact functioning without any problem, ensuring its stability.

4.4.2 Relationship between %TS, % VS, pH, alkalinity, TAN, TKN, CODs, VFA

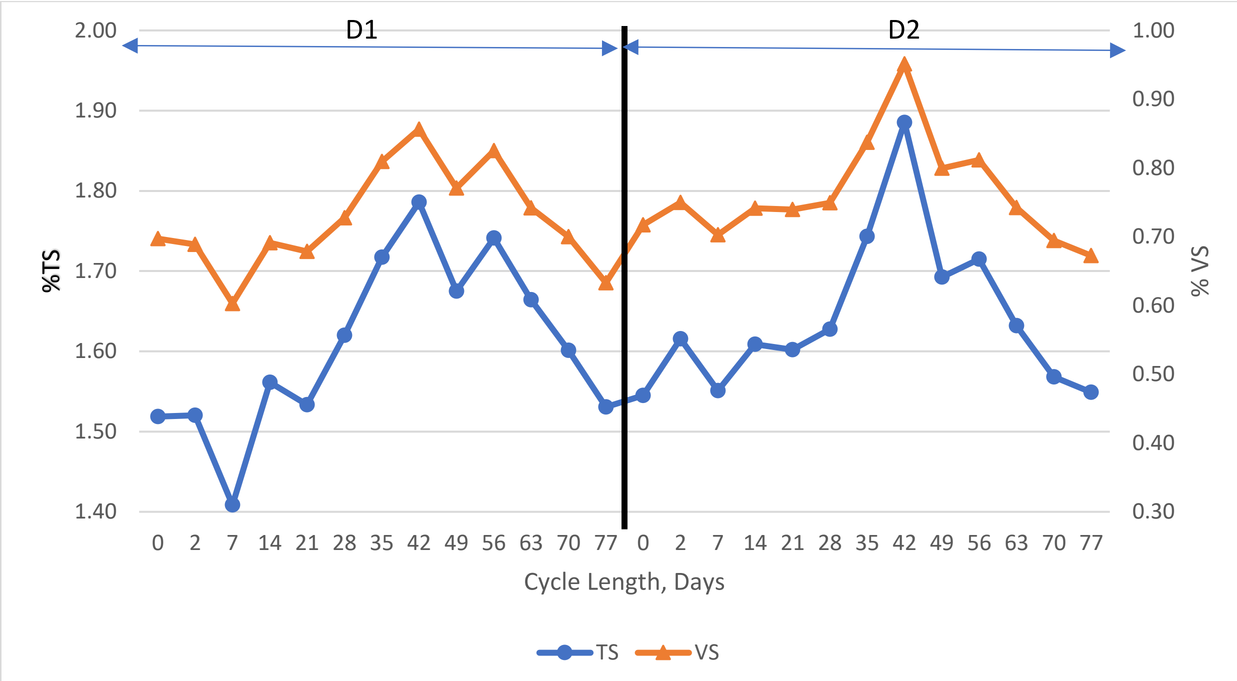

Figure 4.5. Relationship between %TS and %VS for the whole cycle of D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage)

There is a similar trend of change in %TS and %VS for both D1 and D2, as seen in Figure 4.5. The notable difference being that on day 49, there is a slight decrease of both in D1, with a steep decrease of both in D2-the reason being in the increased recirculation of the inoculum. Overall, it seems there is a gentle increase and then a notable decrease as the cycle reaches its end. The values decreasing shows that the OM is being degraded to release nutrients for methanogenic activity. Since, % TS value was the same at the beginning and end of the cycles (except for the minute decrease in D1), the % VS decreased at the end increasing the resultant amount which is % free solids (FS). This means that there is some undegraded OM left in the digestate that could have played a role in the struvite crystal mass, which is discussed in *section 4.4.3*.

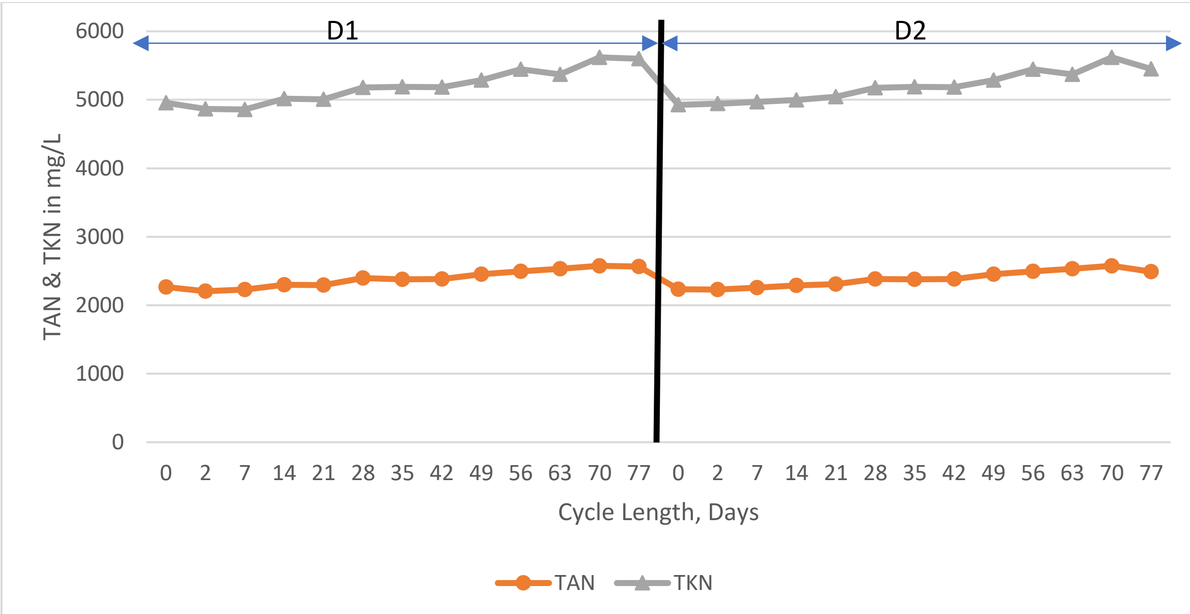

Figure 4.6. Relationship between TAN and TKN for the whole cycle of D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage*)*

As seen in Figure 4.6, it is interesting to note there was a gradual increase of both TAN and TKN for both cases- as the TAN content is directly correlated to initial TKN (as its value comprises of all the nitrogen present) of the feedstock (Monlau et al., 2015) which is why the TAN for both D1 and D2 are also high. Quite interestingly, the addition of swine manure seems to unlikely have an effect, seeing that the ranges produced for both are marginal to each other. Furthermore TAN/TKN ratio was a high value of 0.85 for both, due to the presence of high degradable feedstock such swine, poultry overshadowing the less degradable feedstock such as corn silage (Möller & Müller., 2012). Also, the increase in TAN, means nutrient availability in the digester was good for the period of the whole cycle. Unlike %TS, %VS, there was no dip or rise on day 49, showing that the increased 10% inoculum recirculation rate had negligible effect on the N present in the digesters;

but TKN had a small rise between days 63-70 and sloped down in both cases. TAN and TKN at the end of the cycle for D1 and D2 were on average 2564 mg/L, 3033mg/L and 2490 mg/L, 2961 mg/L respectively. This is the key to note, as that means that there is still nitrogen that can further valorized at the end of the cycle to reduce the TAN present in the digestate (which is discussed later in *section 4.4.3*).

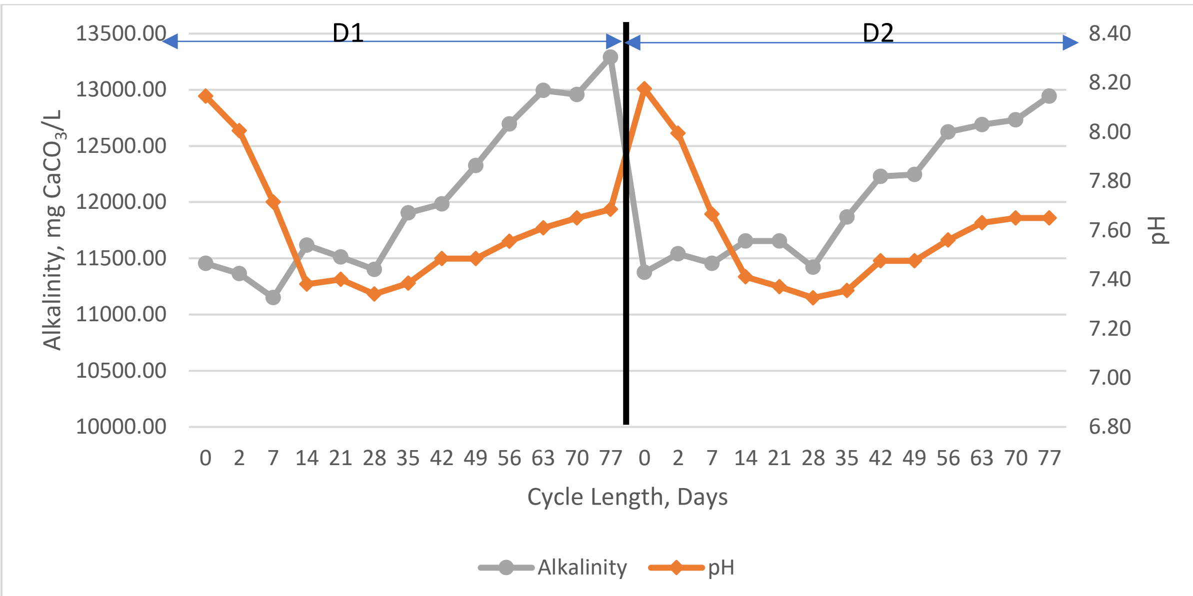

Figure 4.7. Relationship between pH and alkalinity for the whole cycle of D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage)

From Figure 4.7, it is seen that pH for the first 14 days decreased rapidly to around pH 7.40 for both, while alkalinity was gradually increasing, despite the fluctuations resulting in a dip on day 7 and day 28. After day 14, however, there was steady increase in pH until the end of the cycle to sustain the production of methane. The final pH was higher by a slight margin in D1 (pH 7.69) than in D2 (pH 7.65) and can be attributed to the characteristics of high pH of poultry manure used. Meanwhile, there was an unsteady increase in the alkalinity in the digesters even after 14 days due to acid formation and depletion. In a similar fashion, D1 had a higher alkalinity than D2.

The high alkalinity is an indication that VFA has been consumed and it acts as a buffer to prevent extreme pH fluctuation of the digester. The pH was found within the acceptable ranges (7.0-7.6) for promoting biogas production (Cecchi et al., 2005). It should be stated that struvite formation can occur from pH 7-11 (Buchanan et al., 1994), no such growth was seen as other requirements for its formation were not met.

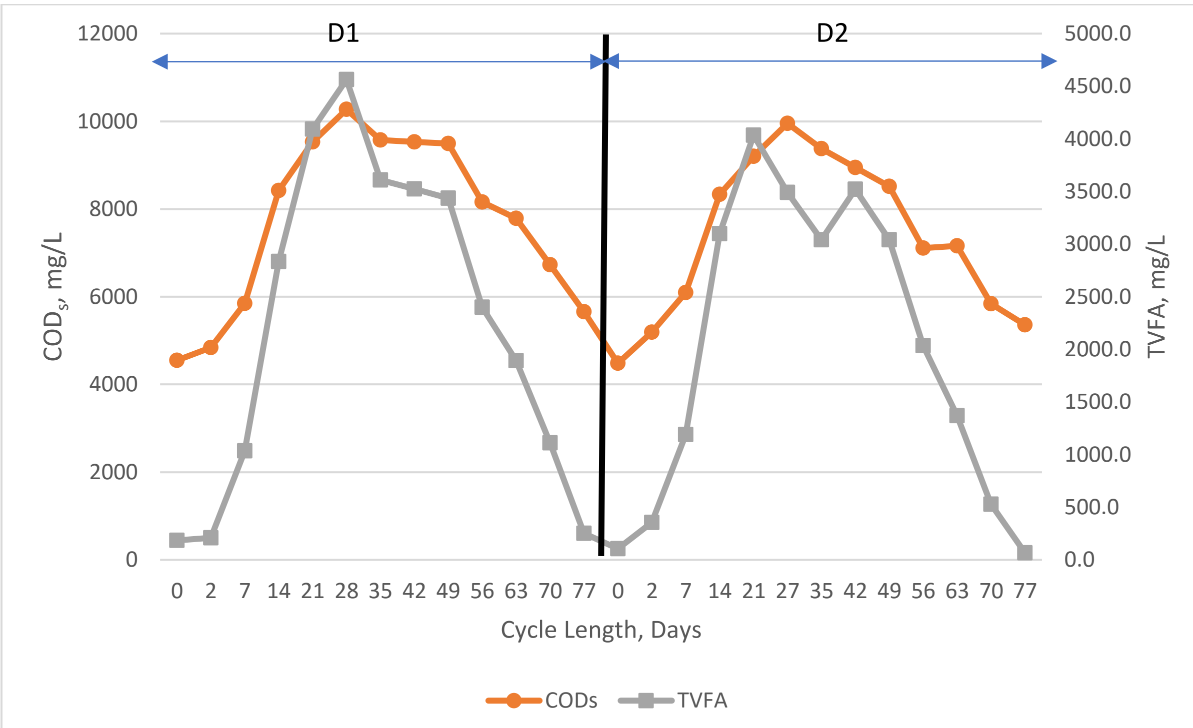

Figure 4.8. Relationship between CODs and TVFA for the whole cycle of D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage)

Both COD_s and TVFA followed a fluctuating pattern as depicted in Figure 4.8 with strong positive correlation of R=0.97. Initially, there was an accumulation of affecting both biogas production and quality, with its final decrease showing its consumption during the cycle. In the case of D1, there was a rapid increase in both TVFA and COD_s until day 28, then a drop followed by a gradual decrease, ending with a steep decline. This could be taken as the day until which acidogenesis and acetogenesis was occurring, resulting in high values of COD_s and TVFA. Then the decrease is occurring due to nutrient usage during methanogenesis. Meanwhile for D2, the same scenario can be ascribed. Acidogenesis and acetogenesis peak was reached at day 21, decreased due to methanogenesis and then had another peak at day 42 before declining steeply till day 77. For both COD_s and TVFA the values were higher than that of D2; COD_s being degraded shows the breakdown of OM in the manures which leads to more nutrient availability and produce VFA's for consumption to produce methane.

4.4.3. Struvite precipitation, nitrogen Recovery and crystal mass produced

The struvite analysis was done to D3, rich in TAN, yielded ≥ 98% N recovery regardless of the phosphorus salt added. The % N recovery was highest with potassium phosphate with 99% with 98% for both phosphoric acid and sodium phosphate. In this case, it can be concluded that different phosphorus salts do not greatly affect the nitrogen recovery. The high TAN values at the end of the cycles seen in *section 4.3.2*, is almost completely captured in the form of struvite as indicated by the below results. The results shown in Figure 4.9 indicate that the protocol devised in Chapter III is suitable for co-digested wastes, yielding higher values than the digestates used for that experiment; it is noteworthy that the composition of the current digestate could have also played a part to yield such high values.

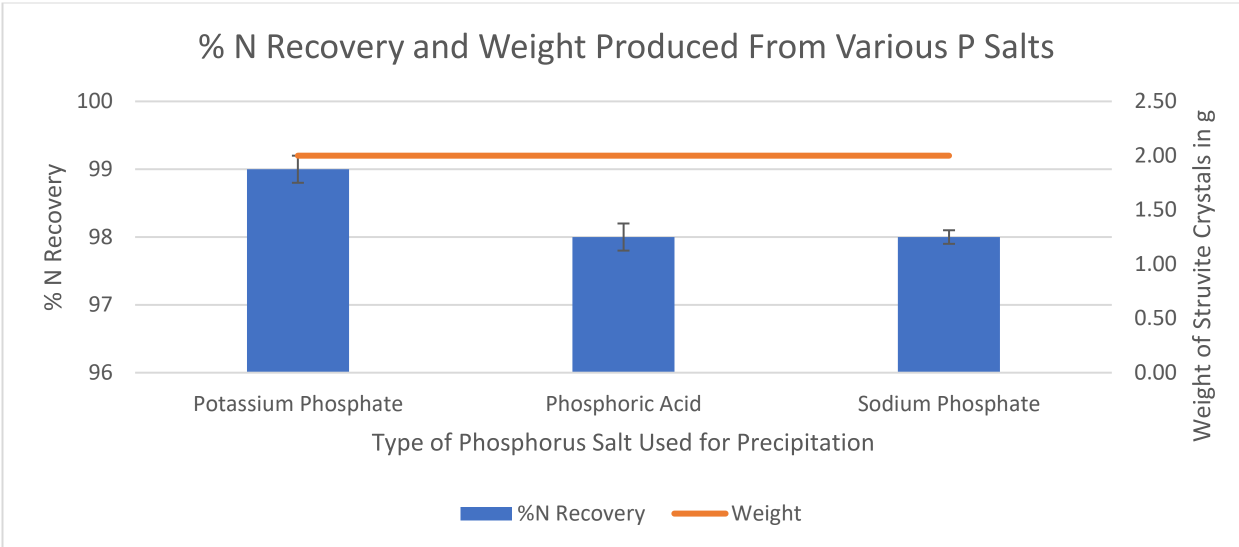

Figure 4.9. % N recovery and weight produced from various phosphorus salts

Interestingly, there was no variation at all with the weights. This means that regardless of the phosphorus salt used, they all produced 0.67 g/10 mL ±0.1 g/10 mL. With this, one can assume that the amount of OM present plays a role in the mass, leading to this similarity. However, in the case of potassium phosphate, there is no proper way to ascertain the amount of K-struvite produced. Overall, all 3 salts are suitable for this process on the notion that the mass produced consists of entirely struvite crystals. Keeping in mind the costs and feasibility in usage, potassium phosphate or sodium phosphate are recommended.

4.5 Conclusion

Anaerobic digestion integrated struvite precipitation is seen as a viable process for sustainable manure management. The HSAD proved to be stable for D1(co-digestion of dairy, poultry, swine manures) and D2 (co-digestion of dairy, poultry, swine manures and corn silage). using multiple substrates as seen by their acid ratios (C3/C2 and C4+C5/C2) and TVFA/TA. The cycle lasted for 77 days, and methane quality produced was good around 74% for both D1 and D2. The SMY ranges were as follows: 0.002- 1.26 L/g $COD_{s\ fed}$ for D1, and 0.002- 1.49 L/g $COD_{s\ fed}$ for D2. The cumulative biogas produced ranged from 15-374 L and 8- 369 L for D1 and D2, respectively. There was also a very high % N recovery of 99% using potassium phosphate and around 0.67 g/10ml produced. Studies could be further done on continuing adapting the AD for co-digestion and scaling up the integration with struvite precipitation.

CHAPTER IV-V CONNECTING STATEMENT

As one has seen from chapter IV, it is a sustainable idea to integrate anerobic digestion with struvite production. It was also seen that amount of recovery also depends on the characteristics of the waste. Nevertheless, this process ensures proper recovery of nutrients that has practical applications. Moving on to chapter V, where we summarize the research, its utilization potential, and impacts.

CHAPTER V

SUMMARY AND CONCLUSION

This thesis has shown that struvite is a potential substitute for commercial fertilizers that promotes sustainability as a whole and in the agricultural sector. Struvite has the inherent characteristics that enable its production to be environment friendly and alleviate the harmful effects of livestock wastes.

The sheer number of livestock plus the wastes produced make it a highly viable waste source to be used for struvite production. Given the nutrients present in these wastes and given the need to find more sustainable fertilizers to promote bioeconomic practices; struvite is the optimal option for incorporating all these ideas.

These ideas were then scrutinized, especially by exploring struvite production from livestock wastes, anaerobic digestates and its clear benefits, which gave us encouragement to research further into its production using livestock waste streams.

The heart of the thesis is the experimental methodology that produced reliable results. It shows that irrespective of the digestate type being used (either from mono-digestion or co-digestion), high levels of N recovery can be obtained. There were slight differences in the % N recovered, however, the weight of crystals obtained did show variation with usage of different salts on the various digestates. VFA can also be drastically decreased. It was noted that the representative HSAD cycle of multi- substrate digestion was stable and yielded good methane volume and quality. Integrating biogas with struvite can deliver both high biogas quality while at the same time decreasing the N content present in the digestate, making the filtrate (wastewater) safe to use for other purposes after treatment.

Apart from practically scaling up this integrated process, there is further scope that can be undertaken to ensure the safety and efficacy of struvite as a fertilizer: pathogenic study, heavy metal study, plant trials, GHG emissions, and K- struvite. Other interesting studies that could be done in probable future, is % P recovery, production without chemical addition, life cycle analysis based on functioning plants and regulations on its usage. Currently studies are being done on using agricultural wastes as a source material; soon it can yield positive results and needed information to explore these topics further. Struvite process of livestock wastes is the viable option at the present.

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