Research Journal of Pharmaceutical, Biological and Chemical Sciences

Process Design for Struvite Precipitation from Industrial Wastewater Stream: Special Design.

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ABSTRACT

A system has been designed in order to assess its efficiency in recovering phosphorous as struvite. The system consists of an equalizer, reactor, settler tank, dryer and packing unit and it was not seeded with previous struvite. A special cost effective concrete tank model coated with "Epoxy" inner surface was suggested. The overall tank volume of cylindrical body is (556.65) m^3. The reactor is fed with industrial fertilizer effluents rich with phosphorous and ammonia. Natural Bittern (MgCl.6H$_2$O) was added as source of low cost magnesium. A mass and Energy balance of belt conveyor dryer is studied using fan with electrical energy 15 W and drying rate 67 Kg/ day. A model was Formulated and studied for the mixed effluent to optimize the struvite precipitation using experimental design methodology where, phosphorous removal efficiency reached 86% - 92 %. This results were verified experimentally 84.74% for model verification. Crystalline struvite is characterized by x-rays diffraction (XRD), scan electron microscope (SEM) and (EDX) for degree purity. An economic study proves it is an eco-friendly process.

Keywords: Design, industrial wastewater, phosphorous, bittern, struvite.

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INTRODUCTION

Struvite precipitation is one ecologically sound technology which is not only cost effective but also sustainable in terms of possible recovery of recyclable constituents from industrial effluent rich in nutrients. Therefore, finding a cheap method to recover such materials from waste streams can provide a break through solution. One of the recent innovations is production and recovery of struvite ($Mg.NH_4.PO_4.6H_2O$) from the industrial effluents. If the formation of struvite is controlled, then it can be beneficiary since its precipitation removes NH_4^+-N and PO_4^{3-}-P from wastewater and the precipitate has the potential use as a fertilizer [1].

Great efforts have been done by researchers for the removal of nitrogen and phosphorous from wastewater before discharging it into water streams [2-4]. Recently, struvite has been produced from municipal and industrial wastewaters containing high concentration of nitrogen and phosphorous [5-8]. Generally, wastewaters contain less magnesium compared with nitrogen and phosphate. So, it is necessary to add a source of magnesium to optimize the struvite crystallization process [9]. Struvite is a threat for nutrient-rich wastewater systems when the nutrient composition exceeds stable saturation limit crystalline deposits of struvite characteristically form in the wastewater treatment infrastructure in high turbulence zone. The very sensitive zone of forming struvite is valves, bends in pipe, separating screens, pumps…etc. The mass of crystalline deposits formed can be extensive and can lead to operational failure by clogging water distribution pipes [10]. Struvite satisfies a need of slow-release fertilizer and so it could also be a promising opportunity for closing phosphate cycle and therefore being less dependent on global phosphate rock reserves which are running off [11].

The formation of struvite in aqueous solutions takes place following the development of supersaturation, the driving force to all crystallization processes. Supersaturation may be developed by increasing the aqueous medium content in ammonium, magnesium or orthophosphate and/or the pH. Although H^+ concentration does not directly enter the solubility product equation for struvite, struvite precipitation is highly pH dependent. The temperature and the presence of other ions in the solution such as calcium (Ca^{2+}) can also affect the struvite crystallization which was previously studied by our team [12].

Battistoni et al. principal for struvite nucleation and growth as well as the efficiency of a variety of crystallization reactors have been widely investigated and are well documented in the literature [13]. The fluidized bed and air-agitated reactors (FBR) are the most commonly used process for crystallizing struvite from industrial wastewater. These reactors normally consist of a vertical column upon which struvite precipitates inside. The process usually makes use of chemical amendment to reach a desired Mg: N: P molar ratio and pH can be adjusted through NaOH addition. After these adjustments struvite particles can precipitate spontaneously and growth occur due to interaction of particles [agglomeration]. The growth of these reactors is such that the flow velocity will decrease upwards, allowing treated effluent to flow out the top of the reactor, while the growing particles remain in the lower section. The particles are recovered from the reactor when they reach an acceptable size for resume, usually between 0.5 – 3 mm, which can take several days or weeks [14]. Therefore the reactor operates continuously for the liquid fraction, but in batches for the solids fraction [15]. Since particle size is a key factor in terms of recovery and reuse of struvite as fertilizer, seed materials can be used to provide nuclie on which struvite can attach to produce larger particles [16]. The initial particles bed is usually either sand, or pellets of the materials to be precipitated. Accordingly to Doyle and Parsons [17], there are a number of reasons for using products as seed. Using products as seed may influence the process kinetics, since crystal growth upon like materials requires less energy than growth on foreign particles. One method for limiting this problem is to recycle the fines a new seed material for the reactor [13].

This paper describes the detailed process flow diagram for process mass and energy balance, for mixed effluents of industrial Chemical fertilizer wastewater.

MATERIALS AND METHODS

The following block flow sheet represents the main industrial wastewater effluents of a Chemical - Fertilizer industrial plant located in Suez city near red sea road, about one hundred meters from the sea shore.

Power plant 500 m3 /day	Ammonium Sulphate plant 350m3 /day	Gas and Ammonia plant 2100 m3 /day	Potable water plant 750 m^3 /day	Treated wastewater Lagoon

3700 m^3 /day | 1

From west to East → | North effluents

Nitric Acid plant (old) 340m^3 /day	Nitric Acid plant(new) 720m^3 /day	Desalination Treatment plant 950 m^3 /day	Potable water treatment plant 340 m^3 /day

a **3700** m^3 | 2

South effluents

Sulphuric Acid Plant 480 m^3 /day

Ammonium nitrate plant 960 m^3 /day

c

Figure 1: Layout for plant area for chemical- fertilizer area in Suez City.

As it was clear in Figure (1) that the industrial area mainly consists of many plants gas and ammonia, Ammonium sulfate, Nitric acid, Sulfuric acid, Ammonium Nitrate, and Nitric acid plant and so mixed streams from all plants of 3700 m^3/day of collective nutrients to be treated separately or after mixing industrial wastewater effluents from different factories. Characteristics of the raw industrial wastewater streams from mixed streams are shown in Table (1).

Table 1: Characteristics of raw industrial wastewater

Major characteristics	Concentration in mg/L		
	a	b	c
NH4+-N	0.226	0.099	0.085
Phosphorous	16.92	53.84	48.47
Magnesium	18.56	4.98	5.57
Calcium	21.98	16.75	14.74
Sulphate	40.00	200	650
TDS	9860	800	2960
COD	8.5	625	280
Conductivity (mS/cm)	19.75	1.60	5.91
pH value	10.04	7.68	3.44
Cu	ND	ND	0.04
Pb	ND	ND	0.021
Ni	ND	ND	0.019
Cd	ND	ND	ND
nitrite	0.079	0.022	0.656

a= mixed effluents, b= ammonium nitrate effluent, c=End pipe effluent

Effluents streams are subjected to full chemical analysis according to standard methods for examination of water and wastewater for ammonia, phosphorous, magnesium, hardness, calcium, conductivity, pH value, dissolved solids and others. Calculated quantities of precipitating agent can be added. Liquid Bittern (LB) as low cost source of magnesium was used and their characteristics are shown in Table (2).

Table 2: Characteristics of liquid bitten used as source of magnesium

Element	Value mg/L
TDS	292
Calcium	1600
Magnesium	73.84
Sodium	21.76
Chlorides	218.63
Sulfate	3.2
Carbonate	0.5
Potassium	9.81
Bicarbonate	1.73
Bromine	12
Boron	70
Iodine	5
Lithium	0.31
Conductivity	583mS/cm

In this study, struvite was recovered from waste effluents of chemical fertilizer industry. The stoichiometric molar ratio of $Mg : NH_4^+ - N : PO_3^{2-}$ was 1: 1: 1which was sufficient for ammonium removal by 80% and produced yield > 1g/L in struvite precipitation. The crystal phase of struvite was obtained by XRD and morphology analyzed by SEM. The crystal morphology indicated high purity in all single streams (ammonium nitrate, ammonium sulphate and nitric acid stream). The recovered crystalline struvite from mixed stream containing calcium ions where Ca/Mg > 1 decreased struvite purity. To overcome inhibition of struvite precipitation from mixed effluents we precipitated calcium with oxalate (oxalic acid – ammonium oxalate) to keep Ca/P <0.5 and to liberate more phosphorous in the solution. The optimum pH for phosphorous precipitation as struvite was 10.3. The slight increase in pH changes the speciation of the struvite constituents leading to more or less favorable conditions for the struvite crystallization/dissolution.

A model was studied for the mixed effluent for optimization of struvite precipitation using experimental design methodology. A 15 liter volume of mixed industrial effluent was treated with 30 m mol oxalic acid – ammonium oxalate to chelate and capture calcium from solution and to free almost all phosphorous present (260 mg/L), then de-calcinate effluent was analyzed again to know starting concentration for struvite. Thus experimental protocol was as follows:-

1. A predetermined volume of de-calcinated bittern was added to mixed effluent to adjust required molar ratio of Mg: NH: P.
2. Adjust effluent pH (10 -11) using 5N NaOH solution.
3. Allow the effluent reaction time 20 -60 minutes
4. Allow struvite crystallization for 2 hour
5. Filter the solid for Phosphorous ions
6. The solid precipitated (struvite) was subjected to XRD and SEM to characterize its composition and morphology.
7. The XRD shows the presence of schetilite with struvite and others a phosphate salt which indicates the interference of foreign ions on phosphate precipitated as pure crystalline struvite.
8. The experimental Matrix and Model was taken as purpose to optimize struvite precipitation using BOX BENNKHEN design.
9. Three dimensional surface response plots were generated using the fitted model by varying two variables within the experimental range and holding the other constant at the central point.
10. The significant factors are: temperature (b_1) , Reaction time (b_2), pH (b_3), three quadric terms (b_{11}, b_{22}, b_{33}) and two interaction terms (b_{13} and b_{23}) to the response % phosphate Ind (Y_1).
11. The resulted models were given by the following equations:

$$Y_1 = b_o = (b_1X_1 + b_2 X_2 + b_3 X_3) + (b_{13}X_1X_3 + b_{23}X_2X_3) + (b_{11}X_1{}^2 + b_{22} X_2{}^2 + b_{33}X_3{}^2)$$

$$Y_1 \text{ (\% PO}_4\text{Ind)} = 78.75 - 8.394 X_1 - 3.02 X_2 + 14.21 X_3 + 7.185 X_1 X_3 - 6.148 X_2 X_3 - 15.586 X_1{}^2 - 3.886 X_2{}^2 - 4.748 X_3{}^2$$

1. The application of RSM (Response surface methodology) yield the following regression equation which was an empirical relationship between % PO_4 Ind(Y_1) recovered from mixed industrial effluent stream.

2. Based on modeling, kinetic relations for struvite crystallization, which was detailed in our previous work on [18, 19] and completed in this study. The proposed design for struvite crystallization is built and so together the economic study was evaluated to judge process applicability.

PROCESS DESIGN

This work describes the detailed process flow diagram for struvite precipitation with reactor "special design" according to process "mass and energy balance". Prior to the main experiment based design parameters, preliminary experimental observations in batch, and pilot scale along with optimization of operating parameters for struvite precipitation were required. The experiments were carried out at ratio Mg: NH: P, 1: 1: 1 and constant pH =9.2. All experiments were conducted and configured at room temperature over 120 minute. A schematic diagram of the struvite fed. Batch system is shown in Figure (2).
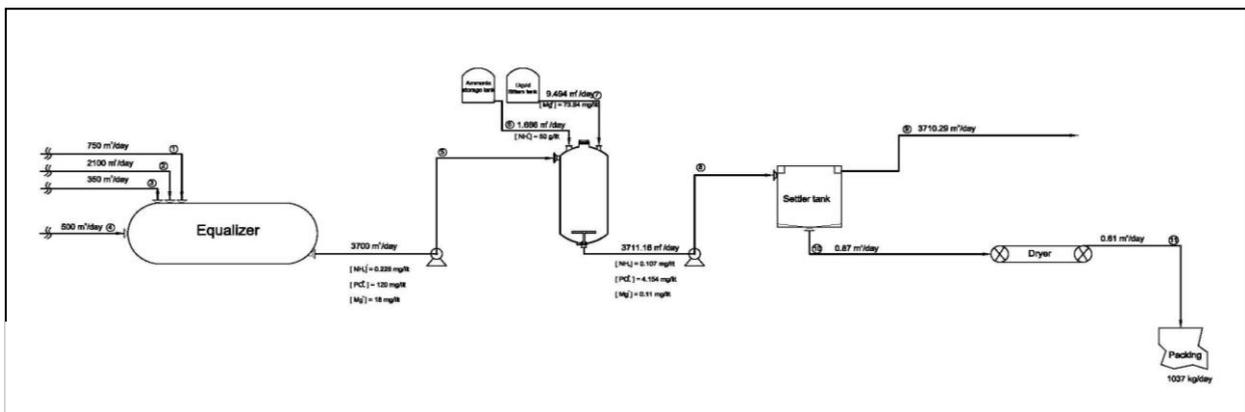
Figure 2: Schematic Process flow Diagram for struvite precipitation system from mixed industrial streams

An equalizer, reactor, settler tank, dryer and packing units were used in this design. A struvite reactor (stirring tank), was used as shown in Fig (2). A pH controller to control pH set point. Dosing pumps were operated for titrant (feed solution additions). The reactor was not seeded with pervious struvite (fine precipitate).For All Industrial Applied Flow sheets Struvite is being separated from wastewater through Gravity Settling. A clarifier for such a process is used in the shape shown in Fig (3).
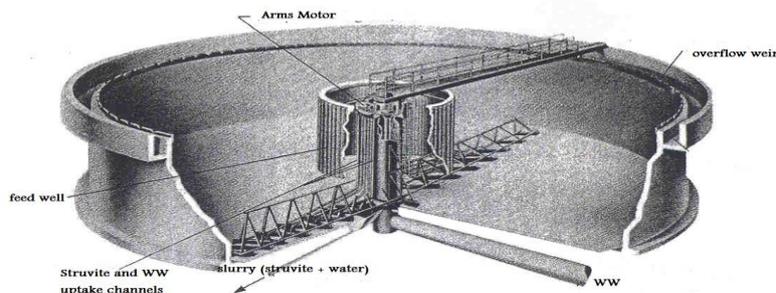
Figure 3: Clarifier for Struvite Production
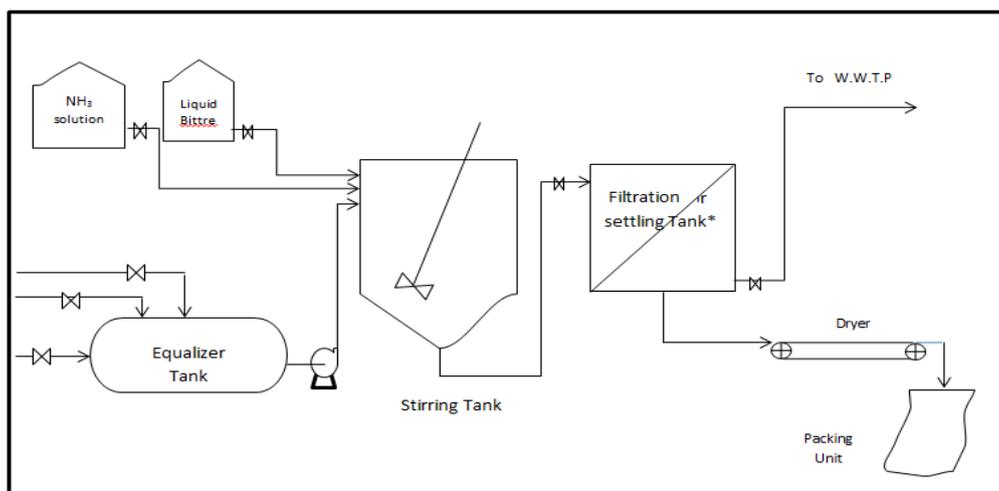
Figure 4: Proposed schematic Diagram for struvite batch system

Design of Experiment:

The fed batch system was maintained using two effluents (mixed industrial effluents). Ammonium nitrate effluents and Ammonium sulphate effluents (liquid bittern) were added as source of Mg. $NH_4 Cl_2$ and NaOH. The main purpose of additives is to maintain a constant molar ratio 1: 1: 1 for Mg: NH_4: P ions relative to phosphorous concentration which is the highest in streams. NaOH 1 N solution used for adjusting pH up to 9.2. A set of fed batch experiments was conducted for struvite precipitation and left 2hr for particles size growing and crystallization.

Sampling and analysis:

At the end of the test the solution is withdrawn from the reactor and filtered through 0.2 μm filters. Supernatants recovered are tested for soluble phosphate ($PO_4 - P$) and ammonium ($NH_4 - N$) by photometric methods adapted from standard methods APHA, 1998 [17]. While the recovered struvite particles are dried at room temperature and weight to assess the remaining quantity of struvite in suspension and preserved in plastic containers to prevent any interference of adsorbed phosphorous into glass. Crystalline struvite was characterized by scan electron microscope (SEM), EDX and X-rays diffraction (XRD), Fig(5), Fig(6), and Fig(7) respectively.
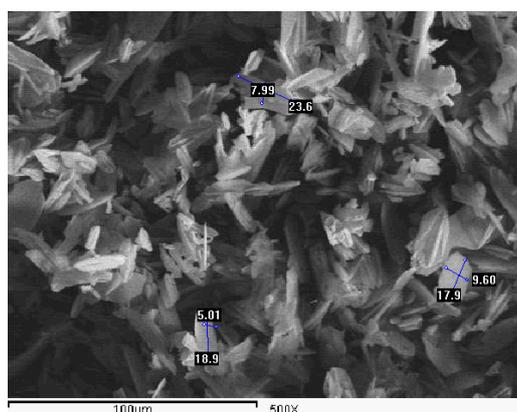
Figure 5: Crystalline struvite precipitated from industrial effluents
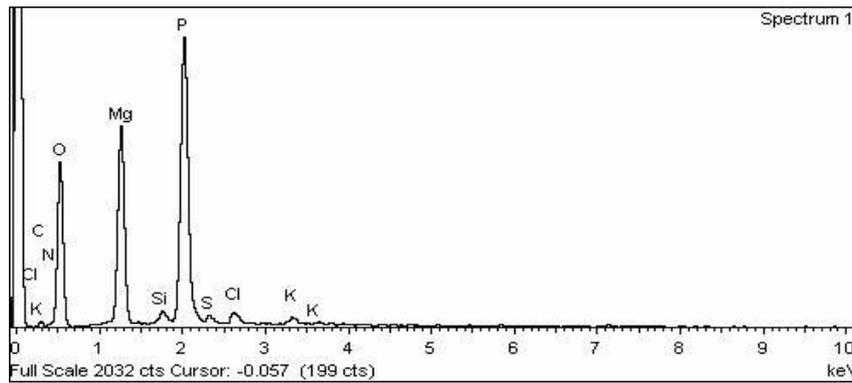
Figure 6: The percent weight of struvite crystals contents
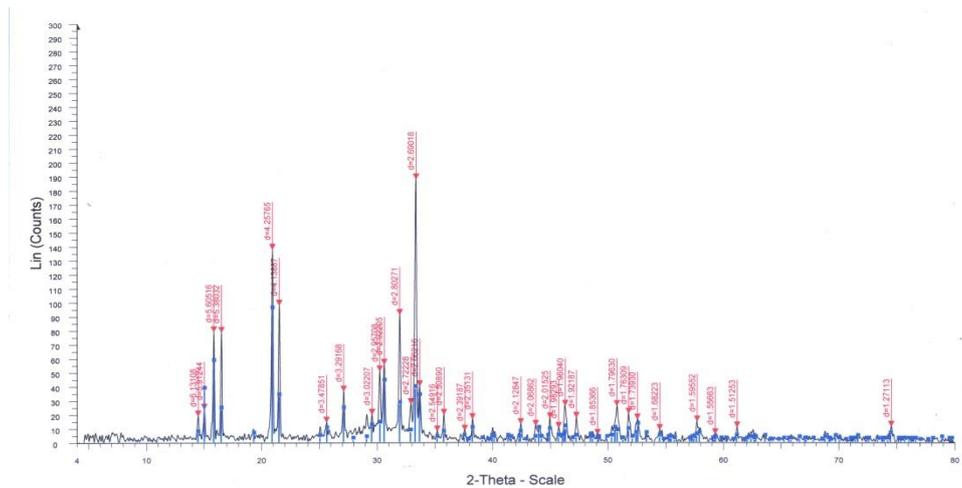
Figure 7: The XRD pattern of crystalline struvite precipitated from industrial effluents

Mechanical Design Sequence:

Material of construction

Looking at the flow sheet and the process of the struvite production, it was noticed that the settler tank is the larger tank in the desired settling. When applying calculation as shown below in the design, we shall have a tank volume of 556.65 meter cube tank which would be a great cost if designed from carbon steel or stainless steel. Sowe suggested a design of concrete tank coated by a suitable substance that would make no interaction between wastewater and tank surface. For such a model we suggested "Epoxy" as coating material to the tank inner surface.

Mechanical parts Design

       Concrete body and walls
       Overall daily rate $3711 \text{ m}^3/\text{day}$
       Complete settling time   3 hours
       Volume of tank 463.875 m^3
       Safety factor   1.2
       Total volume 556.65 m^3

So we needed an overall tank volume= $(3711/24)*3*1.2 = 556.65 \text{ m}^3$

The body is divided into two parts (cylindrical body – conical bottom and the concrete wall as shown in figure 8):

Cylindrical Body:

Taken volume of cylindrical body = 556.65 − 1 = 555.65 m^3

By optimizing the dimensions according to the most appropriate settling height with respected to wall resistance

Dimensions of cylinder   H = 11.23 m
                                D = 5.61   m

Conical Bottom:

volume of settled slurry	0.764	m3
volume of the conical bottom (take)	1	m3
dimensions of conical bottom		
H= 20 cm		
d =5.6 m		

Concrete Walls:

Concrete walls thickness are taken from Tank Design Indexes as (t = .6 m)

Figure 8: Concrete body dimensions

Inner Body and Mechanism:

Figure 9: Showing the Facilities of stair, motor, tube, squeezes, wastewater out let and struvite outlet

Figure 10: CSTR Mechanical design for precipitation of struvite from mixed industrial wastewater

Stairs, Truss and Handrail, are made to facilitate the operation of the Moving Part that collect the precipitated Struvite and To be able to apply any control for the process. **Motor,** used to move the stainless steel arm that has squeegees to move the precipitation to the outlet hole.**Central vertical Tube,** are made with a hole in a level that almost higher than the precipitation level to make the tank empty through it. **Squeegees,**are responsible for moving the precipitation to the outlet hole. **Wastewater outlet,** where the outlet wastewater get out, and it is made through the concrete. **Struvite outlet,** where Struvite is collected and it is also go through the concrete. Illustrated in Figures (9) and then a special design parameters were presented in Figure (10).

Mass Balance of Belt Conveyor Dryer

The belt conveyer dryer was selected as the most applicable dryer for struvite crystals nature as it needs no vacuum or specialty precautions in their drying mechanism.

$m_a(W_4 - W_3) = m_p(w_1 - w_2)$

$(1/\rho_{avg}) = (x_w/\rho_w) + (x_p/\rho_p)$
$x_w = 0.2$, $\rho_w = 1000$ kg/m^3 , $\rho_p = 1700$ kg/m^3
$\rho_{avg} = 1492$ kg/m^3
$m_p = 0.426$ m^3/day * 1492 = 635.592 kg/day

T of drying = 100 ^{o}C, T of input = 25 ^{o}C, T of output = 42 ^{o}C (from psychometric chart at 80% humidity)
$W_4 = 0.046$ kg water/kg dry air, $W_4 = 0.02$ kg water/kg dry air(from psychometric chart at 80% humidity)
Given: 20% water content by volume
$w_1 = 0.2$* density of water/density of Struvite = 0.2 *(1000/1711) = 0.117 kg water/kg dry product

Energy Balance:

$m_aH_{a3} + m_pH_{p1} = m_aH_{a4} + m_pH_{p2} + q$
Assume no heat loss (q = 0)
$H_{a3} = 192$ kJ/kg , $H_{a4} = 80$ kJ/kg (from psychometric chart at 80% humidity)
$H_p = C_{pp}(T_p - T_o) + wC_{pw}(T_p - T_o)$
$C_{pp} = 0.72$ kJ/kg.^{o}C
$T_o = 0^o$C
$H_{p1} = 30.2265$ kJ/kg , $H_{p2} = 30.24 + 175.56w_2$
The result are not reasonable therefore take $w_2 = 0.1*w_1 = 0.0117$ kg water/kg dry product
$m_a = 2574.1476$ kg/day
Thermal Energy and Heat Transfer Area:
$H_o = 334$ kJ/kgΔ
$X_o = w_1$, $X = w_2$, $Y_o = w_3$
$F = m_p$, $F_a = m_a$
$C_{PL} = 4.18$ kJ/kg.^{o}C
$C_{PV} = 1.864$ kJ/kg.^{o}C
$C_{PS} = 0.72$ kJ/kg.^{o}C
$C_{PA} = 1.005$ kJ/kg.^{o}C
$Q_{we} = 6853.41057$ kJ/day
$Q_{sh} = 57635.16476$ kJ/day
$Q_{ah} = 201223.692$ kJ/day
$Q = 265712.2673$ kJ/day
$U_s = 100$ W/m^2.^{o}C
$A_s = 0.5$ m^2

ECONOMIC PRE-FEASIBILITY STUDY FOR STRUVITE PRECIPITATION

Pricing is done Based on the nitrogen and phosphorus content in the MAP, a sales price of $234/t is possible. This would be the price if the MAP was to be used in broad-scale agriculture.

However, MAP is a unique product that we may call it a boutique fertilizer that's used for specific applications then we sell the value with the product. So, a much higher sales price can be achieved.
In Japan for example, a sales price of $3800/t has been reported.

So in all our calculations we would set our budget and plans for a $2000/t as a sales price. It's not a separate factory but it's only a WW treatment unit in a plant. - All prices and Numbers in USD.

Table 4: Total Capital Investment.

Total Capital Investment		Price
Equipments	Equalizer (77 m3) St.St	65000 $
	NH$_4$CL tank (1.99 m3) St.St	11800$
	liq bat. Tank (22.8 m3) St.St	40000$
	Reactor S.S	17000 $
	settler (556.6 m3) concrete	22000 $
Total cost of equipment	Belt –conveyor dryer	16200 $
land cost	E	172000 $
Installation	Free	
Piping	0.4 E	68800 $
Instrumentation	0.7 E	120400 $
Electrical	0.2 E	34400 $
Buildings	0.1 E	17200 $
Storages	0.15 E	25800 $
Utilities	0.15 E	25800 $
Site development	0.5 E	86000 $
Auxiliary Buildings	0.1 E	17200 $
PPC	0.15 E	25800
Design and Engineering	2.45 E	421400 $
Contractor's fee		
Contingency		
IPC	0.3 PPC	126420$
	0.05 PPC	21070 $
FCI	0.1 PPC	42140 $
	0.45 PPC	189630 $
WCI	PPC+ IPC	611030 $
TCI	0.15 FCI	91654.5 $
	TCI	**702684.5 $**

Table 5: Monthly Variable Costs

	Variable costs per month	
		Price ($)
Raw Material	NH4CL	12000
	Water bat	1000
transportation	5%	
working force	8 workers/shift	
1 $ per hour	3 shifts	
	8 hrs/shift	
	7 days week	5760
energy	N/A assume 500	800
total costs/ month		19560
other + 10%		**21516 $**

Table 6: Expected Revenue Table

Production rate	1.037	ton/day
Sales price	2000	$
Monthly revenue	62220	USD/month
Annual Revenue	684420	USD/year

Table 7: Payback Period and ROI -First year profits

first year calculations			
month 1	variable costs	monthly	monthly gross profit ($)
month 2	21516	62220	40704
month 3	21516	62220	40704
month 4	21516	62220	40704
month 5	21516	62220	40704
month 6	21516	62220	40704
month 7	21516	62220	40704
month 9	21516	62220	40704
month 10	21516	62220	40704
month 11	21516	62220	40704

Table 8: First year net profit

first year profits	407040
Taxes 10%	40704
net profit	366336

Table 9: NCF & ROI

	year 1	year 2	Year3	Year 4	Year 5	Year 6
net profit	366336	366336	366336	366336	366336	366336
all annual extra costs + dep.	raise the sales price each year to make the annual profit fixed at this number					
NCF	-336348.5	29987.5	396323.5	762659.5	1128995.5	1495331.5
ROI each year %	-47.86621877	4.267562469	56.4013437	108.5351249	160.6689062	212.8026874
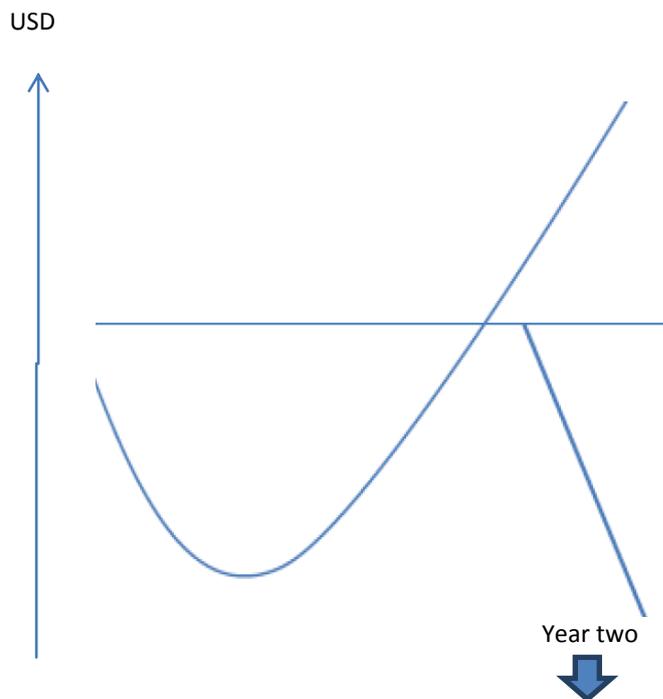
Figure 11: Payback period chart.

So, as shown in the pre table that it's not only eco-friendly process but also it has a good profit that could reach 200% on original investment if it was a 6 years investment.

CONCLUSION

- The present study investigates removal and recovery of nutrients by struvite formation from industrial effluents of a chemical fertilizer industry.
- 2-Both ammonium and phosphorous removal were achieved by adding necessary amounts of Mg as (Liquid Bittern) to struvite precipitation. Removal efficiency reached 86% - 92 % . The presence of calcium affected struvite and crystalinity.
- 3-The for Factors BOX Behnken design was employed in order to model and optimize % PO4 removals struvite. Two valid model were established, to which precipitation of phosphate salts and struvite of 25.23 °C and pH of industrial effluents of 10.89 for a time of 34.76 minutes at starting molar ratio between Mg : PO4 2.25 giving PO4 recovered which was verified experimentally 84.74% for model verification.
- 4-In the absence of calcium in effluent stream, the SEM analysis, XRD analysis and chemical composition results confirmed that the recovery product was almost pure struvite.
- 5-In the current work the performance of a concrete settler for struvite crystallization in industrial effluents has been investigated; tests demonstrated that the reactor was able to accumulate struvite under optimum conditions of precipitation and to achieve excellent PO4removalup to 86%.
- 6-These results are of major importance for the design of struvite precipitation reactor and for development of crystal growth control methodology.
- 7- An economic study for the process proved it is an only eco-friendly process.

ACKNOWLEDGEMENT

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