Students' Card 1 Phosphorus Recovery from Wastewaters

Module 1

Objective: Extraction of struvite

Introduction

Urine, a product of human excretion, is a component of urban wastewater. Urine is one of the richest and most accessible sources of **phosphorus** and **nitrogen** to make struvite. The mineral could be obtained by a precipitation reaction at basic pH, helped by magnesium dependence (see the reaction below).

Reaction of precipitation of the struvite:

$$Mg^{2+} + NH_4^{+} + H_2PO_4^{-} \rightarrow MgNH_4PO_4 \cdot 6H_2O \downarrow + 2H^{+}$$

OBJECTIVE

The aim of the laboratory activity is to produce the synthetic urine, from which the struvite is extracted

Necessities

List of materials/tools

- Beaker 500 mL
- Spatulas
- Funnel
- Erlenmeyer flask 500 mL
- Precision balance (accuracy: 0.01g)
- Magnetic stirrer
- Magnetic anchor
- Universal pH indictor/ pH meter
- Filter paper
- Protective glasses
- Gloves

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Reagents	Formula		Quantity (g) or Concentration (M)
Sodium hydroxide	NaOH		0.5 M
Urea	CH_4N_2O		10 g
Sodium bicarbonate	$NaHCO_3$		1.05 g
Sodium sulphate decahydrate	$Na_2SO_4.10H_2O$		1.60 g
Ammonium chloride	NH_4Cl		0.65 g
Sodium chloride	NaCl		2.60 g
Potassium dihydrogen phosphate	KH_2PO_4		0.48 g
Potassium hydrogen phosphate	K_2HPO_4		0.60 g
Calcium dihydrate chloride	$CaCl_2.2H_2O$		0.19 g
Distilled or demineralized water			
Magnesium sulphate	$MgSO_4$		0.25 g/L

Lab Procedure

Wear your gloves and protective glasses.

I. Preparation of synthetic urine

1. 10 g of urea are transferred into a 500 mL beaker and 100 mL of distilled or demineralized water is added;
2. place the beaker on the magnetic stirrer, setting between 100 and 300 rpm of velocity;
3. add the reagents on indicated order and values, stirring;
4. add almost 400 mL of water and wait until a complete solubilization;

*The indicated quantities are those necessary to prepare nearly 500 mL of wastewater synthetic

II. Preparation of struvite

5. weigh the magnesium sulphate and add it to the solution;
6. control the pH with a pH meter or an universal pH indicator; eventually add 1/2 drops of NaOH 0.5 M till pH = 8 and continue stirring;

The range of pH needed to obtain an optimal precipitation is pH = 8.0 - 8.5.

Leave the solution in agitation at least one or two hours to foster the precipitation of the struvite.

The precipitation starts after nearly 3 hours;

7. filter the solution with the filter paper in order to restore the precipitate and leave it then dry at room-temperature;
8. pick up the struvite in a clean and dry container .

The students will be facilitated in the struvite preparation by watching the video

Additional Safety Notes *(if necessary)*

Be careful using NaOH.

Questions

1. Which aspect does the precipitate take after the drying?
2. Why do you think it was used the magnetic stirrer?
3. Which function does NaOH have?

Additional materials to better support the activity proposed by the toolkit is proposed in:

- Additional Information
- tutorial videos for explaining the experimental phases

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Additional Information

Nitrogen Cycle

Nitrogen is an essential ingredient for life as we know it. It a component of **proteins**, **nucleic acids** and **chlorophyll**. Different steps characterize **N cycle** (Figure 1):

-Fixation

-Assimilation

-Ammonification

- Nitrification

-Denitrification

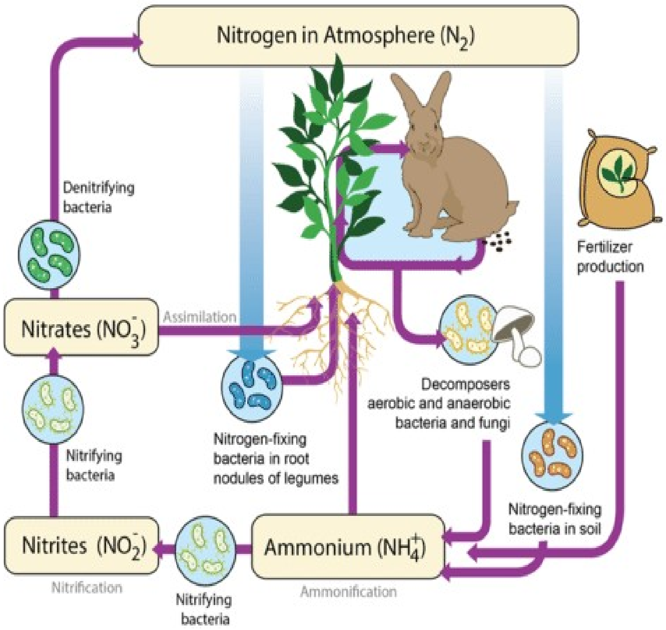


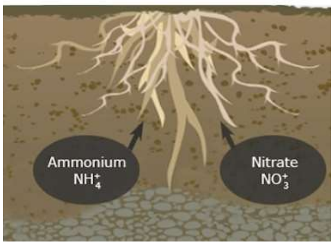
Figure 1. The nitrogen cycle in soil.

Nitrogen in each step is characterized by several oxidation state

Molecule	Name	Oxidation state	
C-NH$_2$	Organic-N	Reduced	
NH_3, NH_4^+	Ammonia, Ammonium	-3	
N_2H_4	Hydrazine	-2	More electrons
NH_2OH	Hydroxylamine	-1	
N_2	Dinitrogen	0	
N_2O	Nitrous oxide	+1	
NO	Nitric oxide	+2	
HNO_2, NO_2^-	Nitrous acid, Nitrite	+3	Fewer electrons
NO_2	Nitrogen dioxide	+4	
HNO_3, NO_3^-	Nitric acid, Nitrate	+5	
		Oxidized	

Current Biology

Which N forms are important for plants?



Plants acquire these forms of "combined" **nitrogen** by:
1) **Addition** of mineral **fertilizer** (ammonia and/or nitrate) or **manure** to soil;
2) **Release** of these compounds during **organic matter** decomposition;
3) **Biological** nitrogen fixation.

Fig.2. Plants uptake different mineral nitrogen forms

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Are there other nitrogen source in the environment?

Most **nitrogen** contained in **wastewaters is** derived from **urea** and **fecal material.** These compounds are converted into **ammonium (ammonification)** by bacteria activity. In presence of oxygen, ammonium is transformed into nitrate **(nitrification)** (Figure 3).

Ammonification **Nitrification**

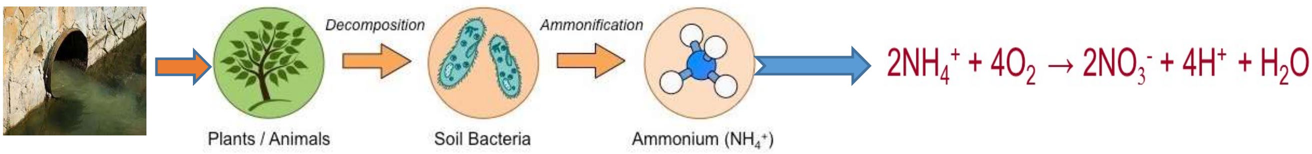

Figure 3. Ammonification and nitrification are important reactions to transform urea and fecal material into ammonium and nitrite, respectively.

Can we recovery phosphorus from wastewater treatment?

Urban **wastewaters** may contain from 5 to 20 mg/L of **total phosphorous** (inorganic, biological and synthetic detergents) Normally secondary treatment can only remove 1-2 mg/L, so a large excess of phosphorous is discharged in the final effluent, causing **eutrophication** in surface waters.

New legislation requires a maximum concentration of P discharges into sensitive water of 2 mg/L.

Phosphorus can be removed and recovered from wastewaters by chemical precipitation in crystal form of **struvite** (Figure. 4)**.**

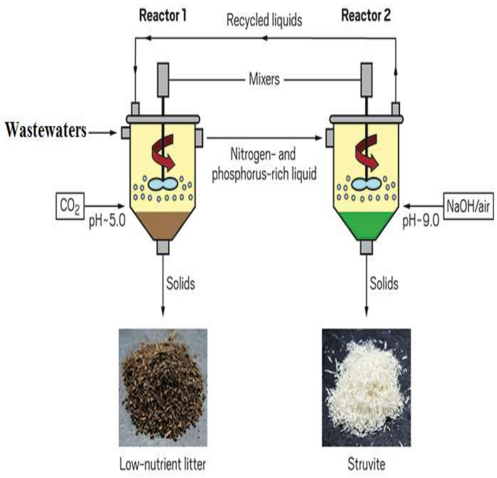

Figure 4. Extraction of struvite from wastewaters