



## Human urine as alternative natural slow release fertilizer

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### ABSTRACT

Organically agricultural products are gaining popularities through out of the world as it gives the consumer satisfaction with a basket of safer and better trusted foods. Besides this fact, organic crops contain fewer nitrates, pesticides residues and trace elements than conventional crops. One of the major constrains of organic forming is decrease of raw organic matters to prepare compost. The second and the most constrain of organic forming in Egypt is the high temperature most of the year. This causes decrease in using of organic matters only four months in the year. In Egypt, scientists are looking forward for different organic source which are plenty in nature and available at little – to – no cost. Human urine is one of them, and has been gaining popularities as a raw material for organic cultivation. However, the direct use of human urine as agricultural fertilizer is problematic and controversial with regard to hygiene, storage, and transport and spreading. By addition of small amount of MgO or Bittern to synthetic or natural human urine a crystalline structure of  $\{Mg(NH_4)(PO_4) \cdot 6H_2O\}$  which is known as struvite precipitates as major component. XRD- analysis and EMS- EDS analysis were used to determine fertilizer quality. The test for slow release Fertilizer and the Activity index are studied to assists struvite as slow release fertilizer which fits the Egyptian whether and soil.

**Key words:** urine, struvite, fertilizer, Bittern, Nutrients Recovery.

### INTRODUCTION

The progress of organic agriculture in the world has been very slow due to rapid declination of organic raw materials such as animal waste, crops residues and green manure which is due to burning of waste and residues and also due to utilization of straw and grass as animal feed [1] and [2].

Human urine is a liquid waste product of the human body secreted by the kidneys by a process of filtration from blood called urination and excreted through the urethra. As urine is produced after filtration of the blood in the kidneys, it contains low-molecular-weight compounds as proteins which are not filtered. Each individual produces from 1-1.5 L of urine per day in 4-5 times and adult person excretes on average of 500 L urine per year [3]. Urine contains the major part of the daily excretion of nitrogen (N), phosphorous (P) and potassium (K) contributing 88%, 67%, 73% respectively [4]. In human urine about 75-90 % of N excreted are urea and the remainder being in the form of creatinine, amino acids and uric acid. When urine is used as fertilizer, most of the nitrogen fractions in it are taken up by plant and which is the same as that of the urea or ammonium fertilization with nitrogen efficiency approximately 90% of that of mineral fertilizer [3] and [5]. Moreover, it comprises of trace elements B, Cu, Mo, Fe, Co and Mn. Although human urine is not totally sterile, as it contains less amount of different entire microorganisms and that can be easily disinfected by storage [6].

Basically struvite is an orthophosphate, containing magnesium, ammonium, and phosphate in equal molar concentrations. The general formula of minerals of the struvite group is  $AMPO_4 \cdot 6H_2O$ , where \*A\* corresponds to potassium (K) or ammonium ion ( $NH_4^+$ ) and \*M\* corresponds to cobalt (CO), magnesium (MS), or nickel (Ni) [7].

Struvite in the form of a magnesium ammonium phosphate hex hydrate ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) crystallizes in an orthorhombic structure (i.e., straight prisms with a rectangular base) when human urine is treated with magnesium ion ( $\text{Mg}^{2+}$ ) and then can be used as an important slow release phosphate fertilizer [8] and [9] and [10] and [11].

Struvite precipitation reaction in an equimolar (1:1:1) follows the general equation:



In this process about 88% of the precipitate present is struvite, besides struvite, other minerals, such as epsomite ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ), brucite  $\{\text{Mg}(\text{OH})_2\}$  and ontgomeryite ( $\text{Ca}_4\text{MgAl}_4(\text{PO}_4)(\text{OH})_4 \cdot 12\text{H}_2\text{O}$ ), may also be formed depending on the amounts of other divalent or trivalent metal cations available in urine. Even through, human urine is a valuable source of different nutrients that has been used since ancient times to enhance the growth of plants, notably leafy vegetables and is universally available at little –to– no cost [3].

Several studies have been carried out to assess potential methods of phosphorus recovery as struvite at a bench and pilot scale, and few processes, integrated in treatment plants already exist and are effective in Japan[12], Netherlands [13] and Italy [14]. Very recently study has showed that human urine has a high content of readily available in nitrogen, phosphorus and potassium and its fertilizing effect is reported to be similar to that of nitrogen rich chemical fertilizer.

Nitrogen is not a depletable raw material, but it is present in urine in the form of ammonia or urea, which is a valuable material for use as a fertilizer. This would replace the chemical synthesis of these compounds, which requires energy and auxiliary materials. Therefore, nitrogen recovery from urine can result in a lower ecological burden in comparison to the use of chemically produced fertilizers [15] and [16].

The authors reported that for cabbage cultivation the fertilizer value of human urine is as good as mineral industrial fertilizer in terms of its chemical contents, and furthermore, its hygienic quality can also be guaranteed. They also concluded that urine-fertilized plants may grow more rapidly, so the plants can be harvested earlier, thus making more efficient use of the land. Besides this, the use of human urine fertilizer could reduce the demand for industrial fertilizer to some extent, which would reduce the environmental pollution released during fertilizer manufacture and transportation [17].

Human urine is generally free of pathogens (germs) when extracted by healthy person. Urine should be stored in a sealed vessel for six months before spreading. In addition the reuse of insufficiently treated human urine in agriculture may result in transmission of pathogens [18] showed that enteric microorganisms are may destroyed in a shorten time, all microorganisms have not been studied. The recent research reveals that it is possible to use human urine as an alternative source if multinutrient fertilizer, which could be achieved only by introducing new areas of scientific research in research frontier. When developing new treatment options in developing countries, it is important to consider, in addition to its ecological\ environmental sustainability, the financial and socio- economic sustainability criteria as well. The majority of wastewater treatment systems fail in developing countries because of the lack of financial resources for maintenance and operation. Because urine contains natural and recyclable resources which have a high ecological and economic value, it processing unit should be designed to be operated as a sustainable business, either by a private or by public organization [19]. Linking urban sanitation infrastructure and service provision to cities can draw sufficient financial resources for building infrastructure and securing operation and maintenance costs, as city planners might see the direct economic benefits. It is also opportunity to close the nutrient loop in urban excreta and wastewater management. Such linkage can be established with agriculture, which contributes an important share to urban food supply [20].

The aim of this study is to evaluate human urine for struvite deposit using synthetic urine solution and authentic fresh urine. Liquid bittern is used as source of low cost magnesium ions to recover nutrients such as phosphorous and ammonia in the form of slow release fertilizer.

## MATERIALS AND METHODS

### 2.1 Urine sources quantity and quality

Plastic cans were used to collect the urine from a dozen persons (6 mail & 6 – female). The collected urine was stored in 1.5 L plastic tank. This quantity was sufficient for one week experiments and analysis.

Synthetic human urine containing 11 solutes Table (1), in concentrations equivalent to average authentic urine of normal healthy men was prepared Table1 according to conventional urological methods [21] and [22] for use in the

experiments. Synthetic urine is free of pyrophosphates, organic macromolecules (matrix) and unspecified substances that could enhance or inhibit the nutrient recovery.

**Table (1) Composition of Synthetic Urine in g/L**

Material	concentration
Urea	25
Criatine	1.1
Soy Broth	10
KCl	1.6
KH <sub>2</sub> PO <sub>4</sub>	2.8
NH <sub>4</sub> Cl	1.0
NaSO <sub>4</sub>	2.3
NaCl	4.6
MgCl <sub>2</sub> .6H <sub>2</sub> O	0.651
CaO <sub>2</sub> .2H <sub>2</sub> O	0.651
pH	5.8

Reference: Griffith *et al.*, 1976 *a, b.*

### 2.2 Struvite precipitation experiments:

Struvite precipitation represents a possibility to produce a solid fertilizer from urine. Struvite (Mg NH<sub>4</sub> PO<sub>4</sub>.6H<sub>2</sub>O) is formed by addition of magnesium in the form of Mg Cl<sub>2</sub>.6H<sub>2</sub>O or (Bittern) the waste brine remaining after salt (NaCl) extraction from seawater, was obtained from EMISAL company-Egypt.

A series of batch investigations was conducted to study the influence of species concentration and pH on struvite precipitation. Experiments were carried out at 20°C in 1000 ml stirred vessel.

Stock solutions of synthetic urine were prepared as stated in section (2.1) Distilled water was used to prepare the synthetic urine, and pH was then adjusted at several pH values ( 8.5 – 10.5 ) by the gradual addition of 0.1N NaOH and kept constant all over the the reaction period of ( 30 min ).

The homogeneity of the solution was performed by a magnetic stirrer. Prior to any analyses done, all samples were allowed to settle over night in order to separate the crystallized precipitate from bulk mother liquid [23]. The formed precipitate was collected by filtration through medium filler paper (Whatman, Maid stone,UK) and dried at ambient temperature.

### 2.3 Analytical methods:

The pH of urine samples (synthetic and Authentic) was measured by pH meter (Hanna Instruments, HI 8314) and a probe (Hanna Instruments, HI 1230). Total dissolved solid (TDS) and conductivity were measured by (HACH\ conductivity meter 44 600-00).

Kjeldahl nitrogen, total phosphorus, ammonia nitrogen, Chemical oxygen demand were conducted by the procedure described in the Standard Methods using a portable HACH DR 2000 photo-spectrometer,[24]. Table (2) shows the properties of human urine under test.

Residual concentrations of ions in solution after filtration were analyzed by atomic absorption (AA). X- Ray diffraction (XRD) and Electron microscope- EDS analyses was used to determine the composition of the precipitates produced [25].

Test for slow release fertilizer and Activity Index were tested according to [26] and [27]. Methods used to test controlled –release fertilizers in Japan.

### 2.4 Methods used to test slow release fertilizers:

#### 2.4.1 Activity Index Test:

Ten grams of the fertilizer is put into a container to which 200 ml water is added. Stirring at different time intervals (15 min, 30 min, 60 min, and 120 min) were performed at room temperature. All liquid in container is transferred into a 250 ml volumetric flask, make up to volume is achieved and the solution is then analyzed. The solid content is air dried and weighed. 200 ml water was added to the solid content and stirred at 100 °C each sample at the same time intervals stated above. The solid content after air drying is weighed and (AI) is calculated as follows:

$$AI = \frac{CWIN - HWIN}{CWIN} * 100 \%$$

Where CWIN=cold water insoluble nitrogen

And HWIN = nitrogen insoluble at 100°C

Fertilizers with a higher AI% have increased N solubility, better slow –N release characteristics.

**Table (2) Properties of human Urine under Test:**

Materials	Concentration in synthetic urine	Concentration in Authentic urine
pH	6.48	5.38
conductivity	2.55 mS/cm	19.62 mS/cm
COD	69.2 mg/L	37216 mg/L
Total P	3.9634 g/L	3.769 g/L
NH <sub>4</sub>	57.12 mg/L	784 mg/L
TDS	1.27 g/L	9.8 mg/L
Temp.	25 °C	30 °C
Mg	15.57 mg/L	9.963 mg/L
Fe	0.114 mg/L	70 mg/L
Mn	0.038 mg/L	0.038 mg/L
B	ND	0.1 mg/l
Ca	H	H
Cu	0.006 mg/L	14 mg/L
Zn	0.016 mg/L	42 mg/L
MO	ND	ND
CO	ND	ND
Cr	ND	ND
Cd	ND	20 mg/L
SO <sub>4</sub>	1.55 g/L	0.0475 g/L
Pb	ND	34 mg/L
K	16.05 mg/L	2.48 mg/L

*ND = not detected*

## 2.5 Economic Study:

Preliminary Techno – Economic Evaluation For Struvite production from Human Urine.

## RESULTS AND DISUSSION

### 3.1 Effect of pH variation on struvite precipitation:

The results of the effect of pH on nutrient removal with struvite precipitation under conditions in which the molar ratio was 1:1:1 and pH values were 8.5, 9, 9.5, 10, and 10.5 are given in Table (3).

**Table (3) Changes in Mg ion concentration at different pH during struvite precipitation using Mg O or Bittern:**

pH Value	Concentration of Mg ion when adding MgO	Concentration of Mg ion when adding Bittern
8.5	0.7	1
9	1.4	1.1
9.5	0.8	1.2
10	1.2	1
10.5	1.1	1.1

*Original pH= 7.9*

The optimum pH value obtained in the present study was investigated within the range 9.5 – 10 in the batch test using MgO and Bittern respectively.

The best Mg<sup>2+</sup> removal efficiency (89.87%) at pH = 9.5 using MgO and 87.34% at pH =10 using Bittern rich with Mg<sup>2+</sup> ions.

It was found that removal efficiency decreased when pH was higher than 9.5 for using MgO and when pH was higher than 10 for using Bittern solution. This phenomenon due to dissolution of struvite when pH is above 9, NH<sub>4</sub><sup>+</sup> will be deprotonated into NH<sub>3</sub> and may result in NH<sub>3</sub> vaporization and inhibition of magnesium ammonium phosphate (MAP) crystallization process. Our results agreed with [28] and [29].

In order to remove struvite from urine by precipitation, the pH value of the solution should be in the range pH (8 - 9.5) where struvite solubility is minimal. But, with adding Bittern as source of Mg<sup>2+</sup> the efficiency of struvite increased from 84.81% up to 87.34% at pH value in solution was 9.5 and 10 respectively. This solution at higher pH=10 giving better struvite precipitation.

## 3.2 XRD, SEM and EDS Analysis:

3.2.1 From XRD pattern of the deposits (Fig1<sub>a</sub>), pure crystalline phase for struvite precipitated by synthetic urine solution and MgO addition. Nine peaks with the high values of intensity at 239, 6390, 533, 572, 382, 5270, 494, and 442, corresponding to 2 theta at 5.9322, 5.6062, 4.2686, 4.1494, 3.2955, 2.9269, 2.8037, 2.6921 and 2.6659 were detected.

The crystal size (L (n m) 79.6) is at position 21- 2theta. The SEM image of the crystals morphology is shown as orthorhombic (prismatic elongated) along the direction they formed Fig 1a.

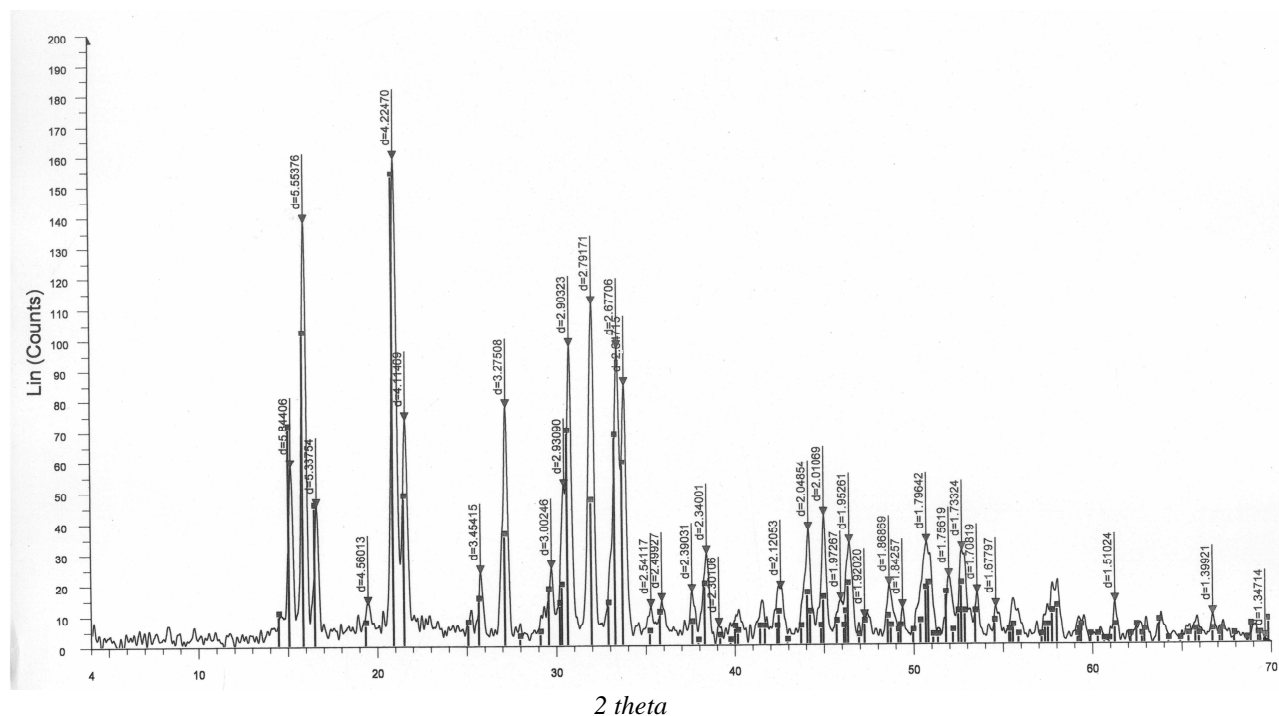


Fig (1) Characteristics of struvite powder precipitated using synthetic urine and MgO

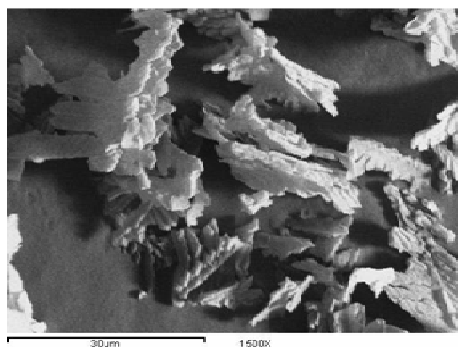
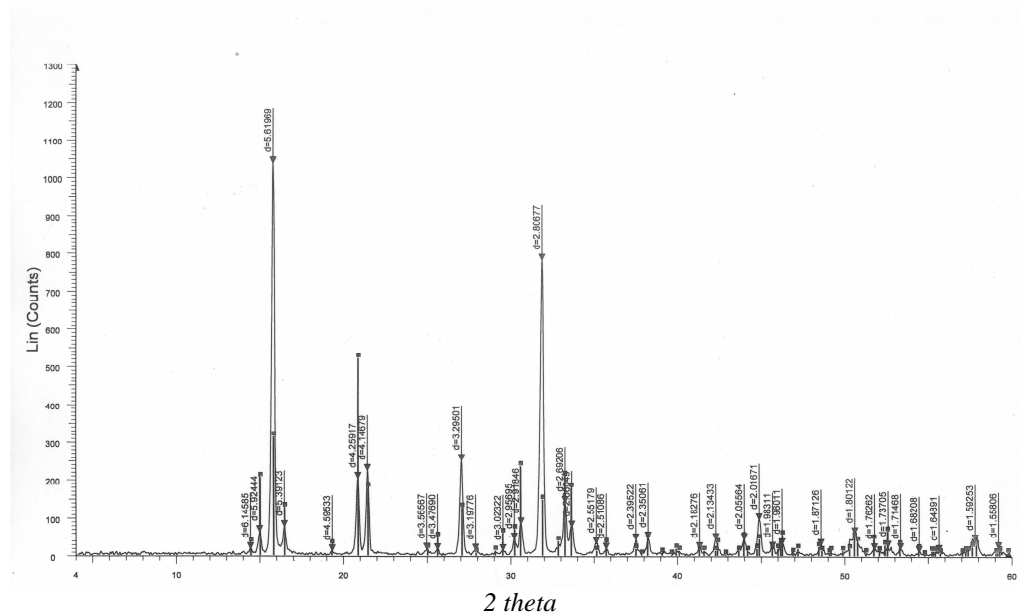


Fig (1a) The SEM image of the struvite crystals morphology formed by synthetic urine and MgO addition is shown as orthorhombic (prismatic elongated) along the direction they formed.

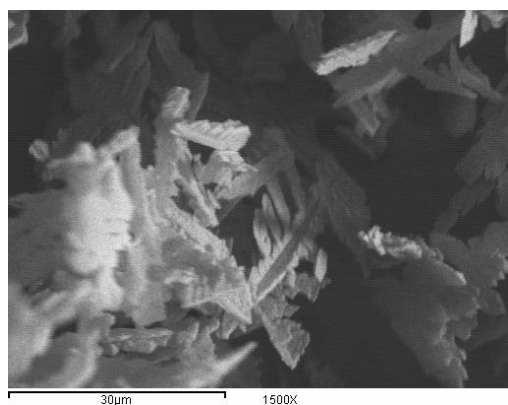
3.2.2 From XRD pattern of the deposits Fig (2), pure crystalline phase for struvite precipitated by synthetic urine solution and Liquid Bittern addition. Nine peaks with the high values of intensity at 23791.76, 1573.26, 175.65, 5116.42, 580.39, 284.89, 20168.22, 4608.43, and 2045.57, corresponding to 2 theta at 14.942, 15.757, 16.429, 20.839, 21.411, 24.952, 27.039, 31.858, and 33.254 were detected. The crystal size (L (n m) 75.5) is at position 31 - 2 theta. The SEM image of the crystals morphology is shown as orthorhombic (prismatic elongated) and branched type along the direction they formed Fig (2a).



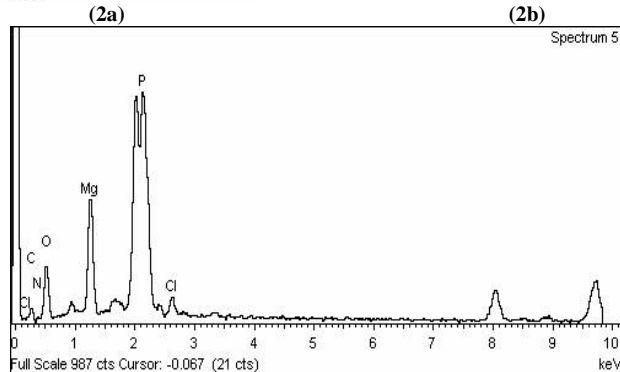
**Fig (2) Characteristics of struvite powder precipitated by synthetic urine and liquid Bittern, (2a)SEM struvite crystals morphology shown as prismatic elongated along the direction they formed, (2b)table of atomic %, and (2c)EDS fertilizer content.**

EDS analysis is shown in Table (2b) and Fig (2c) confirming that the recovered product is struvite and mainly high phosphorus content. Calculation based on composition of the precipitate indicated that total phosphorous 5.76%, magnesium 8.55%, nitrogen 13.67% , potassium 0.86% besides  $\text{CaCO}_3$  28.9% and  $\text{SiO}_2$  42.27% calculated by their atomic %.

From the stoichiometric content of Mg:  $\text{NH}_4\text{:PO}_4$  in struvite formed by weight is 12.74: 11.74: 10.93



Element	Weight%	Atomic%
C K	21.28	28.90
N K	11.74	13.67
O K	41.45	42.27
Mg K	12.74	8.55
P K	10.93	5.76
Cl K	1.86	0.86
Totals	100.00	



(2c)



3.2.3 Struvite precipitated by authentic human urine and liquid bittern is shown in XRD pattern (Fig 3). Nine peaks with densities at 5.8804, 5.5702, 5.3632, 4.2418, 4.1224, 3.2782, 2.9109, 2.7931 and 2.6831 corresponding to 2 theta at 15.054, 15.898, 16.516, 20.925, 21.539, 27.18, 30.689, 32.017 and 33.368. The SEM image (3a) of the dense crystal morphology is orthorhombic (prismatic elongated in direction of formation and EDS analysis (3c) confirms that recovered product is struvite with phosphorus 7.95%, magnesium 8.81%, nitrogen 9.9%, potassium 0.16%. Besides Ca 16.55% and SiO<sub>2</sub> 0.28% and O<sub>2</sub> 55.84% as shown in Table (3b) are formed and calculated by their atomic %.

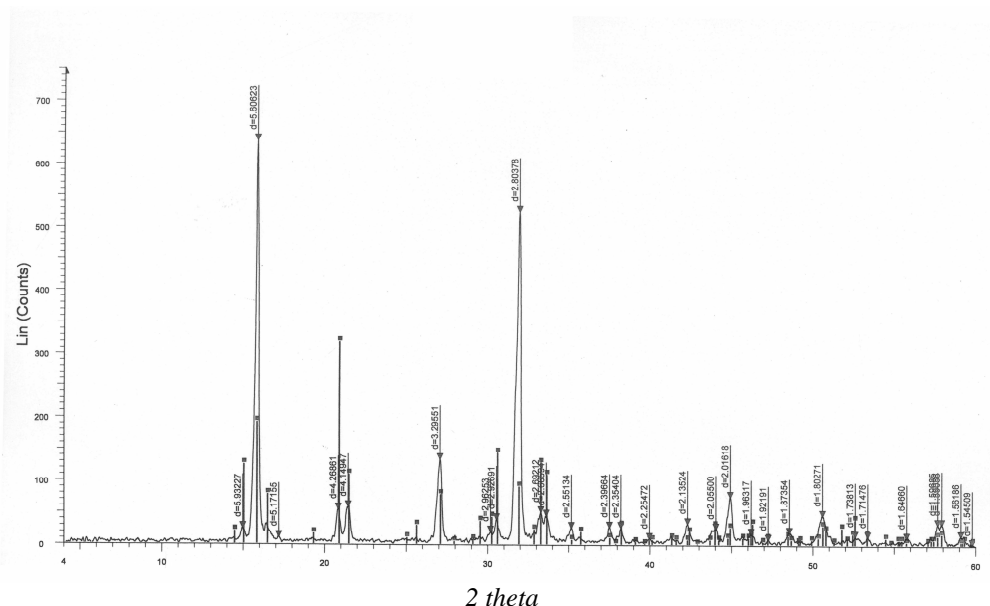
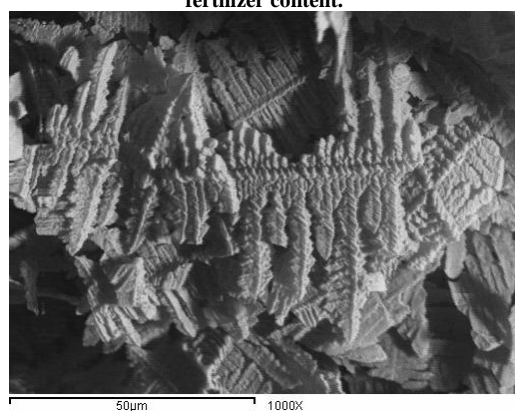


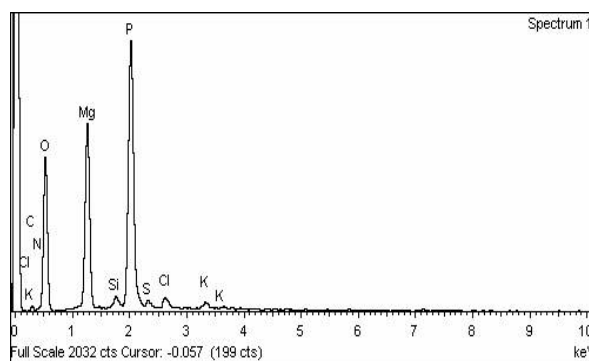
Fig (3) Characteristics of struvite powder precipitated by human urine and liquid Bittern, (3a)SEM struvite crystals morphology shown as prismatic elongated along the direction they formed, (3b)table of atomic %, and (3c)EDS fertilizer content.



element	Weight%	Atomic%
C K	11.54	16.55
N K	8.05	9.90
O K	51.85	55.84
Mg K	12.44	8.81
Si K	0.45	0.28
P K	14.30	7.95
S K	0.40	0.22
Cl K	0.61	0.29
K	0.37	0.16

(3a)

(3b)



(3c)

3.3 Methods used to test slow release fertilizers:

3.3.1 Activity Index Test:

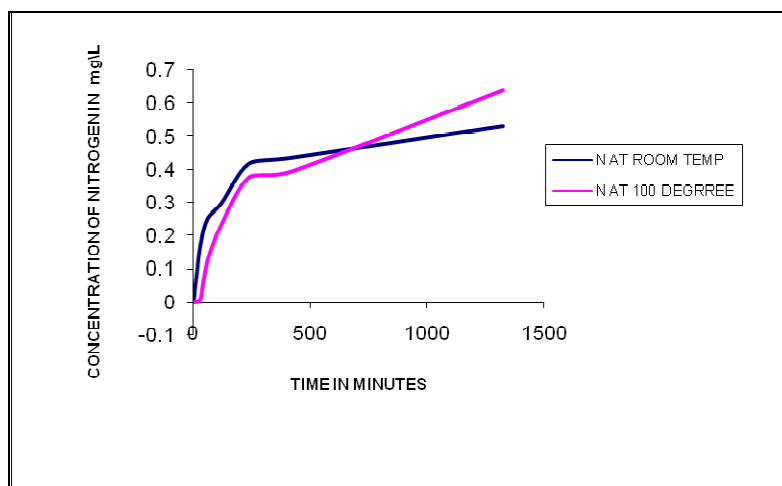
The solid content after air drying is weighed is calculated as follows:

$$AI = \frac{CWIN - HWIN}{CWIN} * 100 \%$$

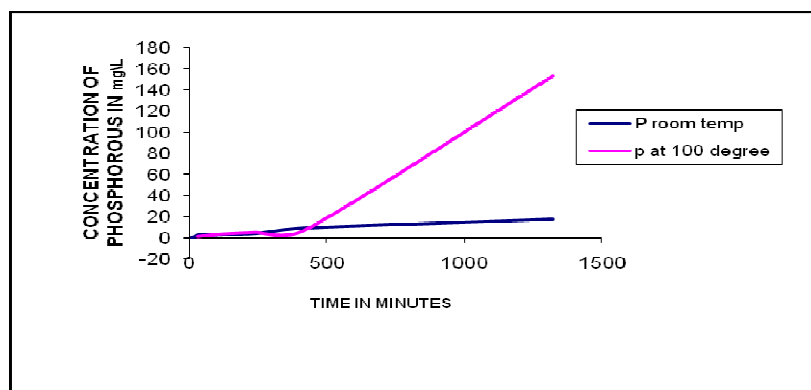
Where CWIN=cold water insoluble nitrogen

And HWIN = nitrogen insoluble at 100°C

The results in Fig (4) and Fig (5) and Fig (6) show the gradual release of nutrients (N, P, K) at ambient temperature.

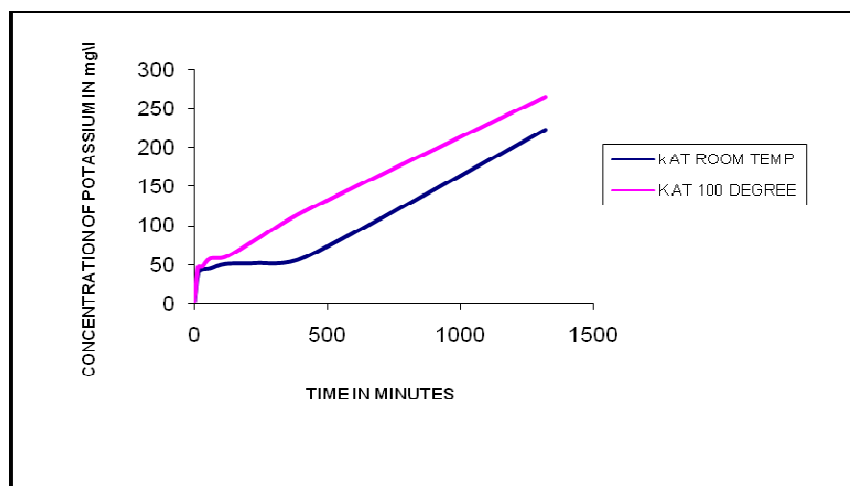


Fig(4) EFFECT OF NITROGEN RELEASE WITH TIME AND TEMPERATURE VARIATION

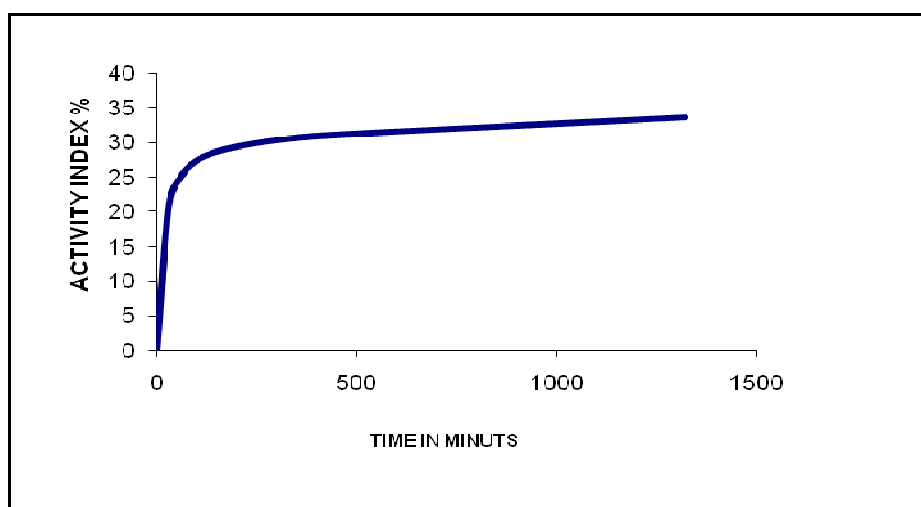


Fig(5) EFFECT OF PHOSPHOROUS RELEASE WITH TIME AND TEMPERATURE VARIATION





Fig(6) EFFECT OF POTASSIUM RELEASE WITH TIME AND TEMPERATURE VARIATION



Fig(7) EFFECT OF ACTIVITY INDEX % WITH TIME AND TEMPERATURE VARIATION

The significant release of nutrients at 100 °C is shown in Fig (4) and Fig (5) and Fig (6). Results shown in Fig (7) indicated struvite as good slow release fertilizer with an AI % > 33% after 22 hours only.

## 2.5 Preliminary Techno – Economic Evaluation for Struvite production from Human Urine

### 2.5.1 Cost Indicator

Simulation and basic engineering of struvite production for proposed capacity 10.8tons/year (36Kg/day or 12Kg/shift) has been concluded. The simulation includes the following: reaction, settling, filtration, drying, granulation and packing.

The process flow-sheets are depicted in Figures (8) and Fig (9) for pilot and industrial scales.

### 2.5.2. Total capital investment costs

Equipment sizes based on the mass production and cost estimation of the process equipment has been estimated from [30] and prices have been adjusted based on cost indices 2012 as presented in Table (4)

### 2.5.3. Operating Cost

In addition to raw materials cost as estimated according to actual consumption from pilot experimental results and prevailing costs, other components of operating costs are presented in Table (4, 5, 6&7).

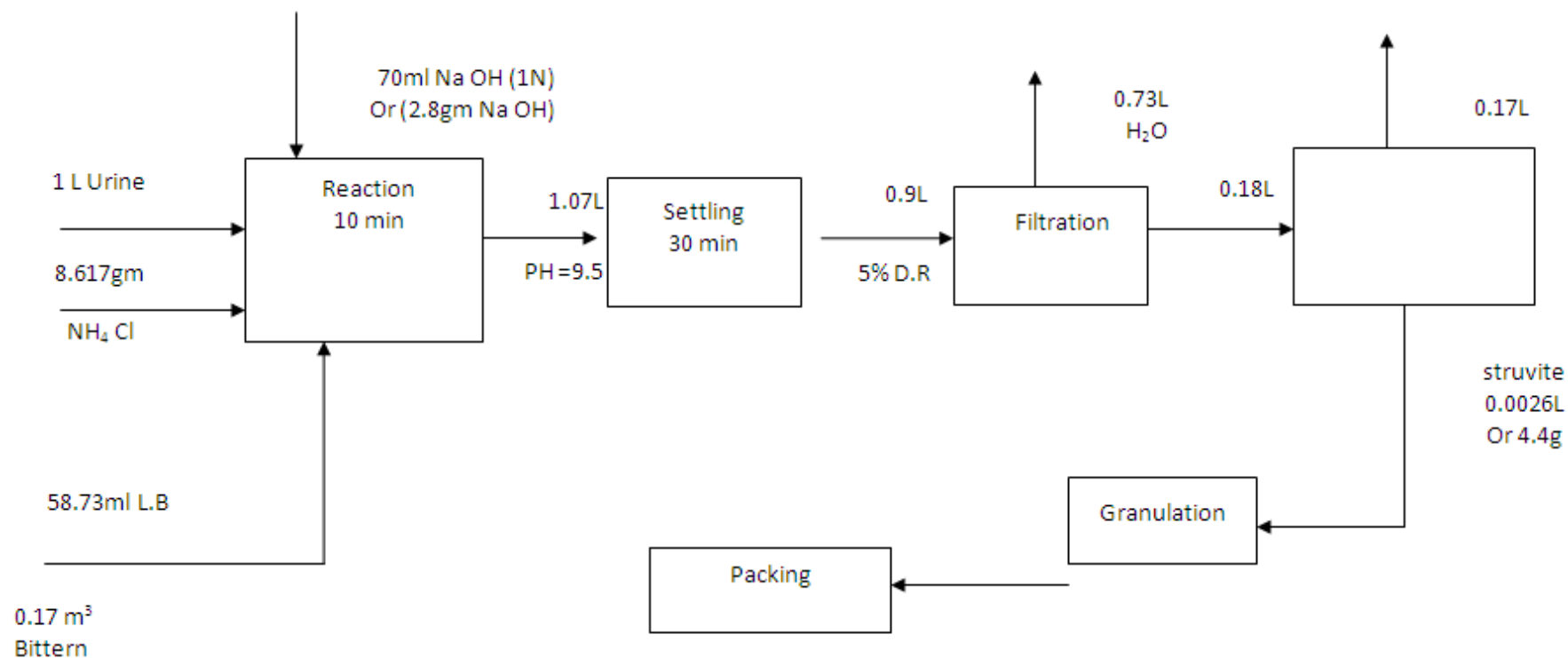


Fig (8) Block Flow Sheet for Struvite Production at Laboratory Scale (4.4g/shift)

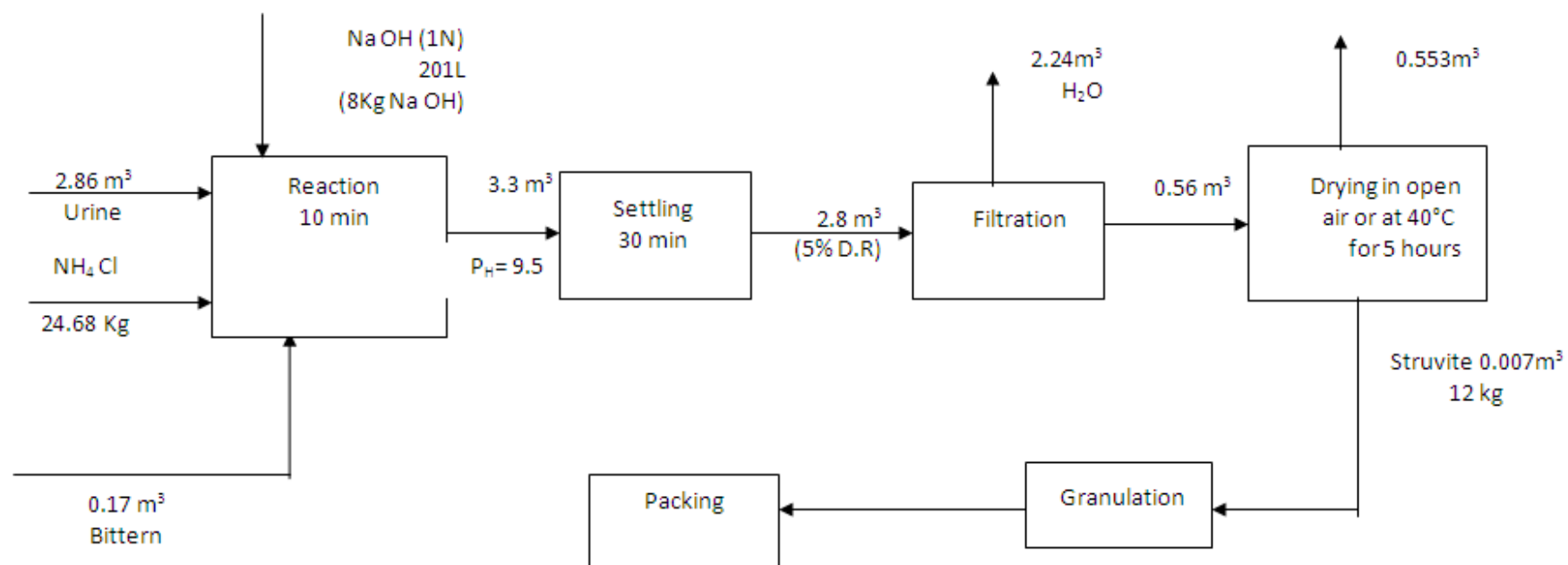


Fig (9) Block Flow Sheet for Struvite Production at Pilot Scale (12Kg/shift)

Table (4) Total Capital Investment (TCI)

Item	No Unit	Cost L.E
<b>A. Direct Cost (DC)</b>		
Purchased Equipment (PE <sup>*</sup> )	1	75000
Urine storage tank concrete lined 5m <sup>3</sup>		
Brine storage tank concrete lined 5m <sup>3</sup>	1	7500
Reactor,	1	15000
Reinforced concrete, Lined & 5m <sup>3</sup> capacity		
Settler,	1	8500
Reinforced concrete, Lined provided with bottom skimmer & 5m <sup>3</sup> capacity		
Filer press (2.5m <sup>3</sup> )	1	30000
Drying beds (0.5m <sup>3</sup> )	2	25000
Packing Unit (12 kg/hr)	1	3000
Granulator	1	10000
<b>Total purchased Equipment</b>		<b>106500</b>
Purchased equipments installation (35% of P.E)		37275
Instrumentation control (10% of P.E)		10650
Piping (15% of P.E)		15975
Electrical (10% of P.E)		10650
Buildings (15% of P.E)		15975
Yard Improvement (4% of P.E)		4260
Land & Service Facilities (10% of P.E)		10650
<b>Total Direct Cost</b>		<b>105435</b>
<b>B. Indirect Cost – (IC) (15% of FCI)</b>		
Engineering & Supervisor (5% DC)		10597
Contractor fee 's (2% of DC)		4239
Contingency (6% of FCI)		14475
<b>Total Indirect Cost (IC)</b>		<b>29311</b>
<b>C. Fixed Capital Investment (FCI)</b>		<b>241246</b>
FCI = DC + IC		<b>24125</b>
<b>D. Working Capital</b>		
<b>Total Capital Investment = DC +IC+WC</b>		<b>265371</b>

\*All equipments are locally made in Egypt.

Table (5) Annual raw materials cost

Name of Material	Price / Ton L.E	Annual amount	Annual rate material cost L.E/year
Sodium hydroxide		8 kg	2
Ammonium chloride	200	22212kg	799632
Bittern* (Mg <sup>++</sup> )	36000		
Urine (PO <sup>-4</sup> )	200	153m <sup>3</sup>	30000
<b>Annual raw materials cost</b>		900L	829634

Table (6) Annual direct production cost

Item	Cost L.E/year
Annual raw materials cost (50% OF TPC)	829634
Operating labors (10% of TPC)	165927
Direct supervisor (10% of OL)	16593
Utilities (10% of TPC)	165927
Maintenance (2% of FCI)	4825
Operating supplies (2% of FCI)	4825
Laboratory charges (10% of OL)	165927
Patents & Roy utilities (1% of TPC)	16593
<b>Direct production cost</b>	<b>1220851</b>

\*Transportation cost of bittern considering no actual for waste bittern.

Table (7) Fixed charges cost

Item	Cost L.E/year
Depreciation (10% of FCI)	24125
Local Taxes (1% of FCI)	2413
Insurance (0.4% of FCI)	965
<b>Total fixed charges</b>	<b>27503</b>
<b>Plant overhead (15% of TPC)</b>	<b>165927</b>

**Manufacturing cost = Direct cost + Fixed charges + Plant overhead = 1414281 L.E**

Table (8) General Expenses

Item	Cost L.E/year
Administration Cost (2% of TPC)	27185
Distribution & Selling Cost (2% of TPC)	27185
Research & Development (2% of TPC)	27185
Financing (1% of TCI)	2654
<b>Total General Expenses</b>	<b>84209</b>

**Total production cost = Manufacturing cost + General Expenses = 1498490 L.E / 10.8 tons  
= 138749 L.E / ton = 139 L.E / kg**

## CONCLUSION

High similarity was observed between struvite powder precipitated using synthetic and original human urine. This reflects the accuracy and applicability of nutrient recovery efficiency from synthetic and authentic urine.

Struvite precipitation from hydrolyses of urine starts with fast nucleation which developed into crystals to average size 75 – 79 L (nm). Factors that do have influence on average crystals are not only super saturation but also pH and low temperature leads to smaller particle size. Struvite precipitation in this work was > 90%.

Separation management of human urine opens up the possibility of effective and circulation of nutrients not contaminated by hazardous substances. In this study, simple methods of concentrations of nutrient compounds in human urine were presented. The freezing – melting method makes it possible to capture 87% of the nutrients.

From the test of slow release fertilizer and activity index (AI)% the gradual release of nutrients (N,P,K) at ambient temperature and the significant increase of (N,P,K) release at 100 °C giving AI >33% after 22 hours only indicates struvite a good slow release fertilizer.

From the economic study as every new product which cannot be considered as economically viable, since the struvite produced is much higher than mixed fertilizer in local market (4000 L.E/ton). The system can be more efficient at a larger scale production and can help to offset the costs of wastewater treatment too.

So, from our results and recent research it revealed that it is possible to use human urine as an alternative source of multi-nutrient fertilizer which could be achieved only with introducing new areas of scientific research in frontier.

## Acknowledgment

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