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## Research Article

# STRUVITE FORMING POSSIBILITY BASED ON THE COMPONENT CONCENTRATION IN LIQUID PHASE OF ANAEROBICALLY DIGESTED SLUDGE AT VARYING TEMPERATURE AND pH AND PHOSPHORUS RECOVERY USING ACETATE AND TRIS BUFFER SOLUTION A CASE STUDY AT AALBORG WEST WASTEWATER TREATMENT PLANT

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### ABSTRACT

In one hand struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) formation is a plague while on other hand is resource recovery technique in sustainable wastewater treatment system, more particularly in sludge digestion process. However, the process and place of formation for struvite plays a vital role whether a problem or method of obtaining nutrients from highly concentrated sludge. Present research is related to release of chemical components into liquid phase from anaerobically digested sludge at various level of temperature. Further, it is investigated for struvite formation possibility from the obtained concentration in the liquid phase. The project aim is restricted to; (i) Analysis for spontaneous struvite formation possibility into the digester and post-digestion phase; and (ii) Nutrients recovery particularly the phosphorus as non-spontaneous struvite formation. Experimentally, the sample sludge was processed under varying temperature from 25oC to 60oC in a laboratory. Hence, the liquid fraction containing dissolved chemical constituents was separated by centrifugation and filtration technique. The residual solid fraction further used for extraction of struvite components using acetate and tris buffer solutions. The final outcome for spontaneous struvite formation from total produced sludge concludes insignificant possibility for struvite problem in the digester and post-digestion phase depending upon the supernatant constituent concentration. This is because of low magnesium concentration. But on adding magnesium source, maximum of 11.70% phosphorus recovery can be done as non-spontaneous struvite from the total daily anaerobic digested sludge.

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### INTRODUCTION

In wastewater treatment system, especially in sludge digestion process, struvite scaling is a plague (1,2). It is an orthorhombic hexahydrate crystal, having chemical formula  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$  and molecular weight of 245.41gm and formed by the combination of ionic magnesium ( $\text{Mg}^{2+}$ ), ammonium ( $\text{NH}_4^+$ ) and phosphate ( $\text{PO}_4^{3-}$ ) that exceeds the solubility product in aqueous solution. Favorable condition and sufficient ionic component concentrations can causes rapid growth of the spontaneous struvite granules that are insoluble or less soluble (3,4) and can clog the pipes and mechanical equipment within months, if left unchecked (5,6,2). Nucleation of struvite in these structures is not noticed until and unless reached to a state of replacement of the clogged unit (7).

On the other hand, struvite itself is a valuable fertilizer that supplies nutrients like nitrogen and phosphorus to the plants/crops for longer time that having lower dissolution rate. At present, the recovery of phosphorus and nitrogen in the form of struvite through chemical precipitation technique is getting global attention (8). Such nutrients are also considered as non-renewable and limited but their demand is increasing with growing world population and food production. Non-spontaneous formation of struvite fertilizer from anaerobic supernatant, urine and/or animal waste has been theoretically and experimentally investigated by researcher and scientist including chemist, biochemist and civil engineers (9,10,11,12,13,14,15). In particular, phosphorus recovery has been recommended from anaerobically digested sludge liquors (16,17) because it releases more feasibly by microorganism during anaerobic condition that has been taken up during

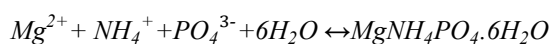
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aerobic condition in secondary treatment process in Enhanced Biological Phosphorus Removal (EBPR)(18).

Furthermore, with increasing environmental concern about the wastewater treatment, the wastewater treatment plants are increasing in number. As a result, the sludge quantity will increase. So, in a sustainable society, the continuous and guaranteed produced sludge could be a source of raw material for fertilizer for recovery of non-renewable phosphorus and expensive nitrogen instead of simply being a waste. Similarly, non-spontaneous struvite production could be of greater importance in solving the scaling and pipeline blocking problems in sludge digestion system and also recovery of nutrients in the form of fertilizer could be addition benefit from wastewater treatment plant. The magnesium ion concentration can be limiting components (2) in sludge supernatant in comparison to dissolved phosphate and ammonium. So, it is needed to supply for reasonable quantity of struvite production.

Basically, the probability for precipitation of the struvite minerals has been determined through saturation index (SI) level, the ratio between Ion Activity Product (IAP) and equilibrium constant ( $K_{sp}$ ) in wastewater treatment plant system (2). Nucleation process for combining struvite ions are more favorable in alkaline condition that increases the super saturation state of solution enhance the struvite precipitation (19). The general reaction of struvite formation is;



And,

$$\text{Saturation Index, } SI = \log \frac{IAP}{K_{sp}}$$

Saturation index (super saturation ratio, SSR) greater than unity indicates the probability of struvite precipitation while negative value indicates the component concentration is in aqueous phase.

In addition, struvite precipitation is also affected by pH, temperature and conductivity. Anaerobic digestion of sludge releases magnesium, ammonium and phosphate that spontaneously form struvite crystals (20) in digester, heat exchanger units, pumping units and other post digestion units.

Here, temperature is the major governing factor for whole designed project. Similarly, pH is considered as second most important factor, particularly for extracting the struvite components from pellet (dry solid) fraction of the sludge. Furthermore in this project, both temperature and pH has been playing important role while investigating the possibility of struvite formation, either as problem into the pipes and heat exchanger units or as resource recovery purpose.

## MATERIALS AND METHODS

### Project design and experimental approach

This project is based on releasing of struvite compounds particularly, phosphorus from anaerobically digested sludge into the liquid phase and investigation of struvite formation possibility based on the component concentration obtained at different temperature. Anaerobically digested sludge at 55°C was collected from Aalborg Wastewater Treatment Plant west (AWWTP) for conducting the project in laboratory. First and foremost effort is to release chemical components into

supernatant with varying temperature of 25°C, 30°C, 35°C, 40°C, 45°C, 50°C, 55°C and 60°C. Second practice is to release the chemical components from separated pellet (dry solid) fraction by adding acetate buffer solution of pH of 4, 4.6 and 5.4; and tris buffer solution of pH of 7.3, 8 and 8.6 in similar range of temperature. Together with, these conceptual frameworks for project designs are based on spontaneous and non-spontaneous struvite formation respectively.

In total, about 10 liters of sludge was collected from outlet of anaerobic digester tank with temperature level of 55°C. Immediately after bringing the sludge sample to laboratory, the whole sample gallon was split into small bottles of 500 ml volume. During splitting of the sample careful consideration was given for maintaining the homogeneity of all samples. These bottles were stored in freezer (at -18°C) for further piloting the aimed project. But before starting the laboratory batch experiment, the frozen bottles were transferred to refrigerator to get totally defreeze at or below 4°C.

### Segregation of sludge supernatant

Bench scale batch experiment was set up by simulating the anaerobic digestion system. Hot water bath of 50 liters capacity, consisting covering hood on top was used to maintain the temperature and preventing water loss from evaporation during experiment operation.

### Process for extraction of assessing components from digested sludge

For each time, two bottles of sample were taken and mixed together in a beaker. Sludge was carefully mixed by pouring repeatedly from one beaker to another for obtaining reliably mixed sample. Properly mixed sludge was transferred to a 500 mL clean Borosilicate bottle. Then it was flushed with N<sub>2</sub>3.0 gas for 5 minutes for removing air content into the sludge. The bottle was placed into hot water bath under identical temperature for 4 hours for digestion. During this time, sludge in the reactor bottle was agitated with the help of magnetic stirrer at 270 rpm speed. Meanwhile, in the mid time of the experiment execution, pH of the sludge was measured with the help of SI Analytics pH meter.

The sludge under hot water bath with specified temperature was centrifuged for 10 minutes at 7100 rcf for separating the solid-liquid phase of the sludge. However, during centrifugation there is no means for temperature control which allow lowering down of specified temperature. The isolated liquid and solid are termed as supernatant and pellet respectively. Further, the supernatant was filtered by using GF-75 filter having pore size diameter of 0.3 μm to remove such particles that could not be centrifuged down and then distributed for analyzing truly dissolved components. In order to preserve the sample for dissolved phosphorus analysis, 4M H<sub>2</sub>SO<sub>4</sub> was added into the filtered sample. The extracted sample was stored in refrigerator at/or below 4°C for further analysis with the specific analytical techniques. Also, supernatant without filtration was stored for measuring the total phosphate.

### **Process for extraction of assessing components from dry pellet**

The pellet was kept into freeze at  $-18^{\circ}\text{C}$  temperature for overnight and then dried with the help of freeze dryer (in between  $-48^{\circ}\text{C}$  to  $-51^{\circ}\text{C}$ ) for 24 hours. The dried pellet were weighted and added prepared acetate and trisbuffer solution in a ratio of 1:30 (1 gmdrypellet: 30 mL buffer solution) for extracting the targeted compounds. Similar as before, the mixture of buffer solution and pellets were flushed with  $\text{N}_2$  3.0 gas for 5 minutes and kept into hot water bath for 4 hours with specified temperature. Magnetic stirrer was used to agitate the mixture and set up with speed 250 rpm. The pH of each mixture was measured in mid time of process running. The solid liquid fraction was separated by centrifugation. Further liquid fraction has been stored and used for analysis process and final solid part was discarded safely.

### **Analytical Method**

Further analytical measurement was done for struvite components, total phosphorus and other metal ions such as  $\text{Al}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{K}^{+}$  and  $\text{Fe}^{2+}$ . The possibility of struvite formation in pipes and heat exchanger units in post digestion section was determined via saturation index (SI) level concept of super saturation/oversaturation ratio.

### **Dissolved phosphorus measurement (Spectrophotometer measurement)**

For measuring the dissolved reactive phosphorus, 3 mL filtered supernatant through Glass Fiber Filter (GF-75) of diameter 25 mm having the pore size  $0.3\ \mu\text{m}$  has been taken and preserved by adding  $30\ \mu\text{L}$  of  $4\text{M H}_2\text{SO}_4$ . Further, the preserved samples were diluted by 1000 times into 50 mL test tube by taking  $50\ \mu\text{L}$  sample and added  $49.95\ \text{mL}$  of  $40\text{mM H}_2\text{SO}_4$ . Dilution of the samples was done for insuring the absorbance lies within standard curve limits.

The measurement method for dissolved reactive phosphorus is called direct colorimetry by ascorbic acid method, is adapted from Danish Standard 292 and Standard Methods 2500-P. Chemically, the ammonium molybdate and potassium antimonyl tartrate react in acidic medium with orthophosphate and form heteropoly acid-phosphomolybdic acid which is reduced to molybdenum blue colour by ascorbic acid (21). The light absorbance of molybdenum blue measured at spectrophotometric wavelength  $880\text{nm}$  gives the measurement for orthophosphate – phosphorus concentration.

### **Total phosphorus measurement (SKALAR analytical robotic measurement)**

Centrifuged supernatant without filtration was used to measure the total phosphate-phosphorus concentration by using Nanocolor robot test. The samples were diluted by 100 times into 50 mL volumetric flask by taking  $500\ \mu\text{L}$  sample and added  $49.50\ \text{mL}$  of demineralized water.

Photometric determination as molybdenum blue method was used technique in total phosphate-phosphorus measurement. Dilution of the sample was done insuring the measured value comes within range of  $0.30\text{-}15.00\ \text{mgP/L}$  using  $690\ \text{nm}$  photometric wavelengths. In this analytic method the photometric absorbance of molybdenum blue by photometer detector was measured after acidic hydrolysis (sulfuric acid 5-

15%) and oxidation (sodium peroxodisulfate 20-100%) at  $120^{\circ}\text{C}$ . Specifically, this test method is called 6802 total Phosphate-15 method and the work flow as full automatic photometric analysis on robot where the samples were taken from original sample. The Skalar-Robotic programming software has been used for computing the whole process.

### **Ionic metal concentration measurement (ICP-OES measurement)**

The dissolved elements such as Al, Ca, K, Fe and Mg were determined by inductively coupled plasma – optical emission spectrometry (ICP-OES) insupernatant sludge and extraction from dry solid (pellet). In total, 8 mL of filtered sample was taken into 15 mL tube and added  $1\ \text{mL}$  of plasma pure 67 - 69% nitric acid ( $\text{HNO}_3$ ) for digestion of selected elements and 10 ppm Yttrium (Y) as internal standard solution. Principally, the ICP-OES measures the emission rays (spectrum rays) that are correspond to the photon wavelength. Here, the selected elemental species in the samples are excited by outside plasma energy, while the excited atom return to low energy state the emission rays called spectrum rays get released. The content of each element determined depending upon the intensity of the spectrum rays. In order to generate plasma energy, the argon gas supplied to torch coil. With high frequency current, the argon gas is ionized and plasma energy is generated and used for exciting the sample elements.

### **Ammonium-nitrogen measurement (Technicon TRAACS 800 auto-analyzer)**

Filtered samples were stored in disposable scintillation vials. Depending on the need, each sample was diluted using double distilled water for  $\text{NH}_4\text{-N}$  analysis. Technicon TRAACS 800, the flow-through auto-analyzer was used to measure the  $\text{NH}_4\text{-N}$  concentration. Principally, this is a colorimetric method is called the Indophenol blue spectrophotometric method. In this method the  $\text{NH}_4\text{-N}$  reacts with phenol and then sodium hypochlorite ( $\text{NaOCl}$ ) in the catalysis of nitroprussidate produce indophenol blue color. The absorbance of indophenol blue has been measured at wavelength of  $640\ \text{nm}$ . This method is based on Berthelot reaction (22).

### **Visual MINTEQ analysis for super/over saturation ratio**

Visual MINTEQ was used for identification of mineral speciation and calculation of solubility status through chemical equilibrium of the chemical elements based on input constituent concentration. It is used to determine the saturation index (SI) of struvite and also equilibrium struvite amount based on measured concentration of  $\text{Mg}^{2+}$ ,  $\text{NH}_4^{+}$  and  $\text{PO}_4^{3-}$  with specified pH and temperature.

## **RESULTS AND DISCUSSION**

### **Metal Concentration in Supernatant and Dry Pellet (Solid Extraction)**

Dry solid content from the sampled sludge is found  $32.5\ \text{mg/g}$  of sludge i.e. 3.25%. From the supernatant (liquid) phase of the sludge that received after 4 hours processing in hot water bath has varying concentration of the metal ions such as Al, Ca, Fe, K and Mg. The wastewater receives from the industrial and municipal sectors so these metals are also enter from the same sources. But the iron compounds also come from nutrient removal technique for phosphorus, where the iron chloride has

been using as precipitants for chemical removal of phosphorus. The possible ligands of these metals in the sludge are in general are insoluble and precipitates in basic pH (23) and lower temperature. But increasing temperature could raise the ion activation along with decreasing pH which increases the dissolved concentration.

**Table 1** Measured metal concentration in supernatant sludge with varying temperature

Temperature (°C)	Al <sup>3+</sup> (mg/L)	Ca <sup>2+</sup> (mg/L)	Fe <sup>2+</sup> (mg/L)	K <sup>+</sup> (mg/L)	Mg <sup>2+</sup> (mg/L)
25	0.68	27.52	2.14	339.36	3.08
30	0.78	17.38	2.81	312.92	6.25
35	0.77	19.96	3.22	305.88	6.09
40	0.78	19.89	3.01	311.98	8.56
45	0.73	18.46	2.76	321.48	12.50
50	0.57	17.23	2.49	330.09	18.53
55	0.71	17.56	2.84	341.74	25.27
60	1.71	18.03	3.91	364.37	23.96

From pellet (solid) part of the sludge after drying, the buffer solution used for dissolution shows higher concentration with lower pH buffer that acetate buffer than the used tris buffer. This further ascertains the complex ligands are not able to dissociate into supernatant phase with slightly basic pH 7.02 at 60°C to 7.50 at 30°C. The lower pH after addition of buffer (as shown in Table 2) is more efficient in dissolving Ca, Fe, K and Mg (except Al) metals present in the sludge, especially pH 4 buffer solution is highly effective. The higher concentration of aluminum with basic tris buffer extraction shows the tendency of dissociation with high pH while the complex aluminum ligands are in precipitating forms (solid) in the acidic pH level (24). However, we cannot see significant and clear pattern of change with increasing temperature for the pellet. It might be due to sudden change of the temperature during whole procedure proceeding during the sample extraction.

The chemical elements allied together with chemical bonds such as covalent, ionic, metallic and/or Vander Waals force. For most of the minerals, two or more types of bonds can and do coexist and form a complex compound under specific temperature and pH conditions. Normally, the metal elements in nature exist as positively ionic (cation) forms and the mineral compounds are ionic. Chemically, the relevant complex mineral type that could present in sludge based on the observed metal ions are Al(OH)<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, boehmite [Al(OH)], brucite [Mg(OH)<sub>2</sub>], Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, Ca<sub>4</sub>H(PO<sub>4</sub>)<sub>3</sub>.3H<sub>2</sub>O(s), CaHPO<sub>4</sub>(s), CaHPO<sub>4</sub>.2H<sub>2</sub>O(s), Fe(OH)<sub>2</sub>, gibbsite [Al(OH)<sub>3</sub>], hercynite [FeAl<sub>2</sub>O<sub>4</sub>], hydroxyapatite [Ca<sub>10</sub>(PO<sub>4</sub>)(OH)<sub>2</sub>], Mg(OH)<sub>2</sub>, Mg<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, MgHPO<sub>4</sub>.3H<sub>2</sub>O, periclase [MgO], portlandite [Ca(OH)<sub>2</sub>], spinel [MgAl<sub>2</sub>O<sub>4</sub>], magnesium struvite [MgNH<sub>4</sub>PO<sub>4</sub>.6H<sub>2</sub>O], variscite [AlPO<sub>4</sub>.2H<sub>2</sub>O], vivianite [Fe<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>.8H<sub>2</sub>O], KOH, K<sub>2</sub>HPO<sub>4</sub>, KH<sub>2</sub>PO<sub>4</sub>, potassium struvite [KNH<sub>4</sub>PO<sub>4</sub>.6H<sub>2</sub>O].

Dissolution or the precipitation of these minerals control by the law of mass action which shows chemical reaction is proportional to the product of masses of the reacting substance and further reaction might proceed depending on the ionic concentration of various elements species and capability to bond each other.

The chemical elements dissociated from the complex ligands into sludge supernatant (liquid) in ionic form. This is a process

of transferring of chemical elements from solid phase into the liquid phase. The order of metal concentrations (mg/L) found is K>Ca>Mg>Fe>Al in supernatant in all temperature condition.

**Table 2** Metal concentration in pellet extraction by using 1gm dry pellet: 30mL buffer solution

Temperature (°C)	Used buffer pH	Concentration of components (mg/L)				
		Al (mg/L)	Ca (mg/L)	Fe (mg/L)	K (mg/L)	Mg (mg/L)
25	4	0.12	357.20	4.36	101.24	121.28
	4.6	0.22	155.45	0.94	107.04	100.80
	5.4	0.29	26.67	0.77	108.80	43.40
	7.3	0.73	12.99	1.79	68.13	15.24
	8	1.07	10.74	2.54	65.13	7.37
	8.6	1.15	8.64	3.49	54.18	3.12
30	4	0.05	381.05	16.34	85.20	129.75
	4.6	0.05	179.65	3.68	90.72	109.20
	5.4	0.14	25.37	0.58	90.10	50.30
	7.3	0.28	10.44	1.26	60.57	31.02
	8	0.51	8.60	2.05	58.68	11.64
	8.6	0.74	7.59	3.29	54.53	6.20
35	4	0.02	361.70	27.60	94.50	130.93
	4.6	0.02	173.45	5.94	101.17	110.36
	5.4	0.16	24.63	0.65	98.68	43.63
	7.3	0.41	11.05	1.35	69.82	29.57
	8	0.67	9.06	2.08	67.12	12.30
	8.6	1.03	8.57	3.25	59.68	4.62
40	4	0.05	391.80	24.05	98.20	126.48
	4.6	0.28	177.40	1.29	101.76	100.35
	5.4	0.40	30.05	1.03	102.75	48.06
	7.3	0.79	12.39	2.14	67.54	30.03
	8	0.68	9.47	1.87	66.51	14.75
	8.6	1.04	8.70	2.96	60.09	6.38
45	4	0.07	340.10	8.80	90.99	109.32
	4.6	0.16	160.95	1.57	97.45	86.85
	5.4	0.32	25.77	0.82	101.30	42.55
	7.3	0.56	10.52	1.32	68.61	22.69
	8	0.69	8.84	1.86	67.31	7.42
	8.6	1.16	8.91	3.06	57.10	4.56
50	4	0.01	286.90	13.27	92.09	125.10
	4.6	0.27	131.45	2.32	98.22	99.28
	5.4	0.69	18.84	1.53	101.25	45.03
	7.3	0.78	10.22	1.81	69.06	17.47
	8	0.88	8.76	2.07	68.40	7.27
	8.6	0.98	8.44	2.48	64.31	4.46
55	4	0.01	319.05	17.90	97.53	106.99
	4.6	0.15	146.10	1.62	100.08	80.03
	5.4	0.38	26.03	1.00	109.75	40.14
	7.3	0.72	11.45	1.72	69.89	19.03
	8	0.71	8.51	1.76	68.23	7.91
	8.6	0.92	8.93	2.77	64.28	6.12
60	4	0.02	302.15	0.89	96.72	97.85
	4.6	0.07	171.00	3.36	108.29	86.42
	5.4	0.27	41.41	0.81	116.45	51.29
	7.3	0.63	13.55	1.43	75.40	24.87
	8	0.88	10.06	2.00	72.26	19.57
	8.6	1.75	13.55	4.71	65.45	6.98

Similarly, after changing the pH of sludge at 25°C the order of metal concentrations obtained is K>Ca>Mg>Fe>Al obtained in supernatant. Further, using buffer solution of pH 4 and 4.6, the obtained order of concentration is Ca>Mg>K>Fe>Al from the dry pellet. With buffer solution of pH 5.4, is K>Mg>Ca>Fe>Al. Similarly, by using buffer solution of pH 7.3, 8 and 8.6, the order is K>Mg>Ca>Fe>Al. The magnitude of Ca, Mg and K is also 2-4 order higher in comparison to Fe and Al with varying pH.

**Concentration of NH<sub>4</sub>-N and dissolved PO<sub>4</sub>-P and total Phosphorus in supernatant phase**

Ammonium- nitrogen, phosphate-phosphorus and total phosphorus found in the supernatant fraction with varying temperature has been mentioned in Table 3.

**Table 3** Measured concentration in supernatant sludge with varying temperature

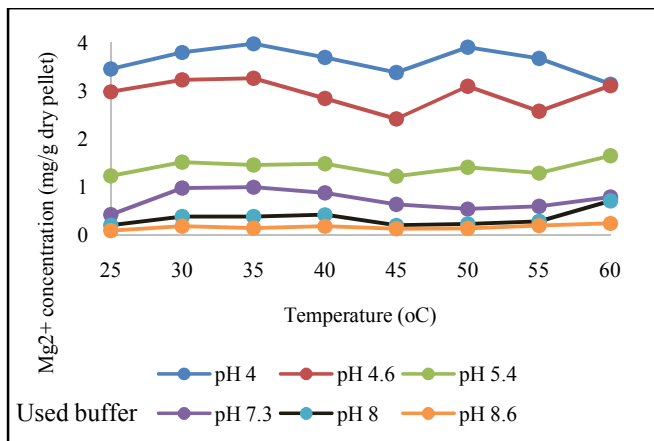
Temperature (°C)	NH <sub>4</sub> -N (mg/L)	PO <sub>4</sub> -P (mg/L)	Total P (mg/L)
25	442.12	409.55	1073.33
30	651.97	416.62	1096.67
35	553.96	434.82	1100.00
40	1133.22	432.46	1123.33
45	1205.05	446.61	1163.33
50	1196.11	461.43	1160.00
55	695.74	483.67	1213.33
60	663.67	504.73	1233.33

A static pattern of NH<sub>4</sub>-N concentration for first three temperatures conditional set up has been found. Later on from 40°C up to 50°C there is sudden rise but again lower down with 55°C and 60°C. Resultant positive rate for NH<sub>4</sub>-N from 25°C to 45°C is 38.15mg/L/°C. Similarly, the negative rate of change from 45°C to 60°C is 36.09 mg/L/°C.

There is a steady increase of dissolved PO<sub>4</sub>-P with increasing temperature from 25°C to 60°C has been observed with different temperature. The rate of PO<sub>4</sub>-P dissolution with respect to temperature is found 2.714 mg/L/°C. Further, total phosphorus concentration in supernatant phase found increasing with increasing temperature from 25°C to 60°C. Comparatively, dissolved phosphorus is found as an average of 39.15% of total P in the supernatant phase.

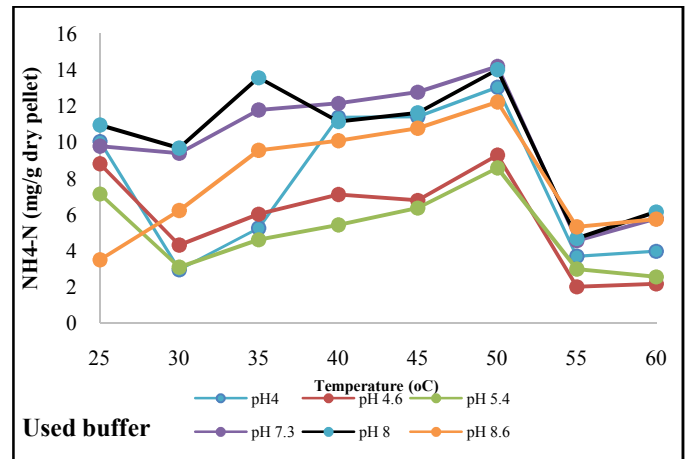
**Extracted Mg, NH<sub>4</sub>-N and PO<sub>4</sub>-P from dry pellet into liquid phase**

Considerably acetate buffer solution with pH of 4, 4.6 and 5.4 has higher extracting amount in comparison to tris buffer with pH of 7.3, 8 and 8.6. Quiet decreasing amount with increasing pH level can be observed in figure 1. However, increasing temperature has no significant change in extraction of Mg<sup>2+</sup> from the pellet with all respective pH buffer solution.



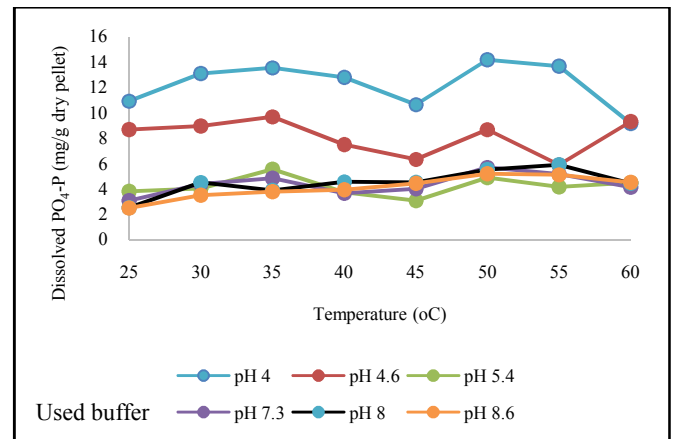
**Figure 1** Mg<sup>2+</sup> amount extracted from unit weight of dry pellet

But tris buffer solution was found more effective for extraction of NH<sub>4</sub>-N from dry pellet than acetate buffer solution as mentioned in Figure 2.



**Figure 2** Extracted NH<sub>4</sub>-N amount from dry pellet

Further, for releasing of PO<sub>4</sub>-P from dry pellet into liquid phase acetate buffer of pH 4 and pH 4.6 was found effective. However, tendency of acetate buffer of pH 5.4 was found somehow similar or lower than tris buffer with higher temperature condition as shown in figure3.



**Figure 3** Extracted dissolved PO<sub>4</sub>-P from dry pellet

The order of struvite component concentration (mg/L) from the supernatant (liquid) phase is NH<sub>4</sub>-N > PO<sub>4</sub>-P > Mg<sup>2+</sup> for all temperature conditions. But, from dry pellet extraction, irregular amount has been found. Most of the phosphate salts dissolved in acidic condition and in combination with thermal hydrolysis, most of biologically bound phosphorus could release. More than 90% of phosphorus in sludge is possible to dissolve depending upon the quality. Similarly, in basic level, the sludge components react differently. The ammonia and hydrogen carbonate dissolved and hydroxide ions left that increase the pH(25). Magnesium is always a limiting factor for struvite formation. However, the molar concentration (mol/L) for all conditions found in NH<sub>4</sub><sup>+</sup> > PO<sub>4</sub><sup>3-</sup> > Mg<sup>2+</sup> order which can illustrate the lack of magnesium and phosphate for equal molar ratio with ammonium. From the results we can see quiet lower magnitude of magnesium concentration that limit the struvite precipitation. Due considering the high ammonium concentration, it is needed to add magnesium and phosphate to precipitate as struvite in the reactors (27). Depending on necessity for addition of limiting components, struvite precipitation could be a technique for recovery of NH<sub>4</sub><sup>+</sup>, PO<sub>4</sub><sup>3-</sup> or both (28). The inflow load of phosphorus into AWWTP is 286 kg/day having flow rate of wastewater 52906 m<sup>3</sup>/day. This

gives 5.4 mg/L of phosphorus in wastewater inflow into the plant (29). At ambient (25°C) temperature the extracted concentration (mg/L) of phosphate phosphorus into liquid phase is found approximately 76 times higher than inflow concentration (mg/L). Similarly, at thermophilic (60°C) temperature it is approximately 93 times greater compared to inflow concentration into treatment plant. Release of phosphorus into supernatant phase found higher with increasing temperature, but from the pellet though using same pH buffer solution for all condition, it is constant in extraction amount with rising temperature. This might be due to limited ability of present project procedure and also some limiting factors in maintaining the temperature and pH level in the laboratory set up for extraction from available orthophosphate in sludge. However, with lower pH buffer solution of pH of 4 and 4.6, comparatively higher amount might be due to hydrolysis of condensed phosphate (such as polyphosphates ( $P_3O_{10}^{5-}$ ) and pyrophosphate ( $H_4P_2O_7$ )) and organically bound phosphate into aqueous solution and converted into orthophosphate form (30).

Further comparing the release of struvite constituents in between supernatant sludge and dry solid by using present project procedure it is found the proportion of pellet (solid) extraction to supernatant (liquid) amount for magnesium is about 0.25 at 50°C using tris buffer of pH 8.6 to maximum 40 at 25°C using acetate buffer of pH 4. But ammonium extraction from pellet is below supernatant fraction. Pellet to supernatant proportion varies from 0.09 at 55°C using acetate buffer pH of 4.6 to 0.94 at 25°C using tris buffer pH of 8 for pellet extraction. Again for phosphate, the supernatant and pellet extraction using acetate buffer has found approximately equal proportion of sharing in unit weight of sludge. For example at 30°C using buffer of pH 4 it is found 1.09 times amount in pellet fraction than supernatant. But tris buffer is not found significant for phosphate extraction such as only 0.22 times of phosphate in pellet fraction than supernatant able to extract using tris buffer of pH 8.6 at 25°C.

Analytically, the value of magnesium, ammonium and phosphate found extremely higher in unit weight of dried pellet. For instant, the magnesium proportion for dry pellet to supernatant ranged between 7.26 at 50°C using tris buffer pH of 8.6 and 1122.71 at 25°C using acetate buffer of pH 4. Ammonium proportion for dry pellet to supernatant ranged between 2.91 at 55°C using acetate buffer of pH 4.6 to higher proportion of 24.75 at 25°C using tris buffer of pH 8. Similarly, the proportion of dry pellet extraction to supernatant for  $PO_4^{3-}$  is 6.05 at 60°C using acetate buffer of pH 4 to 31.41 at 25°C using acetate buffer of pH 4.6. On other hand, quite higher proportion of total phosphorus in extracted liquid samples verify significant amount of phosphorus bound with small particles after centrifugation techniques for solid-liquid separation. This will not take part in crystal formation in the system.

#### Saturation index (SI) level for struvite

##### SI level of struvite from constituent concentration of supernatant

Measured database for ionic concentration of struvite components ( $Mg^{2+}$ ,  $NH_4^+$  and  $PO_4^{3-}$ ) obtained in supernatant

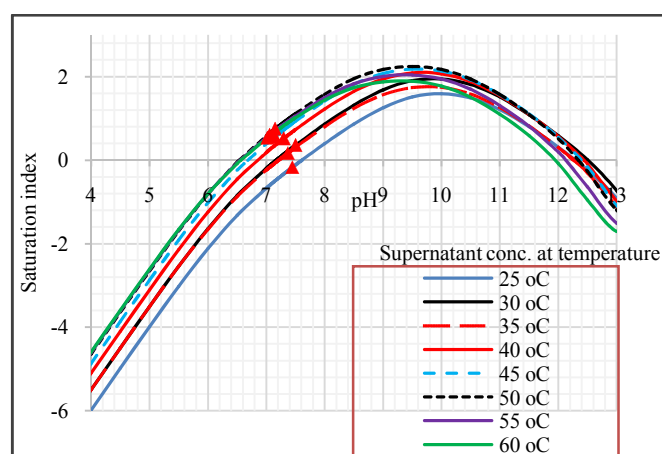
fraction used to calculate the level of saturation index (SI) with the help of Visual MINTEQ 3.0 version. The SI level indicates the ionic activity product of the dissolved components that is being correlated with constituent concentration, temperature (and solubility constant is temperature dependent) and alkalinity. This software also gives the chemical mineral speciation based on input concentration of measured chemical parameters. The measured concentration of  $NH_4^+$  &  $PO_4^{3-}$  that is used for calculating SI value with different temperature is given in Table 4.

**Table 4** Struvite constituent concentration obtained in supernatant fraction of sludge

Temp	$NH_4^+$ (mg/L)	$PO_4^{3-}$ (mg/L)
25	569.39	1255.35
30	839.64	1277.04
35	713.43	1332.81
40	1459.43	1325.58
45	1551.94	1368.95
50	1540.42	1414.39
55	896.01	1482.56
60	854.72	1547.10

The aqueous inorganic species that can be formed depending upon the input parameters are  $H_2PO_4^-$ ,  $H_3PO_4$ ,  $HPO_4^{2-}$ ,  $Mg(NH_3)_2^{2+}$ ,  $Mg^{2+}$ ,  $MgHPO_4$  (aq),  $MgOH^+$ ,  $MgPO_4^-$ ,  $NH_3$  (aq),  $NH_4^+$ ,  $OH^-$ ,  $PO_4^{3-}$ . Further, the percentage variation for dissolved species such as  $Mg^{2+}$ ,  $NH_4^+$  &  $PO_4^{3-}$  and adsorbed species such as  $H_2PO_4^-$ ,  $H_3PO_4$ ,  $HPO_4^{2-}$ ,  $Mg(NH_3)_2^{2+}$ ,  $MgHPO_4$  (aq),  $MgOH^+$ ,  $MgPO_4^-$  based on set up temperature and pH conditions can be specified from the constituent concentration input into the program.

Further, the other possible minerals that can formed along with struvite on inserting the magnesium, ammonium and phosphate ion concentration are brucite, periclase,  $Mg(OH)_2$ ,  $Mg_3(PO_4)_2$  and  $MgHPO_4 \cdot 3H_2O$ . However, struvite is the focused mineral component here for the present project. Hence, the saturation index for struvite for determining the possibility for precipitation has been determined and also simulated with respect to pH.



**Figure 4** Saturation index level for struvite at different temperature condition with varying pH

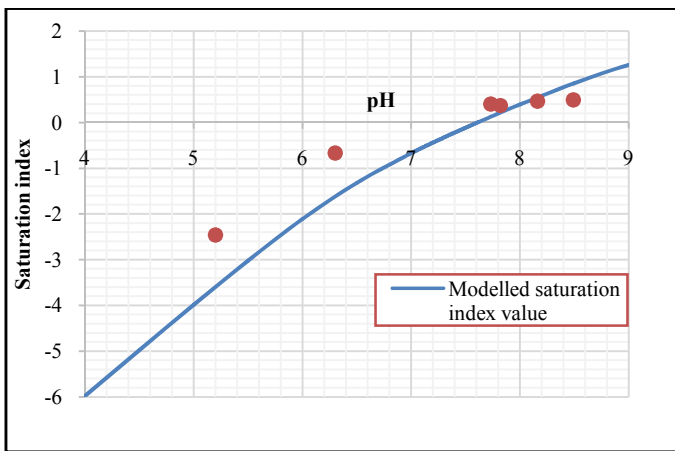
Saturation index (SI) level for struvite mineral based on the extracted supernatant concentration of  $Mg^{2+}$ ,  $NH_4^+$  and  $PO_4^{3-}$  in different set up temperature condition is presented in Figure 4. The highest saturation index (SI) for struvite found is 0.748 at 50°C followed by 0.605 at 55°C, 0.562 at 45°C, 0.537 at 60°C,

0.521 at 40°C, 0.36 at 30°C, 0.171 at 35°C and lowest value of -0.167 at 25°C (red points indicate the saturation index value for different temperature in figure 4). Accordingly, for set up temperature condition, the obtained concentrations of the component have been modeled with varying pH. The maximum possibility of struvite formation via the perspective of saturation index greater than 1, obtained value can be stated in between pH of 8.5 and 11 and further started lowering down with increasing pH level. With constant temperature and varying pH, two equilibrium levels (where the SI value is zero) have been observed. The lower and upper pH limit for apparent equilibrium level for saturation index of struvite with different constant temperature has been mentioned in Table 5. Below the lower equilibrium limit and above the upper apparent equilibrium limit, the saturation index levels are in negative numeric meaning total amount of components are in dissolved form.

**Table 5** Lower and upper apparent equilibrium (SI level is zero) level for struvite saturation index

Temperature (°C)	25	30	35	40	45	50	55	60
Lower equilibrium pH	7.61	7.17	7.21	6.85	6.68	6.53	6.55	6.56
Upper equilibrium pH	12.293	12.51	12.27	12.44	12.40	12.35	12.13	11.95

Again, pH of sludge changed by adding acetate and tris buffer in order to release of chemical components and to see the possibility for struvite precipitation. The volume of sludge after changing the pH level has maintained. It was simply done by dewatering sludge to 30% in freeze drier and added required volume of buffer for gaining the lost water content so as to retain the sludge volume as before.



**Figure 5** Saturation index level for supernatant sludge with changing pH at 25°C In

Figure 5, struvite saturation index (SI) level for the supernatant sludge at 25°C with different pH level and the solid line is the modeled saturation indices level from the real constituent's concentration of struvite at same temperature. The measured component concentration of  $Mg^{2+}$ ,  $NH_4^+$  &  $PO_4^{3-}$  with varying pH are as shown in Table 6.

**Table 6** Measured concentration of struvite constituents at 25°C by changing pH of sludge

pH	$Mg^{2+}$ (mg/L)	$NH_4^+$ (mg/L)	$PO_4^{3-}$ (mg/L)
5.2	42.67	839.70	940.80
6.3	35.90	732.91	689.86
7.3	5.52	869.50	531.86
7.82	3.63	932.89	646.49
8.16	2.17	950.18	522.56
8.49	1.43	785.77	426.52

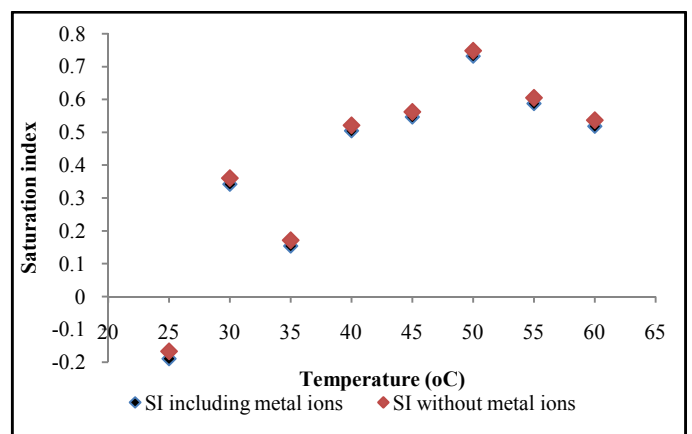
As shown in Table 5 the apparent equilibrium for struvite found at pH of 7.61 for the constant constituent concentration of 25°C. From the measured concentration for same temperature at changing pH of 5.2 and 6.3 also found negative that is  $IAP < K_{sp}$ . Likewise, at pH of 7.73, 7.82, 8.16 and 8.49 found positive which indicate  $IAP > K_{sp}$  and there is probability for struvite formation with raising pH in constant 25°C temperature. However, by lowering pH the struvite precipitation will not take place with same constituent concentration at constant ambient temperature.

Many literatures reported higher probability of struvite crystallization is found within the pH range of 7-11 (31). Besides, in this range of pH, crystals trimagnesium phosphate [ $Mg_3(PO_4)_2 \cdot 6H_2O$ ], magnesium hydrogen phosphate [ $MgHPO_4 \cdot 3H_2O$ ], and bobierrite [ $Mg_3(PO_4)_2 \cdot 8H_2O$ ] can be formed (32). The SI level with existing constituent concentration of struvite in supernatant phase alone is quiet below i.e. lower than 1 at measured level of pH with specified temperature of different set up condition as shown in.

Figure 4, that shows also prove the insignificant possibility of struvite crystallization.

**Influence of metals on saturation index of struvite mineral**

Presence of other chemical elements including metals undoubtedly forms more complex compounds in the sludge system. It also interferes in struvite constituent's dissociation and possibility of formation. Some of the light metal cations also inhibit the anaerobic digestion along with heavy metals and considered as toxic substance for the microbial population. The metal cations such  $Al^{3+}$ ,  $Ca^{2+}$ ,  $Fe^{2+}$  and  $K^+$  measured in supernatant sludge with varying temperature have been shown in Table 1. Along with the struvite components concentration measured into the supernatant phase these metal ions also inserted for Visual MINTEQ calculation for SI level and found slight variation obtained for SI value for struvite component with and without these metal concentrations is mentioned in Figure 6. Difference in SI value after including these metal concentrations shows possibility for formation of multiple other species and reduced tendency of struvite precipitation.



**Figure 6** Difference in saturation index of struvite precipitation with and without including metal ions

Most probably, re-union of the chemicals into complex sludge matrix could be only after when they get released into the liquid phase with thermo-chemical hydrolysis of sludge matrix. Thus, it can be said the problem creating struvite precipitate is most probably from sludge supernatant fraction. But presence

of some other metallic ions decreases the struvite precipitation possibility and have possibility to form other solid compounds as such as brucite  $[Mg(OH)_2]$ , gibbsite  $[Al(OH)_3]$ , hercynite  $[FeAl_2O_4]$ , hydroxyapatite  $[Ca_{10}(PO_4)(OH)_2]$ , variscite  $[AlPO_4 \cdot 2H_2O]$  and vivianite  $[Fe_3(PO_4)_2 \cdot 8H_2O]$ .

#### Temperature effect on equilibrium amount (mg/L) of precipitated struvite from supernatant

Simulated value of equilibrium struvite amount (mg/L) for dissolved component concentration of  $Mg^{2+}$ ,  $NH_4^+$  &  $PO_4^{3-}$  obtained at different temperature conditions is given in Figure 7. Similarly, simulation made by lowering temperature level. This indicates the dissolved components concentration for struvite that obtained into supernatant (liquid) phase from sludge matrix at certain level of temperature condition could crystallize on dropping down temperature. The solubility constant is fixed to -13.26 and a standard enthalpy of reaction (dHr) 23.62 Kcal/mol (98.83 kJ/mol) for struvite is also specified for correcting the solubility constant for determination of struvite equilibrium amount (mg/L).

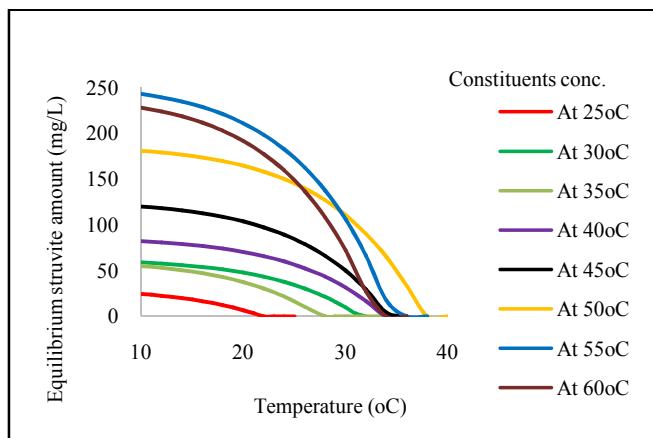


Figure 7 Simulated equilibrium struvite amount with respect to temperature

It has been noticed that the highest struvite amount (mg/L) found with the released components concentration at 55°C followed by 60°C, and precipitating around 35°C. The constituent concentration obtained at 50°C is precipitating around 38°C. Further, released struvite components at 45°C, 40°C, 35°C, 30°C and 25°C started to precipitate around 34°C, 32°C, 31°C, 26°C and 21°C.

With specified condition of pH and temperature the dissolved components concentration balanced between dissolved, sorbed and precipitating phase. However, the precipitation of specified struvite mineral found higher with decreased temperature. Temperature lower down after digestion and spontaneously precipitated struvite into the heat exchanger, pumping and other post-digestion units is hard to dissolve on decreasing temperature. For example, temperature of digested sludge at 55°C in AWWTP has reported to lower down upto 18°C in storage tank depending upon time of storage.

One can say, once precipitated struvite components during sufficient concentration and reach to super saturation condition can be deposited into the pipelines. Based on dissolved concentration level and specified temperature and pH, the precipitated proportion of magnesium varies approximately in between 94% to 97% (dissolved approximately 3% to 6%),

ammonium 2% to 11% (dissolved 89% to 98%) and phosphate approximately in between 8% to 20% (dissolved 80% to 92%).

Similarly, modeling for measured dissolved concentration of  $Mg^{2+}$ ,  $NH_4^+$  &  $PO_4^{3-}$  in supernatant sludge by varying pH level and constant temperature for each individual concentration level was made and presented in Figure 8. In this time, the constant temperature that was set up for reactor bottles during project operation was specified for simulation.

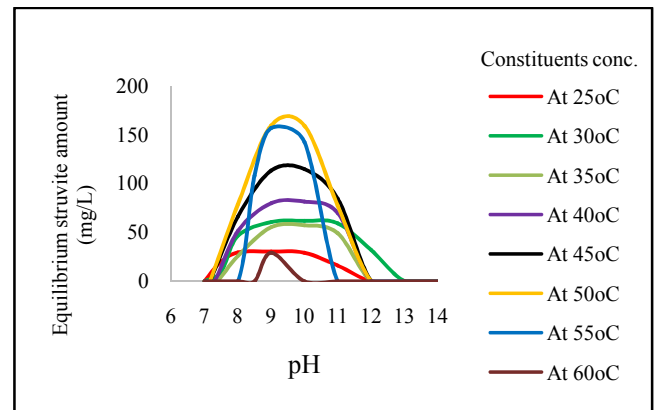


Figure 8 Simulated equilibrium struvite amount with respect to varying pH

Depending upon the proportion of dissolved constituent concentration of  $Mg^{2+}$ ,  $NH_4^+$  &  $PO_4^{3-}$ , the highest equilibrium amount of struvite (mg/L) found at 50°C followed by 55°C, 45°C, 40°C, 30°C, 35°C, 25°C and 60°C. Likewise, wide range of pH range for struvite precipitation found for constituent concentration released at 30°C is approximately in between 7 to 13. Further, the constituent concentration at 25°C, 35°C, 40°C, 45°C and 50°C get precipitated approximately in between 7 to 12. Similarly, for concentration obtained at 55°C precipitated in between 8 to 11 and for 60°C ranged in between 8.5 to 10.

#### Equilibrium amount (mg/L) of precipitated struvite from combined supernatant and pellet concentration

Equilibrium struvite amount from total released concentration into liquid phase, both from supernatant and pellet extraction has been determined by specifying solubility constant of -13.26 and a standard enthalpy of reaction (dHr) 23.62 Kcal/mol (98.83 kJ/mol) for struvite for correcting the solubility constant. Extraction from pellet (dry solid) was done by regaining the lost liquid fraction through addition of buffer solutions in a ratio of 1 g dry pellet:30 mL buffer solution. The specified temperature is 25°C and specified pH values for components concentration obtained at 25°C, 30°C, 35°C, 40°C, 45°C, 50°C, 55°C and 60°C are 7.45, 7.5, 7.37, 7.3, 7.15, 7.15, 7.06 and 7.02 respectively. These specified pH values into the program are the measured pH for the sludge matrix in reactor bottles during project operation for each set up temperature condition.



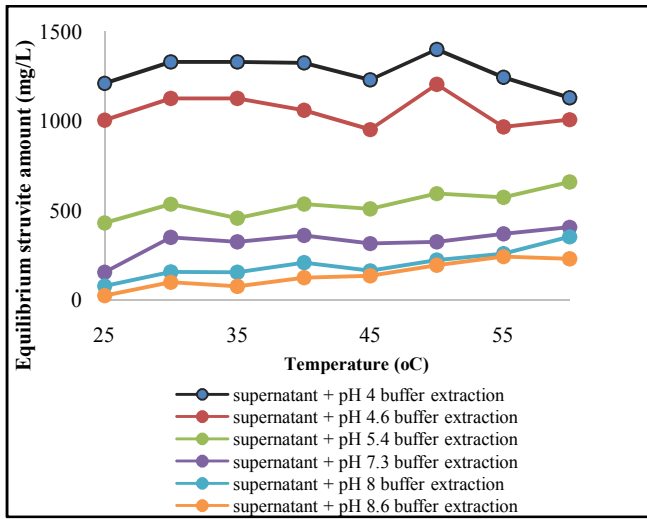


Figure 9 Equilibrium struvite amount for sludge at different temperature

From

Figure 9 higher equilibrium struvite amount (mg/L) has been found with concentration from supernatant and buffer extraction using pH of 4, followed by pH of 4.6, 5.4, 7.3, 8 and 8.6. And also the slope of the line for equilibrium struvite amount shows static type for sludge extraction using acetate buffer of pH 4, 4.6 with varying temperature. However, the pellet extraction using buffer solution of pH 5.4, 7.3, 8 and 8.6 shows slight increase with rising temperature.

During analysis with Visual MINTEQ the precipitated proportion for magnesium has found approximately in between 72% to 97% (dissolved 18% to 3%), ammonium precipitation from less than 1% to 11% (dissolved 89% to more than 99%) and phosphate ranges 1.80% to 20% (dissolved 80% to approximately 98%).

**Possible struvite production (by adding Mg<sup>2+</sup> source)**

Magnesium salt can be added to recover nutrient from the daily produced sludge especially phosphate in the form of struvite. Total struvite that can be produced with respect to increasing temperature and extracting components from pellet using buffer solutions has been shown in Figure 10.

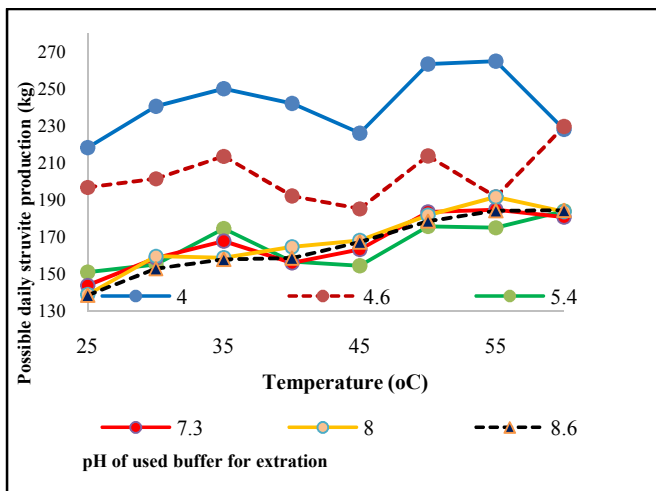


Figure 10 Possible daily struvite recovery from digested sludge by adding Mg<sup>2+</sup>

The lowest struvite production possibility is calculated 138.47 kg/day having 23.40 kg/day contribution from the pellet extraction using tris buffer solution pH of 8.6 (i.e. about 83% from supernatant fraction and 17% from pellet fraction) at 25°C. Similarly, the highest possibility is 264.90 kg/day found at 55°C where pellet contribution is 129.01kg/day (i.e. about 51.30% from supernatant fraction and 48.70% from pellet fraction) by using acetate buffer solution pH of 4.

By above calculation, on daily basis the possible proportion of phosphate recovery from the treatment plant in the form of struvite ranges in between 6.11% at 25°C (including the pellet extraction using tris buffer pH of 8.6) to 11.70% of total phosphorus load into the treatment plant at 55°C (including the pellet extraction using acetate buffer pH of 4).

The recovered supernatant and pellet part of the sludge were analyzed for the recovery of nutrients such as phosphorus and nitrogen. Together with spontaneous struvite formation there is possibility of more production by supplying extra amount of magnesium salt and also sometime phosphate source where ammonium recovery has been targeted. For this project the recovery of the phosphate has been put into the priority both from supernatant and pellet solid fraction. Depending upon the thermodynamic effect for release of phosphate, the highest efficiency of dissolution found at thermophilic condition of 55-60°C. Similarly, on analyzing the individual condition for molar ratio of 1Mg<sup>2+</sup>:1NH<sub>4</sub><sup>+</sup>:1PO<sub>4</sub><sup>3-</sup> of chemical elements from pellet, magnesium and/or phosphate becomes the limiting factor. The higher efficiency of release is observed by using lower pH buffer solutions. Addition of magnesium salt has increases the struvite production by 5-9 folds by using acetate buffer pH of 4, 4.6 and 5.3. Similarly, the production amount has huge difference with higher pH of 7.3, 8 and 8.6 extraction such as the amount increase by 10-64 folds. Production amount accounted approximately 1.5 times greater including supernatant and extraction amount using buffer of pH of 4 in comparison to supernatant and extraction amount using buffer of pH of 8.6.

Shows the sharing of phosphorus recovery from supernatant and pellet fraction from present project procedure where acetate and tris buffer were used as solution to extraction from the dry pellet.

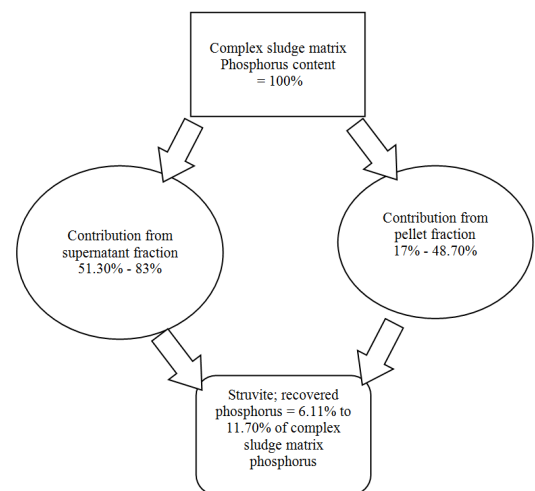


Figure 11 Recovery of phosphorus from sludge matrix and contribution from supernatant and pellet fraction

By present project practice the maximum possibility for recovery of phosphate from sludge in the form of struvite is 11.70 % of total possible daily struvite production based on phosphorus load into the treatment plant. The limiting factors at present project for phosphorus recovery might be inability for maintaining selected temperature during whole process procedure. Such as the sludge sample under hot water bath with 60°C for laboratory digestion lowered down to 38.5°C after centrifugation for solid-liquid separation. Also in an average about 40 minutes of time lapse was ensued for sample taken from the reactor bottles, centrifugation, solid-liquid separation, filtration and preparation for further parameter analysis following the necessary steps for storage such as addition of 4M H<sub>2</sub>SO<sub>4</sub> into filtered sample for dissolved phosphorus analysis. More analytically, laboratory limitations for maintaining temperature during these practices could be the reason behind overall low efficiency of present methodology for releasing amount of phosphorus into liquid phase. Simultaneously, the released chemical constituents also have enough time and condition for forming other chemical matrix such as brucite, gibbsite, hercynite, hydroxyapatite, variscite and vivianite other than struvite in the system.

Besides, various process parameters including temperature, pH, Mg:P ratio, degree of mixing and super saturation ratio are probably affect the struvite precipitation (33). The addition of magnesium salt has been considered for full recovery of phosphate with a molar ratio of 1Mg<sup>2+</sup>:1NH<sub>4</sub><sup>+</sup>:1PO<sub>4</sub><sup>3-</sup> but many research reported 80-90% recovery from anaerobic digester sludge (34). Similarly the precipitation efficiency obtained 78-95% and recovery efficiency value obtained 46-86% experimentally from anaerobically digested sludge. Considering 78-95% struvite precipitation efficiency (35) from the supernatant (liquid) fraction of sludge, about 90-109 kg/day at ambient temperature and 111-135 kg/day at thermophilic temperature can be produced from AWWTP by applying present project methods as well and adding magnesium. With similar approach by including the thermochemical release of phosphate from pellet (solid) fraction and supernatant fraction about 108-132 kg/day at ambient temperature (25°C) and 207-252 kg/day at thermophilic temperature (55°C) can be precipitated. Addition of Mg(OH)<sub>2</sub> can be recommended for pH maintenance and limiting magnesium source in order to obtained feasible quantity of struvite.

## CONCLUSION

From the present project, it can be concluded that temperature and pH are highly determining factors for struvite formation from the released constituent concentrations into liquid phase. Also we can say phosphorus release from the sludge matrix is positively related with temperature. However, the spontaneous struvite formation from available component concentration is very insignificant amount in comparison to daily produced sludge volume. This shows lesser probability of struvite formation problem into the anaerobic sludge digestion system. And also presence of metal cations such as Al<sup>3+</sup>, Ca<sup>2+</sup>, K<sup>+</sup> and also Fe<sup>2+</sup> shows negative influence for struvite crystallization and but shows probability for other complex ligands into the system.

Recovery of phosphate in the form of struvite requires supply of many folds magnesium. In an aggregate for sludge matrix, extraction from pellet fraction especially with using acetate buffer of lower pH shows a significant contribution for phosphorus recovery in the form of struvite. Similarly, for all condition the molar concentration for ammonium ion is sufficient in comparison to magnesium and phosphate for struvite precipitation. Finally, based on the applied methodology for resource recovery, it can be recommended to select thermophilic temperature (50-60°C) for obtaining higher struvite amount into sludge supernatant fraction. Similarly, acidic buffer solution with lower pH is beneficial for obtaining higher phosphorus from pellet fractions. And for struvite crystallization needed higher pH level.

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