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공학석사학위논문

Banana Peel Ash as An Additive to Derive  
Struvite Fertilizer from Source-separated Urine

바나나 껍질 재를 분리 된 소변에 첨가제로  
활용하여 스트루바이트 비료로 전환하는 방법

2021년 2월

서울대학교 대학원

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박 은 하

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## Abstract

# Banana Peel Ash as An Additive to Derive Struvite Fertilizer from Source-separated Urine

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Nearly 30% of people worldwide does not have access to proper sanitation facilities due to reasons such as lack of water resources. Dry toilets have been introduced to overcome these challenges and urine diverting dry toilet (UDDT) is a type of a dry toilet that collects urine and feces separately. Human urine can be used as a good source of Nitrogen and phosphorous in agriculture but, prior to application urine should be treated. Many additives have been investigated that could stabilize urine but, in developing countries, usage of these materials is not economically feasible. As such, many researchers have investigated nature-based materials such as wood ash (WA) to use as a Magnesium source to promote struvite generation. However, when WA is used as an additive for phosphorous recovery, researchers have observed high heavy metal concentrations in the precipitate, rendering the precipitate unusable as a fertilizer. Banana is a cheap and widely available crop, and its peel ash (BPA) could be used as a good alternative. In this study, we

investigated the effect of BPA in stabilizing fresh urine. When BPA was added in doses higher than 10 g/ 500 ml urine, the pH increased above 11 thus, urea hydrolysis was obstructed. Moreover, this research focused on investigating the effect of BPA on nitrogen and phosphorous recovery from synthetic wastewater and stored urine. Synthetic wastewater mixed with BPA doses above 10 g/ 500 ml showed no significant improvement in Nitrogen and Phosphate recovery. Nitrogen and Phosphate reduced significantly compared to the control sample due to struvite generation. This was validated by repeating the experiment on stored urine where, the precipitation demonstrated similar band pattern on XRD analysis as to that of struvite. Therefore, authors have concluded that the BPA can be used to recover nutrients in urine and a dosage of 10 g/ 500 ml of urine is optimum to achieve the highest struvite yield. Authors propose an innovative toilet design based on the findings of this research.

**Keyword:** UDDTs; human urine; nutrient recovery; struvite; banana peel ash; urea hydrolysis

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## Table of Contents

List of Figures .....	6
List of Tables.....	8
CHAPTER 1. INTRODUCTION .....	9
1.1. Current global sanitation situation .....	9
1.2. Urine-diverting dry toilets.....	10
1.3. Human urine.....	11
1.3.1. Urea hydrolysis .....	13
1.3.2. Struvite precipitation.....	13
1.4 Banana peel ash.....	15
1.5. Objective of the study .....	18
1.6. Dissertation structure.....	18
CHAPTER 2. INVESTIGATE THE EFFECT OF PH CONTROL FOR UREA STABILIZATION.....	20
2.1 Material and methods.....	20
2.1.1 Fresh urine, Banana peel ash.....	20
2.1.2. Reactors.....	23
2.1.3 Experimental setup.....	24
2.1.4 Physico-chemical analysis.....	25
2.1.5 Adsorption experiment.....	26

2.2 Results and discussion.....	27
2.2.1 Evaluation of the effect of adding BPA on pH change of human urine .....	27
2.2.2 NH <sub>4</sub> <sup>+</sup> change by adding different dosage of BPA .....	29
2.2.3 TN change by adding different dosage of BPA .....	30
2.2.4 PO <sub>4</sub> <sup>3-</sup> change by adding different dosage of BPA .....	32
2.2.5 PO <sub>4</sub> <sup>3-</sup> adsorption test with different dosage of BPA .....	32
2.3 Summary .....	34
 CHAPTER 3. INVESTIGATING P AND N RECOVERY BY STRUVITE PRODUCTION FROM SYNTHETIC WASTE WATER AND STORED URINE.....	
3.1 Material and methods .....	35
3.1.1. Stored urine .....	35
3.1.2 Synthetic waste water.....	36
3.1.3. Identifying the characteristics of precipitation .....	38
3.2. Results and discussion.....	38
3.2.1. The effect of BPA on pH of SWW.....	38
3.2.2. The effect of BPA on NH <sub>4</sub> <sup>+</sup> removal .....	40
3.2.3. The effect of BPA on phosphate removal .....	41
3.3.4. Precipitation analysis.....	43

3.4. Summary .....	44
CHAPTER 4. SUGGEST BPA APPLICATION GUIDELINE FOR CONVERTING URINE INTO FERTILIZER.....	45
4.1. Urine storage tank under the UDDT .....	45
4.1.1. Calculation .....	45
4.1.2. Simulation of system.....	50
CHAPTER 5. CONCLUSIONS AND RECOMMENDATIONS .....	61
5.1 Conclusions.....	61
5.2 Recommendation for further study .....	61
Reference.....	65

## List of Figures

Figure 1. 1 Percentage of people without access to improved sanitation facilities in different regions in 2017 (Adapted from WHO/UNICEF, 2019) .....	10
Figure 1. 2 Urine-diverting dry toilets (UDDTs) .....	11
Figure 1. 3 Banana peel.....	16
Figure 1. 4 Dissertation structure .....	19
Figure 2. 1 Urine container .....	21
Figure 2. 2 Banana peel ash (BPA) production process .....	22
Figure 2. 3 BET plots of BPA .....	23
Figure 2. 4 Urine reactors with banana peel ash .....	24
Figure 2. 5 adsorption experimental set-up .....	26
Figure 2. 6 pH changes with different dosage of BPA over time.....	28
Figure 2. 7 Urea hydrolysis occur based on pH and Temperature. (Randall et al., 2016).....	28
Figure 2. 8 $\text{NH}_4^+$ changes with different dosage of BPA over time .....	30
Figure 2. 9 $\text{NH}_4^+$ and $\text{NH}_3$ distribution as a function of pH (AIRTON KUNZ, 2016) .....	31
Figure 2. 10 TN changes with different dosage of BPA over time .....	31
Figure 2. 11 $\text{PO}_4^{3-}$ changes with different dosage of BPA over time .....	32
Figure 2. 12 $\text{PO}_4^{3-}$ changes with different dosage of BPA over time .....	33
Figure 3. 1 Storage tank used to Store urine .....	36
Figure 3. 2 Variation of the pH of SWW and SU with time .....	39
Figure 3. 3 Change in $\text{NH}_4^+$ concentration of SWW and SU with time .....	41

Figure 3. 4 Variation of Phosphate concentration of SWW and SU with time (for a total of 5 days).....	42
Figure 3. 5 Variation of Phosphate concentration of SWW and SU with time .....	42
Figure 3. 6 XRD result of BPA precipitation after treatment .....	43
Figure 4. 1 Top view of urine tank under UDDT.....	46
Figure 4. 2 Section A-A of urine tank under UDDT.....	47
Figure 4. 3 Section B-B of urine tank under UDDT .....	47
Figure 4. 4 Section C-C of urine tank under UDDT .....	48
Figure 4. 5 Section D-D empty urine tank and each section.....	49
Figure 4. 6 Section D-D of urine tank (Part 1: Inhibit urea hydrolysis).....	51
Figure 4. 7 Section D-D of urine tank (Part 2: Urea hydrolysis by decreasing pH) .....	53
Figure 4. 8 Section D-D of urine tank (Part 3: Struvite production).....	55
Figure 4. 9 Section D-D of urine tank (Part 4: Storage).....	57
Figure 4. 10 Section D-D of urine tank (Collect urine).....	59
Figure 4. 11 Section D-D of urine tank (Collect precipitation).....	60

## **List of Tables**

Table 1. 1 Composition of human urine (Adapted from Alfey (2016)) .....	12
Table 1. 2 The composition of banana peel ash .....	17
Table 2. 1 Initial physio-chemical characteristics of human urine.....	21
Table 2. 2 Physical characteristics of BPA .....	23
Table 2. 3 Dosage of urine and BPA reactors .....	25
Table 3. 1 Initial physio-chemical characteristics of stored human urine.....	35
Table 3. 2 Initial physio-chemical characteristics of average human urine and synthetic waste water .....	38
Table 3. 3 Composition of BPA before and after treating Stored urine .....	44

## CHAPTER 1. INTRODUCTION

### 1.1. Current global sanitation situation

In 2017, Joint Monitoring Program for Water Supply, Sanitation and Hygiene (JMP) by WHO/UNICEF reported that, more than 2.0 billion people still do not have toilets or latrines, and 673 million people still practice open defecation (Organization, 2019). As a result, about 827,000 people die per annum in developing countries due to issues mentioned above (WHO, 2019). As illustrated in Figure 1.1., improvement of sanitation facilities has been disproportionate worldwide. There is a big disparity between developing countries and developed countries in terms of sanitation. The regions with the poorest sanitary conditions are developing countries as Sub-Saharan Africa (51%), Oceania (66%), Least Developed Countries (50%) and Landlocked Developing Countries (49%) (Organization, 2019).

Therefore, United Nations (UN) in 2015 (UN, 2015) introduced 17 Sustainable Development Goals (SDGs) among which, target 6.2: “By 2030, achieve access to adequate and equitable sanitation and hygiene for all and end open defecation, paying special attention to the needs of women and girls and those in vulnerable situations”, emphasize on providing improved sanitation facilities to an additional 26% of global population (2.0 billion people) by 2030.

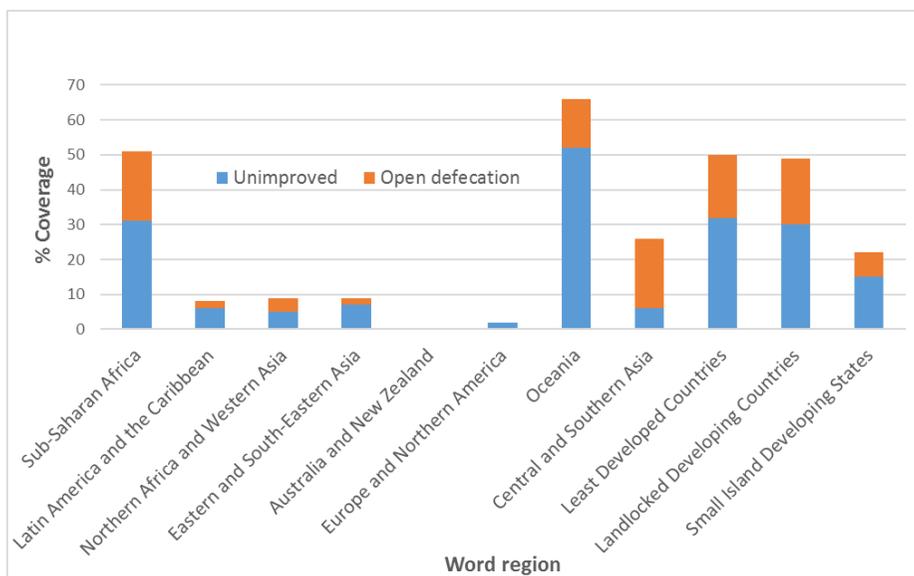


Figure 1. 1 Percentage of people without access to improved sanitation facilities in different regions in 2017 (Adapted from WHO/UNICEF, 2019)

## 1.2. Urine-diverting dry toilets

Urine-diverting dry toilet (UDDT) is a type of dry toilet, where feces and urine are collected and treated separately. UDDT are considered as a sustainable sanitation method for excretal management due to benefits such as zero water consumption, low investment cost and relatively easy adoptability (Andreev et al., 2017).

Global population growth, depletion of freshwater bodies and nutrients in soil, and lack of access to proper sanitation facilities have promoted the application of UDDTs in rural areas of developing countries (Lalander et al., 2013). However, there are challenges such as odor generation, lack of cost-effective technologies to treat feces and urine, and higher retention times that

needs to be resolved for better acceptance of UDDTs by communities. Among these issues, urine treatment is stressed by many researchers.



(a)



(b)

Figure 1. 2 Urine-diverting dry toilets (UDDTs)

(a), and sitting toilet (b) squatting toilet

### 1.3. Human urine

Approximately 0.5 m<sup>3</sup> of urine and 55 m<sup>3</sup> of wastewater per person is generated annually. Most of the macronutrient such as nitrogen (79%), phosphorus (47%) and potassium (71%) in wastewater is from urine (Friedler et al., 2013). Every day, the human body excretes approximately 1.3 L (1.0 – 1.5 L) of urine, which contains a lot of essential nutrients (nitrogen, phosphorus, potassium)(Chang et al., 2013). Most of nitrogen (N) in fresh urine exist as urea (85%) whereas, ammoniacal nitrogen and other organic nitrogen such as creatinine, uric acid and creatine are present in small amounts (5% and 10%, respectively) (Udert et al., 2006). Phosphorus is predominantly present as PO<sub>4</sub><sup>3-</sup> form, and many micronutrients such as B, Fe, Zn, Mo, Mn, Cu are also available

in human urine (Udert et al., 2006). The composition of urine is presented as Table 1.1.

Table 1. 1 Composition of human urine (Adapted from Alfey (2016))

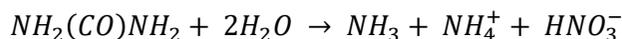
Composition	Formula	Concentration (%)
Water	H <sub>2</sub> O	95
Urea	CH <sub>4</sub> N <sub>2</sub> O	2
Ammonia	NH <sub>3</sub>	0.05
Sulphate	SO <sub>4</sub> <sup>2-</sup>	0.18
Phosphate	PO <sub>4</sub> <sup>3-</sup>	0.12
Chloride	Cl-	0.6
Magnesium	Mg	0.01
Calcium	Ca	0.015
Potassium	K	0.6
Sodium	Na	0.1
Creatinine	C <sub>4</sub> H <sub>7</sub> N <sub>3</sub> O	0.1
Uric acid	C <sub>5</sub> H <sub>4</sub> N <sub>4</sub> O <sub>3</sub>	0.03

Besides nutrients, human urine may contain about 240 bacterial species (Lahr et al., 2016). Some species such as *Schistosoma haematobium*, *Salmonella typhi*, *Salmonella paratyphi*, *Leptospira interrogans* are pathogenic (Feachem et al., 1983). However, most of these bacteria come from cross-contamination with feces at UDDT facilities (Höglund et al., 1998). Though urine can be used as fertilizer, there are some factors such as complex

transposition to move urine from the house to agricultural lands (Jönsson et al., 1999), and possible health risks of using urine on crops directly (Höglund et al., 2002) that has limited its application.

### ***1.3.1. Urea hydrolysis***

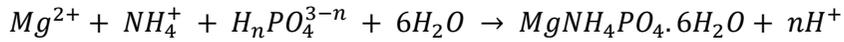
The urea hydrolysis (ureolysis) is a process, where enzyme urease hydrolyzes urea into ammonia and carbamate (Mobley & Hausinger, 1989; Udert et al., 2003a). During this process the pH increases due to ammonia generation. The ureolysis reaction is as follows:



There are many factors that affect ureolysis such as the availability of nitrogen, the pH, and the presence of some bacteria (Mobley et al., 1995). The half-life of non-enzymatic urea hydrolysis is about 3.6 years at 38 ° C (ANDREWS et al., 1984). Therefore, urea has to be stored for a longer time for stabilization. But, during storage, nutrients in urine can be lost thus, we need a method to preserve the nutrients in urine. Struvite precipitation is as a simple method to overcome these problems also, by doing so, phosphorus; one of the important nutrients for crop growth (Larsen & Gujer, 1996) can be recovered.

### ***1.3.2. Struvite precipitation***

Struvite (magnesium ammonium phosphate hexahydrate) is a type of phosphate mineral ( $MgNH_4PO_4 \cdot 6H_2O$ ). Struvite precipitation occurs when Mg and  $PO_4^{3-}$  are present in urine. Struvite generation is as follows (Siciliano et al., 2020):



Where n = 0, 1, or 2

As struvite capture the  $NH_4^+$  in urine, volatilization and subsequent loss of Nitrogen as ammonia gas can be minimized. However, because of the low pH of urine, struvite precipitation does not start immediately such that most of Nitrogen is lost before struvite precipitation starts. Therefore, many additives have been investigated that can be used as a catalyst to speed-up this process. The pH of stored urine plays a key role for phosphorus recovery from the urine through struvite precipitation (Udert et al., 2003b). Typical sources of Mg such as magnesium chloride ( $MgCl_2$ ), magnesium oxide ( $MgO$ ) and magnesium sulfate ( $MgSO_4$ ) are widely used to treat wastewater by struvite precipitation. (Uludag-Demirer et al., 2005). Antonini et al. (2011) reported that by adding a  $MgO$  dose of 1.5 mol Mg / mol, 98% of  $PO_4^{3-}$  in urine was recovered. However, the production costs of these salts are high thus, a cost-effective source is needed specially, for developing countries. Etter et al. (2011) found that the bittern, a type of waste from salt product, contains high level of Mg, which can react with urine to produce struvite. In addition, 90% of P in urine is removed even with a low dose of Mg in bittern (1.1 mol Mg / mol P). Sakthivel et al. (2012) used wood ash as a magnesium source to recover phosphorus from source-separated urine. In their study, they found that 99% of the phosphate was precipitated with a magnesium dosage of 2.7 mol Mg/mol P, and only 50% of magnesium in the wood ash reacted with urine to generate the struvite.

However, due to the presence of heavy metals, WA treated urine cannot be used as a fertilizer.

#### **1.4 Banana peel ash**

Banana is one of the most popular fruits that contain micronutrients needed for a healthy life (Aurore et al., 2009). Recently, demand for banana has increased as banana is a good source of vitamin. The biggest banana yield is from developing countries such as Ecuador, Philippines, Costa Rica, and Colombia (FAOSTAT, 2012).

About 102 million tons of banana is produced every year (FAOSTAT, 2012). The peel of this fruit makes up 35 % of the total weight of the fruit (Vu et al., 2017). Therefore, a significant quantity of banana peel (26 million tons) is generated annually, and there are no uses for banana peels so, they are discarded as waste. Currently, most of the peel is landfilled as a general municipal soil waste (Schieber et al., 2001). However, organic matter in banana peel can be used as a fuel in rural areas when it is dry. By burning the banana peel we can obtain banana peel ash (Figure 1.3).



(a)

(b)

Figure 1. 3 Banana peel

(a) Discarded banana peels, (b) Banana peel ash

Banana peel ash (BPA) contain many types of nutrients which were measured using S 4 PIONEER X-ray fluorescence (WDXRF) spectrometry (Bruker AXS, Germany) and compound concentrations measured were then converted to element concentrations. Nutrients detected including macro-and micro-nutrients such as phosphorus, potassium, calcium, sulfur, magnesium, iron, manganese, etc. are presented in Table 1.2. The macronutrient (nitrogen, phosphorus, potassium, calcium, sulfur, magnesium, etc.) and micronutrients (iron, boron, chorine, manganese, zinc, copper, nickel, etc.) are essential nutrients for plants growth (Panwar, 2015). These elements can be taken up by plants through roots. Therefore, BPA can be considered a potential fertilizer that can provide nutrients for plants instead of discarding them as waste.

Famers form Masindi, Kapapchorwa, Mukonom Buikwe and Nwoya districts in (Africa) add BPA on their stored urine before using it as a fertilizer. This practice has reduced the risk of disease caused by micro-organisms.(Foundation, 2012)

BPA contains 7.6 g/kg of Mg and can be used to control the pH of urine.

Table 1. 2 The composition of banana peel ash

Composition	Formula	Concentration (%)
Calcium	Ca	4.274
Magnesium	Mg	0.76
Phosphorus	P	0.585
Potassium	K	31.74
Silicon	Si	1.552
Chloride	Cl	9.411
Iron (III)	Fe	0.038
Manganese (II)	Mn	0.021
Sulfur	S	0.184
Aluminum	Al	0.041
Zinc	Zn	0.0247
Strontium	Sr	0.0145
Copper(II)	Cu	0.0096
Rubidium	Rb	0.008
Chromium(III)	Cr	0.003
Bromine	Br	0.025

### **1.5. Objective of the study**

The objective of this study is to investigate the feasibility of using Banana Peel Ash (BPA) as an additive for human urine treatment. Further, the study investigates the effect of BPA on both fresh and stored urine, focusing on recovering P and N. Finally, this study proposes a field applicable design. Therefore, this study covers three parts, followings:

- 1) Investigate the effect of BPA on controlling the pH of urea
- 2) Investigate P and N recovery by struvite precipitation with BPA as an additive from synthetic wastewater and stored urine.
- 3) Suggesting an application guideline for converting urine into fertilizer using BPA for fresh and stored urine

### **1.6. Dissertation structure**

There are 5 chapters in this dissertation. Chapter 1 gives details of current sanitation situation in the world, the challenges of human urine treatment, characteristics of banana peel ash, research objectives, and dissertation structure. Chapter 2 discuss the experimental results obtained for treating fresh urine by banana peel ash. Chapter 3 focuses on recovering P and N from synthetics wastewater and stored urine by adding BPA. Chapter 4 suggests a design for applying banana peel ash into UDDT system for nutrient recovery. The structure of this dissertation is presented in Figure 1.4

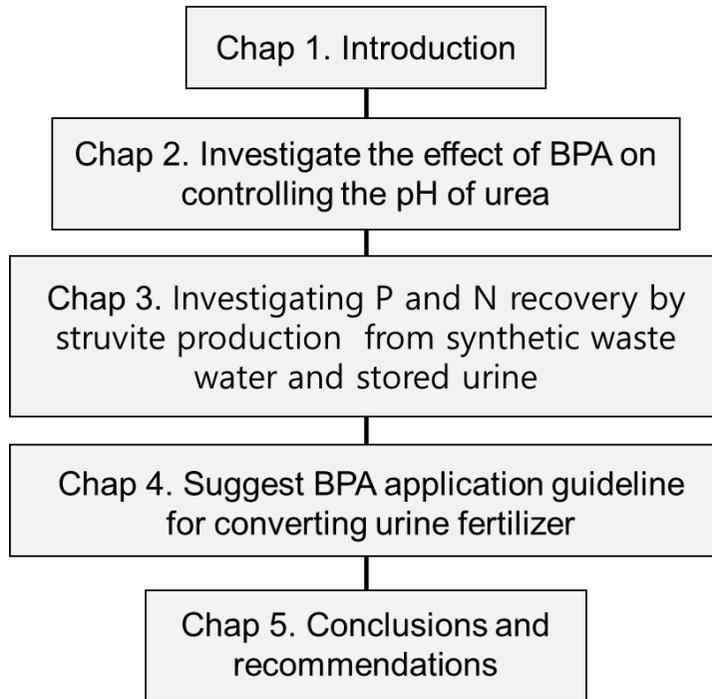


Figure 1. 4 Dissertation structure

## CHAPTER 2. INVESTIGATE THE EFFECT OF PH CONTROL FOR UREA STABILIZATION

### 2.1 Material and methods

#### 2.1.1 *Fresh urine, Banana peel ash*

Fresh urine was collected from a urine-diverting dry toilet (UDDT) at the rooftop of building 35, Seoul National University, South Korea to a plastic container as shown in Figure 2.1. Urine was collected within a day and used immediately for experiments. Hydrolysis of Urea occurs after 72 to 84 hours (H) of collection (Ray et al., 2018); a process accelerated by the presence of Urease therefore, physio-chemical parameters of urine were measured immediately. Fresh urine contained a high nitrogen (N) amount in the form of Urea, the pH ranged from 6 to 8 (Andrade & Costa, 2007; Maurer et al., 2006; Miki & Sudo, 1998) The physio-chemical characters of Urea can vary depending on the dietary and metabolic diversities of individuals. (Siener & Hesse, 2002) In this study, we focused on preserving the nutrients present in Urine thus, the parameters measured were limited to those presented in Table 2.1.



Figure 2. 1 Urine container

Table 2. 1 Initial physio-chemical characteristics of human urine

Parameter	Fresh urine
pH	6.14 - 6.87
Total Nitrogen (mg/L)	1781 - 4310
Urea (mg/L)	1426.5 - 3945.5
Ammonium (mg/L)	325 - 457.2
Nitrate (mg/L)	39.5 – 75
Phosphorous (mg/L)	30 – 40

Banana peel (*Musa acuminata cavendish*) was collected and dried at 40 °C for 3 days. Dried banana peel was burned at 500 °C for 15 min to produce Banana Peel Ash (BPA). The process diagram for obtaining BPA is presented in Figure 2.2. Surface area of BPA was measured using Brunnaunauer Emmeett Tellet (BET) with BELPREP-vac III system with nitrogen as the adsorbate gas.

The adsorbent capacity of BPA is as shown in Figure 3.2 and Table 3.3. BPA used for experiment had a very low surface area compared to (Jedidiah, 2019). BPA used by (Jedidiah, 2019) had a higher surface area due to the chemical purification.



Figure 2. 2 Banana peel ash (BPA) production process

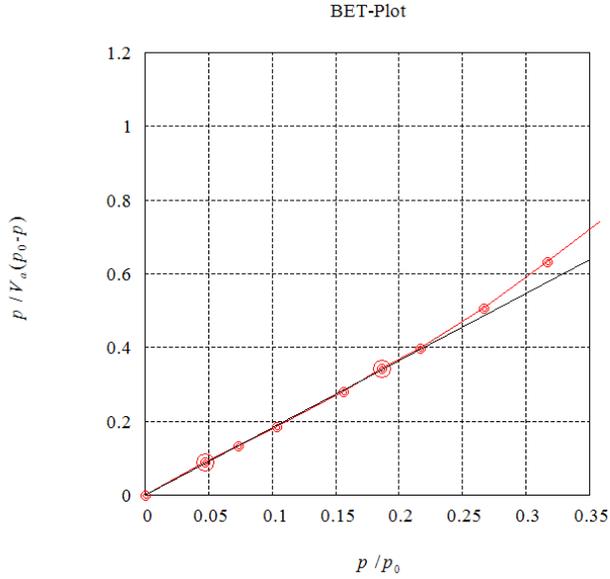


Figure 2. 3 BET plots of BPA

Table 2. 2 Physical characteristics of BPA

Parameter	Referece(Jedidiah, 2019)	Range of BET for u
$A_{sBET}$	63.67 m <sup>2</sup> /g	2.391m <sup>2</sup> /g
$V_m$	-	0.5495cm <sup>3</sup> /g
Pore size	21.07nm	25.831nm

### 2.1.2. Reactors

Glass bottles with a capacity of 500 ml were used as reactors for this experiment (Figure 2.4). During the experiment, all the bottles were covered by plastic covers to create a closed system.

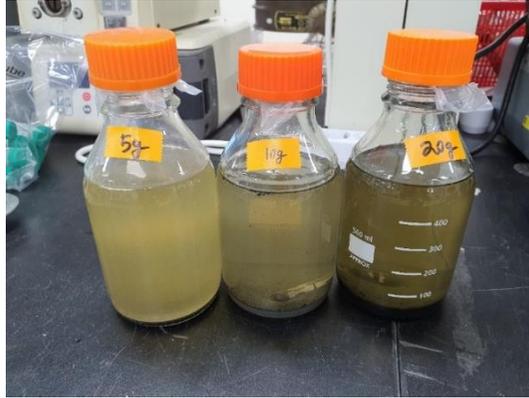


Figure 2. 4Urine reactors with banana peel ash

### ***2.1.3 Experimental setup***

Four sets of reactors were prepared namely, the control (fresh urine) and three Fresh urine reactors with different BPA dosages. BPA dosages considered in the study is presented in Table 2.3. After introducing BPA to 500 ml of Urine, the solution was mixed with magnetic stir at 350 rpm for 15 minutes (Ray et al., 2018) The mixed solution was kept at room temperature ( $25 \pm 3$  °C) for more than one month to observe the changes of Nitrogen and phosphorous content over time. A sample of 4 mL was extracted three times during first week, after which sampling frequency was reduced to once a week.

Table 2. 3 Dosage of urine and BPA reactors

Mark	Dosage			
	Control (fresh urine	5g (5 g/500 mL)	10g (10 g/500 mL)	20g (20 g/500 mL)
Fresh urine (ml)		500		
BPA (g)	-	5	10	20

#### 2.1.4 Physico-chemical analysis

Chemical composition (Total N,  $\text{NH}_4^+\text{-N}$ ,  $\text{PO}_4^{3-}\text{-P}$ ) were analyzed immediately upon Urine collection. When immediate analysis was impossible, the urine samples were kept in the refrigerator at 4 °C to preserve the chemical composition. The pH was measured using DKK-TOA Portable pH meter HM-31P, Japan. Total nitrogen (TN) was measured according to Cadmium reduction method, ammonium-nitrogen ( $\text{NH}_4^+\text{-N}$ ) according to Nessler method, and phosphate-Phosphorous ( $\text{PO}_4^{3-}\text{-P}$ ) according to Ascorbic acid method was derived using the two previous observations. All the analysis was performed via spectrometry using the HUMAS UV/Visible spectrometer, Model HS-3300 (HUMAS, Korea)

### 2.1.5 Adsorption experiment

Firstly, a standard solution of  $\text{PO}_4^{3-}$  was made following standard methods (환경보건기술연구원, 2017).  $\text{KH}_2\text{PO}_4$  (Daejung chemicals, Korea) was oven dried for 24 hours at 120 °C, and 0.439 g of the oven-dried sample was mixed with 1 L of distilled water using a magnetic stir to produce a 100 mg/L solution of  $\text{PO}_4^{3-}$ . Four sets of glass bottles with 100 ml capacity were used as reactors for the experiment. Three various of dosage of BPA was added into each bottle; 1 g/100 ml, 2 g/100 ml, 4 g/100 ml, respectively. After introducing BPA to 500 ml of standard solution, the solution was mixed with magnetic stir at 200 rpm for 6 hours (Ahmed et al., 2014) The mixed solution was kept at room temperature ( $25 \pm 3$  °C) for a day to observe the changes of  $\text{PO}_4^{3-}$  content over time. A sample of 1 mL was extracted 2 hours during 6 hours in the first day after which, another sample was taken the next day. Experimental set-up is shown in Figure 2.5

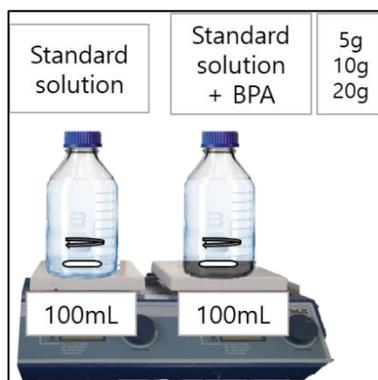


Figure 2. 5 adsorption experimental set-up

## **2.2 Results and discussion**

### ***2.2.1 Evaluation of the effect of adding BPA on pH change of human urine***

Development of acidic conditions in soil is a common problem when using chemical fertilizer. Such conditions could be traced to loss of nutrients retained by the soils also, affecting flora in a negative way (Guo et al., 2010; Wright et al., 2001; Yang et al., 2012). This phenomenon has limited the application of Urine as fertilizer because urea hydrolysis lowers the pH of Urine. Therefore, Urine has to be stabilized prior to using as a fertilizer.

Addition of BPA has been observed to increase the pH of aqueous medium by many researchers. Without the addition of BPA, the pH increased to about 9 after 45 days from the initial pH of 6.14 (Figure 2.6) because of urea hydrolysis. However, addition of BPA immediately increased the pH over 10. A BPA dosage of 5 g/ 500 mL increased the pH to around 10, and following the findings of (Randall et al., 2016) enzymatic urea hydrolysis can still occur at room temperature (25 °C) under this pH condition (Figure 2.7). For the case of dosages above 10 g/500 ml, pH increased beyond 11 so, loss of urea by urea hydrolysis was minimum. During one-month of storage, pH slightly decreased to around 11. Though, this is still acceptable, for the stable storage of urine for longer periods, more than 10 g/500 ml dose is recommended. This can be observed for the case of 20 g/ 500 ml dosage where the pH was constantly above 11 during experiment period (42 days). Furthermore, the increase of pH during

the urine storage can contribute to pathogen die-off (Von Muench & Winker, 2011).

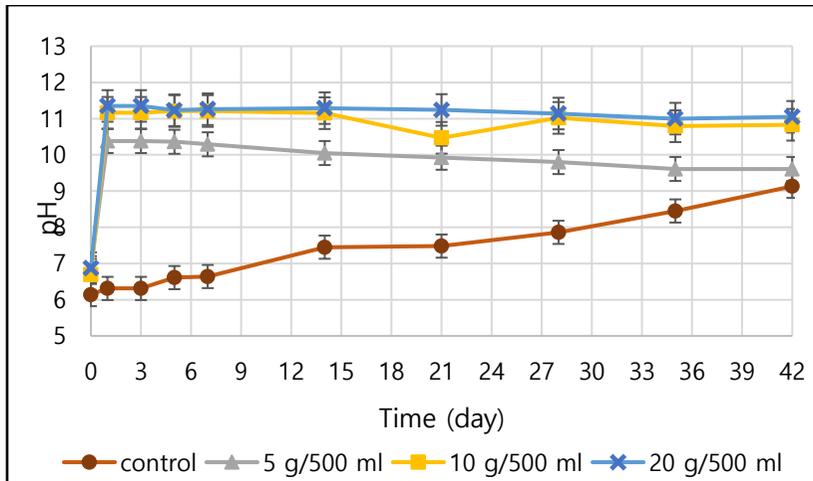


Figure 2. 6 pH changes with different dosage of BPA over time

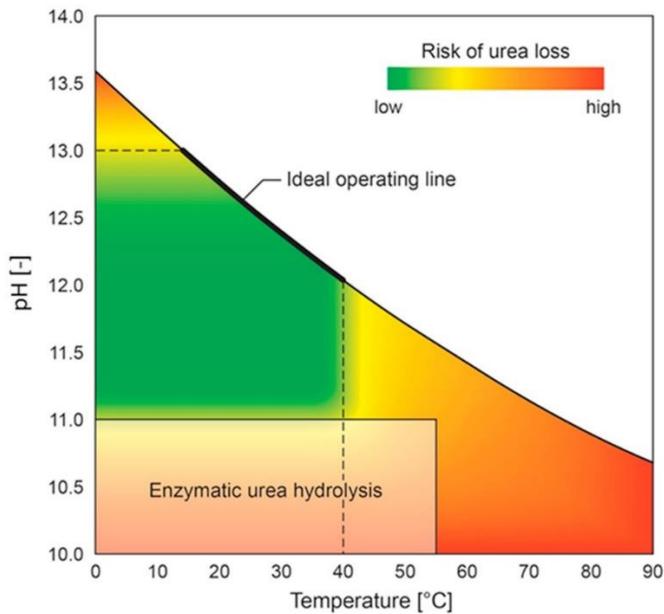


Figure 2. 7 Urea hydrolysis occur based on pH and Temperature. (Randall et al., 2016)

### **2.2.2 $\text{NH}_4^+$ change by adding different dosage of BPA**

Ammonium ( $\text{NH}_4^+$ ) is generated naturally as urea hydrolyze. In the control sample,  $\text{NH}_4^+$  concentration increased slightly to 435 mg-N/L from 325 mg-N/L (Figure 2.8) during first 20 days. From 20<sup>th</sup> day to 42<sup>nd</sup> day  $\text{NH}_4^+$  concentration nearly doubled (602.5 mg-N/L).

Addition of BPA reduced the production of  $\text{NH}_4^+$ . A dosage of 5 g/500 ml reduced ammonium production by a factor greater than two during the first three days after which,  $\text{NH}_4^+$  concentrations has risen due to the volatilization of  $\text{NH}_4^+$  as  $\text{NH}_3$  gas. The pH was at 10 thus,  $\text{NH}_4^+$  concentration rapidly increased to 406.5 mg-N/L as urea hydrolyzed.

The dosage of 10 g/500 ml also inhibited urea hydrolysis but, after 21 days, the pH decreased below 11, restarting the urea hydrolysis process.

A BPA dose of 20 g/500 ml inhibited  $\text{NH}_4^+$  production as the pH was maintained above 11.  $\text{NH}_4^+$  concentration was stable at 170 mg-N/L under this dosage.

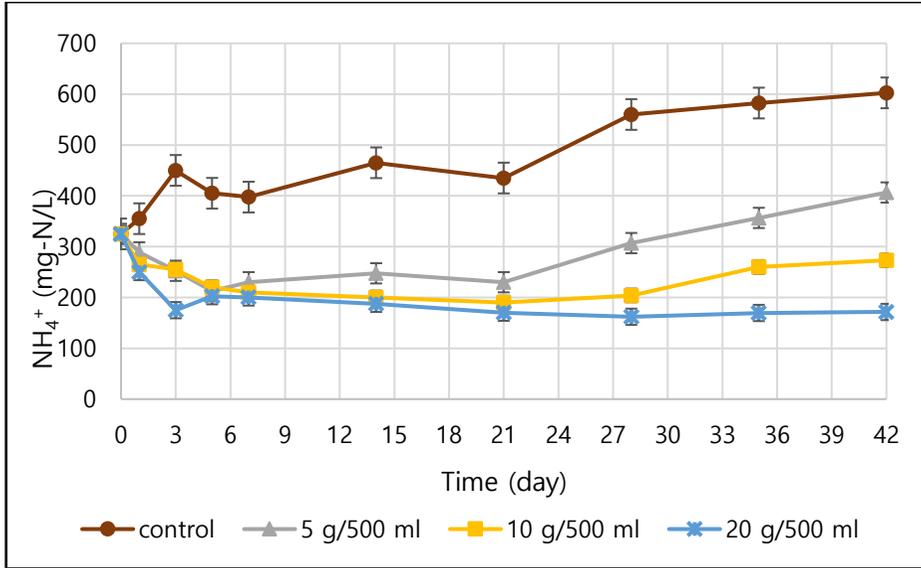


Figure 2. 8  $\text{NH}_4^+$  changes with different dosage of BPA over time

### 2.2.3 TN change by adding different dosage of BPA

TN in the reactor has reduced over time (Figure 2.10). Urea hydrolysis create more  $\text{NH}_4^+$  in the aqueous medium thus, Nitrogen can be lost because  $\text{NH}_4^+$  is easily volatilized as  $\text{NH}_3$  gas. Though the system was closed,  $\text{NH}_3$  gas was released from the system when the reactors were opened for sampling purposes. This can be attributed to the reduction in TN. This phenomenon is influenced by the pH of the medium as shown in Figure 2.9. If pH is above 9,  $\text{NH}_3$  gas is predominant. Addition of BPA has reduced the TN loss by more than 1000 mg-N/L if a dose of 20 g/ 500 ml is administered.

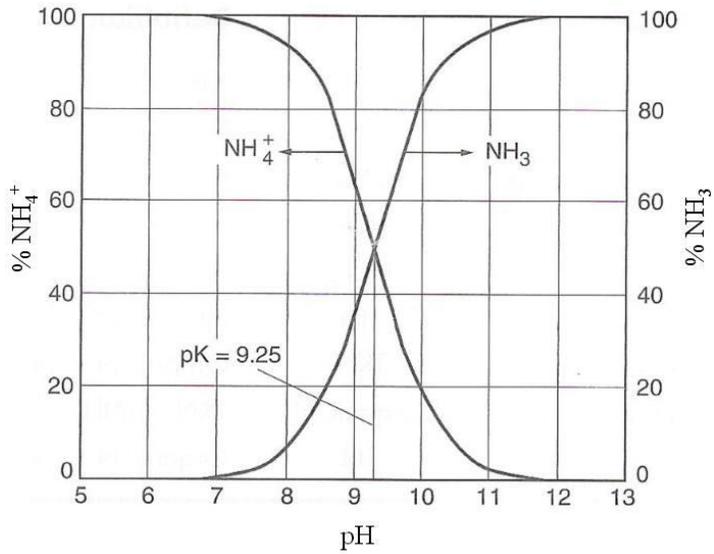


Figure 2. 9 NH<sub>4</sub><sup>+</sup> and NH<sub>3</sub> distribution as a function of pH (AIRTON KUNZ, 2016)

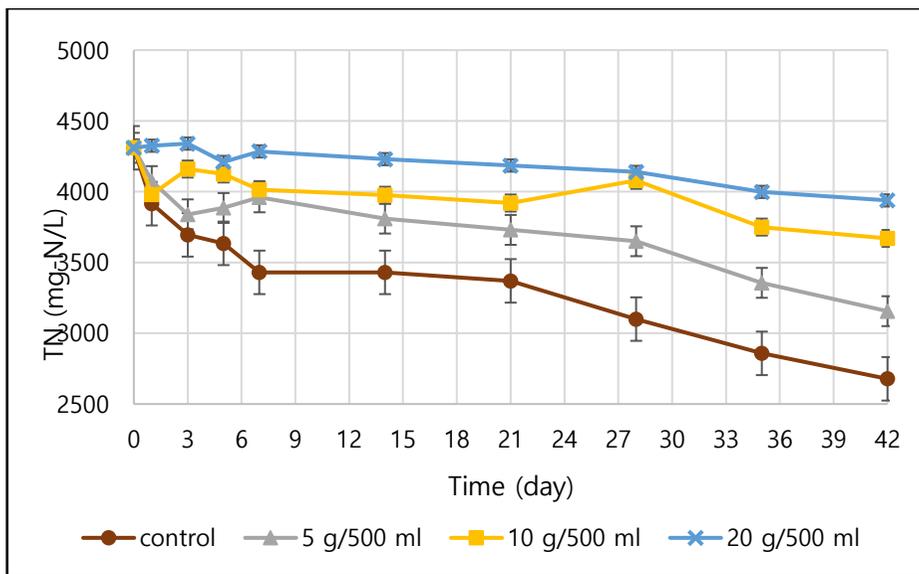


Figure 2. 10 TN changes with different dosage of BPA over time

### 2.2.4 $PO_4^{3-}$ change by adding different dosage of BPA

Fresh urine (control) had a  $PO_4^{3-}$  concentration of 35 mg/L which was constant throughout the experiment. When BPA was introduced,  $PO_4^{3-}$  level increased because  $PO_4^{3-}$  constituents in BPA gets dissolved in Urine. This can be observed easily in Figure 2.8 where the  $PO_4^{3-}$  concentration is increased with the BPA dosage. By adding 5, 10 and 15 g of BPA increased the  $PO_4^{3-}$  concentration to 165, 230 and 262.5 mg-P/L. For BPA added reactors, we can observe a slight reduction in  $PO_4^{3-}$  concentrations. Mg in Urine can react with  $PO_4^{3-}$  and precipitate as struvite. This is discussed in detail in the next chapter.

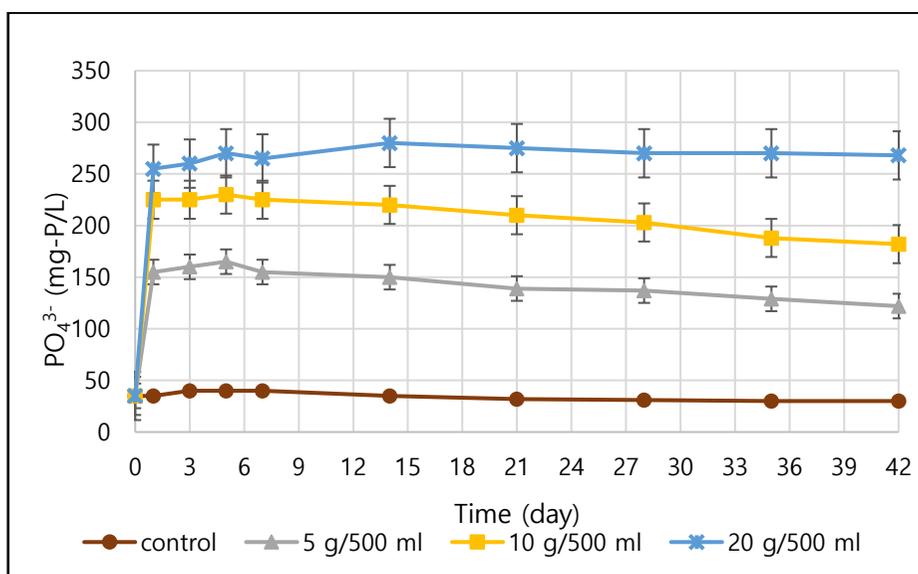


Figure 2. 11  $PO_4^{3-}$  changes with different dosage of BPA over time

### 2.2.5 $PO_4^{3-}$ adsorption test with different dosage of BPA

As mentioned in previous chapter, the pH will decrease during urea hydrolysis. Once urea hydrolysis begins, struvite production can start if the pH

is higher than 7 at room temperature (Doyle & Parsons, 2002; Tansel et al., 2018) However,  $\text{NH}_4^+$ ,  $\text{PO}_4^{3-}$ , Mg have to be present for struvite generation (Booker et al., 1999). BPA has a very low surface area therefore, the contribution of  $\text{PO}_4^{3-}$  sorption is negligible. Figure 2.12 illustrates the effect of BPA when introduced to  $\text{PO}_4^{3-}$  standard solution. By adding 1, 2 and 4 g of BPA increased the  $\text{PO}_4^{3-}$  concentration to 150, 205 and 248 mg-P/L, respectively. During the experiment time there was no reduction of  $\text{PO}_4^{3-}$  concentration.

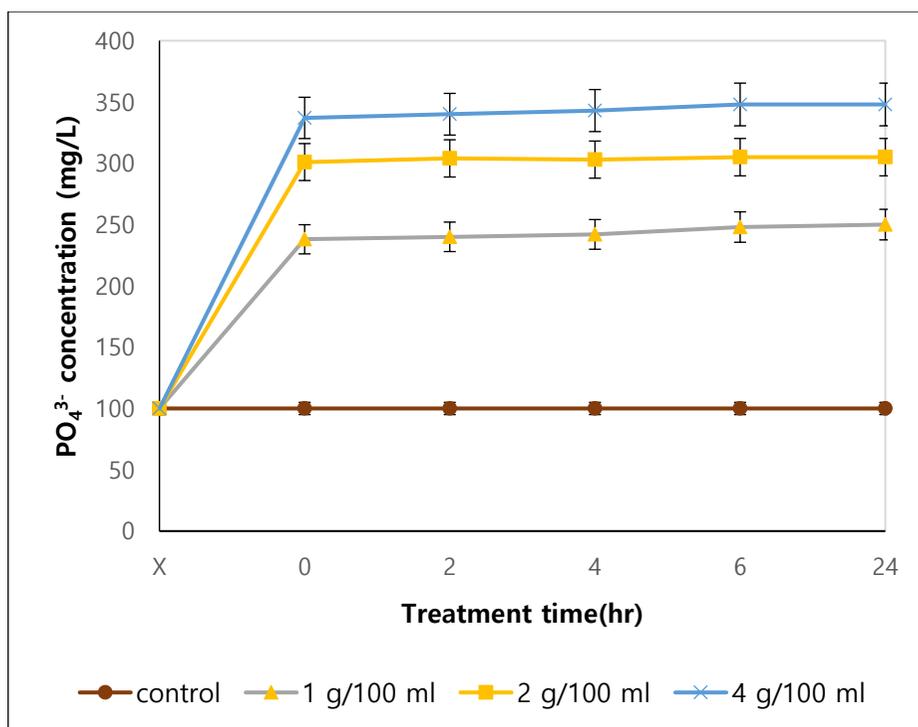


Figure 2. 12  $\text{PO}_4^{3-}$  changes with different dosage of BPA over time

### 2.3 Summary

Fresh urine was collected from a UDDT and BPA have the solution was mixed with magnetic stir at 350 rpm for 15 minutes (Ray et al., 2018). Four sets of reactors were prepared namely, the control (fresh urine) and three Fresh urine reactors with different BPA dosages. (5 g/500 ml, 10 g/500 ml, 20 g/500 ml) The mixed solution was kept at room temperature ( $25 \pm 3$  °C) for more than one month to observe the changes of N and P content over time.

Addition of BPA increased the pH of the aqueous medium as observed by many researchers. Without the addition of BPA, the pH increased to about 9 after 45 days from the initial pH of 6.14 because of urea hydrolysis. However, addition of BPA immediately increased the pH over 10.

Addition of BPA reduced the production of  $\text{NH}_4^+$ . A dosage of 5 g/500 ml reduced  $\text{NH}_4^+$  production by a factor greater than two during the first three days after which  $\text{NH}_4^+$  concentrations has risen due to the volatilization of  $\text{NH}_4^+$  as  $\text{NH}_3$  gas. The Dosage of 20 g/500 ml inhibited  $\text{NH}_4^+$  production as the pH was maintained above 11. Addition of BPA has reduced the TN loss by more than 1000 mg/L if a dosage of 20 g/ 500 ml is introduced. Urea hydrolysis create more  $\text{NH}_4^+$  in the aqueous medium thus, N can be lost because  $\text{NH}_4^+$  is easily volatilized as  $\text{NH}_3$  gas.  $\text{PO}_4^{3-}$  concentration increased with the BPA dosage. By adding 5, 10 and 15 g of BPA increased the  $\text{PO}_4^{3-}$  concentration to 165, 230 and 262.5 mg-P/L, respectively. For BPA added reactors, we can observe a slight reduction in  $\text{PO}_4^{3-}$  concentrations. Mg in Urine can react with  $\text{PO}_4^{3-}$  and precipitate as struvite.

# CHAPTER 3. INVESTIGATING P AND N RECOVERY BY STRUVITE PRODUCTION FROM SYNTHETIC WASTE WATER AND STORED URINE

## 3.1 Material and methods

BPA and its physical chemical properties are as described in chapter 2. SWW bottle was kept open so, CO<sub>2</sub> and CO may have volatilized which may have contributed to the higher pH. (Le Corre et al., 2009) This phenomenon may have caused the significant NH<sub>4</sub><sup>+</sup> loss. However, the cover of SU was closed to prevent N gas lose.

### 3.1.1. Stored urine (SU)

Fresh urine was collected from UDDT in building 35 and kept two months until the pH stabilized around 9. At this stage urea completely hydrolyze. Constitution of stored urine is shown in Table 3.1 and the urine tank used for storing urine is presented in Figure 3.1.

Table 3. 1 Initial physio-chemical characteristics of stored human urine

Parameter	Range of stored urine
Initial pH	9
Ammonium (mg-N/L)	2900
Phosphorous (mg-P/L)	242



Figure 3. 1 Storage tank used to Store urine

### 3.1.2 Synthetic waste water (SWW)

Constituents and the pH of Urine vary significantly from person to person. Therefore, it is hard to generalize the findings of chapter 2. Therefore, a proper chemical analysis is needed to verify the hypothesis of chapter 2. In light of this, authors have made synthetic wastewater by adding a known concentration of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$ . Firstly, standard solution of  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  were made following standard methods (환경보건기술연구원, 2017)  $\text{NH}_4\text{Cl}$  (Dae jung, Korea) was oven dried for one day at 120 °C, and 3.19 g was taken and mixed with 1 L of distilled water by magnetic stir to obtain a 1 g/L of  $\text{NH}_4^+$ .  $\text{KH}_2\text{PO}_4$  (Dae jung, Korea) was oven dried for one day at 120 °C, and 4.39 g of dried sample was mixed with 1 L of distilled water by magnetic stir to produce a 1 g/L solution of  $\text{PO}_4^{3-}$ . Then, 200 ml of standard  $\text{NH}_4^+$  solution

and 140 ml of standard Phosphate solution was mixed together with 160 ml of distilled water to make 500 ml of synthetic wastewater. Finally, NaOH was added to adjust the pH. The pH was measured continuously by the pH meter (DKK-TOA Portable pH meter HM-31P, Japan) and 1 M NaOH was added until the pH reached 9. Properties of average stored urine and synthetic wastewater used in the experiment is presented in Table 3.1.  $\text{NH}_4\text{Cl}$  was dry for one day in 120 °C oven and weighted 3.19 g accurately for mixed in 1 L of distilled water by magnetic stir and produced 1 g/L of  $\text{NH}_4^+$ .  $\text{KH}_2\text{PO}_4$  was dry for one day in 120 °C oven and weighted 4.39g accurately for mix in 1 L of distilled water by magnetic stir and produced 1g/l of  $\text{PO}_4^{3-}$ . To make 400 mg-N/L of  $\text{NH}_4^+$  and 280 mg-P/L concentration of 500 ml synthetic water each  $\text{NH}_4^+$  standard solution and phosphate standard solution taken 200 ml and 140 ml and mixed and diluted 160 ml of additional distilled water has been used. And the end of the stage to adjust pH NaOH. During magnetic stir is working pH measured continuously by pH meter (DKK-TOA Portable pH meter HM-31P) and NaOH 1M added drop by drop until it reaches target pH (9). Properties of average stored urine and synthetic waste water which used on experiment show as Table 3.2

Table 3. 2 Initial physio-chemical characteristics of average human urine and synthetic waste water

Parameter	Range of stored urine	Synthetic waste water
Initial pH	9 – 9.5	9
Ammonium (mg-N/L)	300 – 4000	400
Phosphorous (mg-P/L)	100 - 500	280

### ***3.1.3. Identifying the characteristics of precipitation***

The precipitation was characterized by separated with coffee filter (Sakthivel et al., 2012) for verifying the X-ray crystallography by a D 8 Powder X-Ray Diffractometry (XRD) (Bruker, Germany) and to observe the metallic components of precipitation X-ray crystallography by S 4 PIONEER X-ray fluorescence (WDXRF) spectrometry (Bruker AXS, Germany) was used. Finally, for elemental analysis, elemental analyzer by Flash 2000 (Thermo Fisher Scientific, Germany) was used

## **3.2. Results and discussion**

### ***3.2.1. The effect of BPA on pH of SWW***

Maintaining a high pH (pH > 9) is of paramount importance, because pH is related to the solubility of certain chemicals. For struvite production, optimum pH is 8.5 - 9.5 (Doyle & Parsons, 2002; Shih & Yan, 2016), and if the pH decreases below 7, crystallization of struvite is very low. Further, if the pH is above 11, calcium-based precipitation is dominant compared to magnesium-

based precipitation (struvite). When 5 g/ 500 ml dosage of BPA was added to synthetic waste water, the pH increased and remained almost constant at 10 with a marginal variation of  $\pm 0.3$ . Similarly, a dose of 10 g/500 ml increased the pH to 10.6 ( $\pm 0.2$ ), and a dose of 20 g/500 ml also increased the pH to 10.6 ( $\pm 0.1$ ). Doses above 10 g/500 ml seems to have no effect on the pH. All three samples demonstrated suitable pH conditions for struvite precipitation.

The pH of stored urine was stable around 9. Adding BPA increased the pH drastically and then stabilized around 10.7 to 11 within first day. From 2<sup>nd</sup> day onwards a slow and steady decrease in the pH was observed that may be due to precipitation. (Shih & Yan, 2016; Tilley et al., 2008)

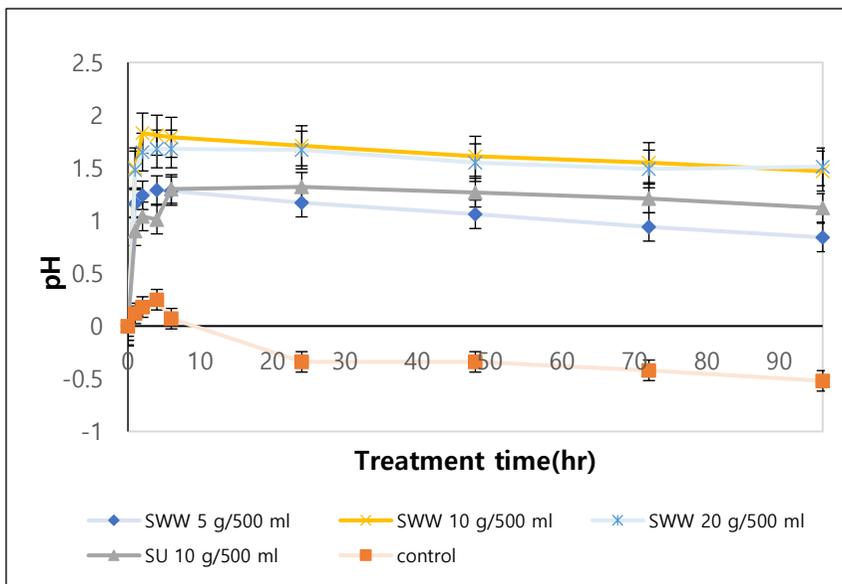


Figure 3. 2 Variation of the pH of SWW and SU with time

### ***3.2.2. The effect of BPA on $\text{NH}_4^+$ removal***

SWW and SU consisted of  $\text{NH}_4^+$  that can easily evaporate as ammonia gas ( $\text{NH}_3\text{-N}$ ). High pH can propel the production of  $\text{NH}_3$  from  $\text{NH}_4^+$ , that results in loss of Nitrogen. Reactor covers were opened while mixing using the magnetic stir, and during sample taking which can be attributed to Nitrogen loss. Some  $\text{NH}_4^+$  in the solution contributed to precipitation. Control sample lost a total of 43 mg-N/L of ammoniacal nitrogen, while the reactor with SWW 5 g/500 ml BPA dose lost 133 mg-N/L. Reactors with BPA doses of 10 g/500 mL and 20 g/ 500 mL lost 186 mg-N/L and for 176 mg-N/L, respectively. The  $\text{NH}_3\text{-N}$  loss was almost constant for doses above 10 g/500 mL with negligible variation, because the pH variation under these doses was minimal.

$\text{NH}_4^+$  concentration of SU decreased for few hours probably due to ammonia evaporation during sample taking. When BPA was added,  $\text{NH}_4^+$  concentration decreased faster in the beginning, due to the low pH conditions created by the addition of BPA. However, after the first day,  $\text{NH}_4^+$  concentration stabilized.

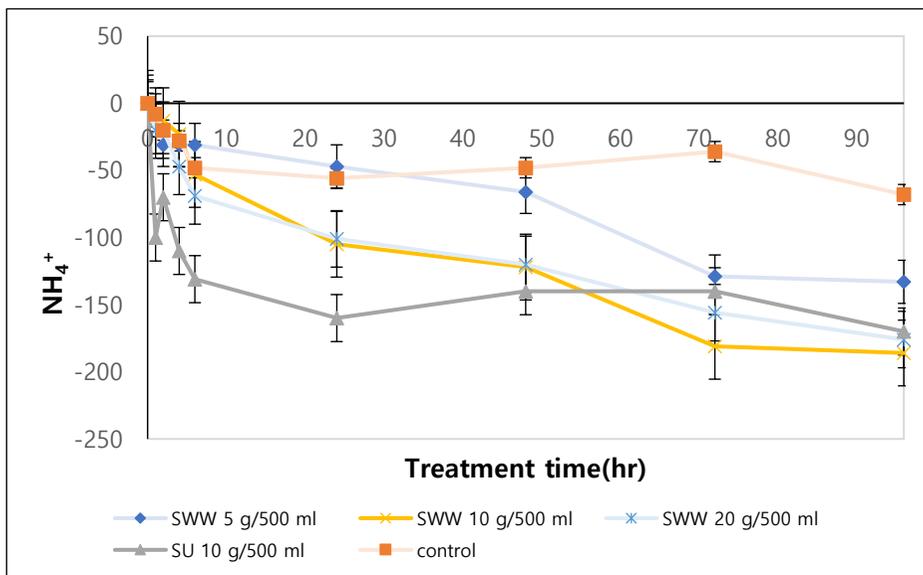


Figure 3. 3 Change in  $\text{NH}_4^+$  concentration of SWW and SU with time

### 3.2.3. The effect of BPA on phosphate removal

Total Phosphorus (TP) in stored urine was primarily present in the form of phosphate ( $\text{PO}_4^{3-}$ ). When BPA was added, Phosphate level increased drastically as BPA contained Phosphate that is readily soluble. However, after one-hour, Phosphate levels decreased because of struvite precipitation.

SU had a phosphate concentration around 230 g-P/L and when BPA was added, phosphate concentration immediately decreased to 170 g-P/L. SU has soluble magnesium that may contribute to struvite precipitation thus, less  $\text{PO}_4^{3-}$  removal in SU. After one-day,  $\text{PO}_4^{3-}$  concentrations stabilized.

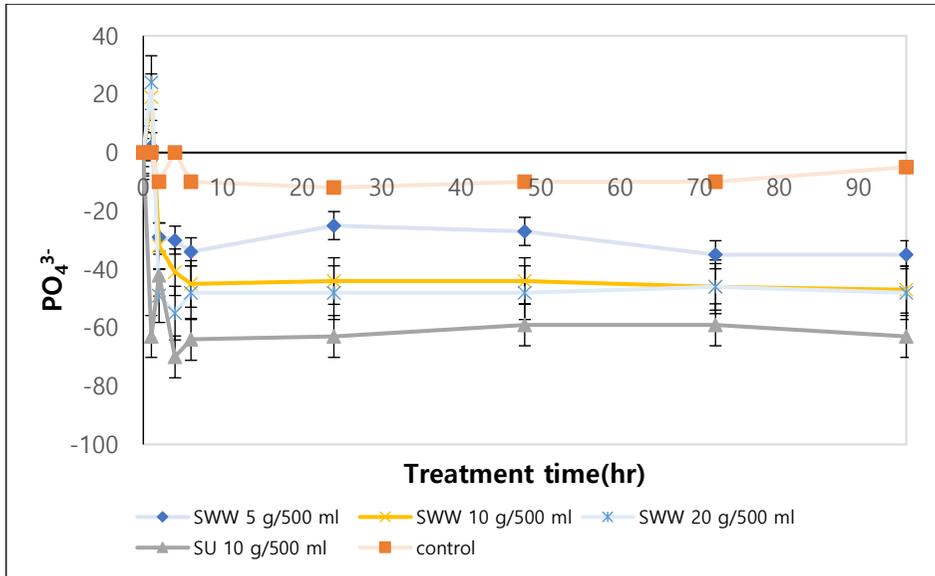


Figure 3. 4 Variation of Phosphate concentration of SWW and SU with time (for a total of 5 days)

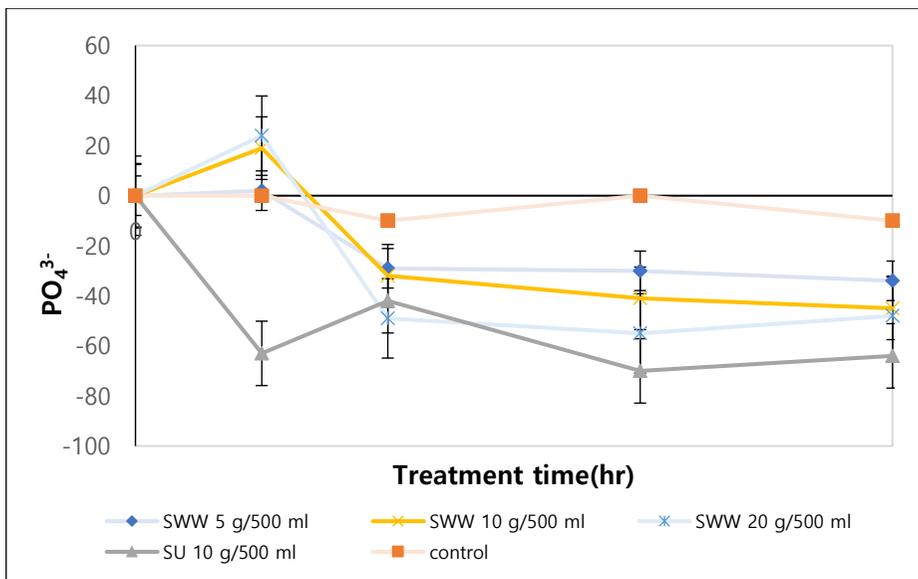


Figure 3. 5 Variation of Phosphate concentration of SWW and SU with time (for a period of 6 hours)

### 3.3.4. Precipitation analysis

Precipitation separated from urine was dried slowly and was analyzed using XRD. XRD result of precipitation is shown in figure 3.3 and it can be seen that BPA precipitation has a high similarity with struvite band pattern. This suggest that the precipitation induced by BPA is struvite and can be used as a commercial fertilizer. Composition of BPA is shown in Table 3.3 show that Ca, Mg, P, and N concentrations are higher after treatment due to crystallization and precipitation (struvite, etc.). The K concentration has decreased because of high solubility of potassium in water.

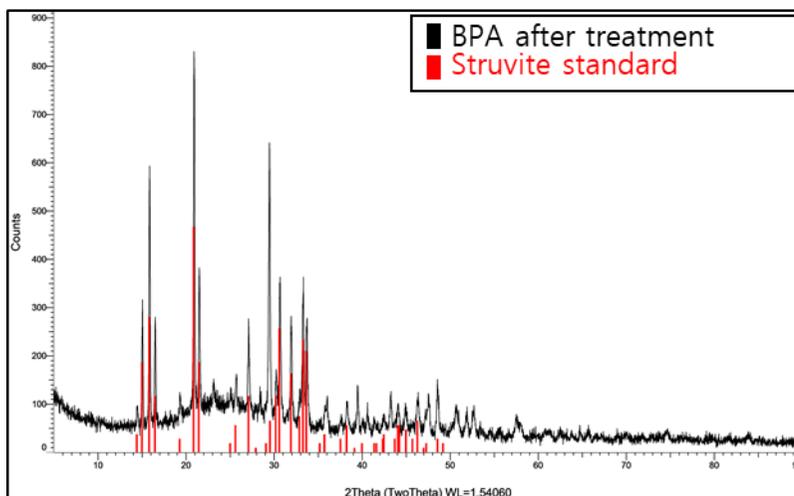


Figure 3. 6 XRD result of BPA precipitation after treatment

Table 3. 3 Composition of BPA before and after treating SWW and SU

Formula	BT*	AT*SWW	AT*SU
	Conc.(%)	Conc.(%)	Conc.(%)
Ca	4.274	18.804	14.63
Mg	0.76	3.847	6.09
P	0.585	3.648	5.127
N	0.3727	2.299	3.448
K	31.77	9.372	4.848

\* Before treatment \*\* After treatment

### 3.4. Summary

In this chapter, effect of banana peel ash (BPA) on nitrogen and phosphorus recovery from synthetic wastewater and stored urine through struvite precipitation was investigated. The BPA increased the pH and acted as a supplementary source of magnesium. This stabilized urine and reduced phosphate and nitrogen loss in human urine. The results showed that doses higher than 10 g/ 500 ml urine, can increase the pH above 11 and, no significant improvement in Nitrogen and Phosphate recovery can be attained. Nitrogen and Phosphate was reduced significantly compared to the control sample due to struvite generation. Therefore, a BPA dosage of 10g/500 ml could be recommended to recover nutrients in urine.

## **CHAPTER 4. SUGGEST BPA APPLICATION GUIDELINE FOR CONVERTING URINE INTO FERTILIZER**

### **4.1. Urine storage tank under the UDDT**

Previous chapters discussed the effect of BPA on source-separated urine. This chapter aims to provide guidance on how this could be applied in a real-life setting (in UDDTs). A sample calculation is provided assuming a daily urine production of 1.5 L/ person/day, and an average family has four members. Urine storage time was selected as three months.

#### ***4.1.1. Calculation***

##### **Tank volume calculation**

Firstly, the tank volume can be calculated assuming an average daily urine production of 1 – 1.5 L/person/day (Chang et al., 2013). Tank have three compartments (section 1, 2, and storage section) volume of each sections is derived as follows:

- Section A volume =  $0.5 \text{ m} \times 0.5 \text{ m} \times 0.9 = 0.225 \text{ m}^3$
- Section B volume =  $0.5 \text{ m} \times 2\text{m} \times 0.8 = 0.8 \text{ m}^3$
- Section C volume =  $0.5 \text{ m} \times 2.5 \text{ m} \times 0.8 \text{ m} = 1 \text{ m}^3$

##### **BPA bag design**

Nylon bag containing BPA was designed for easy handling with dimensions 40 x 40 x 5 cm. Two handles designed on top of BPA bag and 60 cm height of support structure was provided for better management. BPA mass required was derived as follows:

- Density of BPA =  $660 \text{ kg/m}^3$
- 1 BPA bag =  $0.4 \text{ m} \times 0.4 \text{ m} \times 0.06 \text{ m} = 9.6 \times 10^{-3} \text{ m}^3 = 6.336 \text{ kg}$
- Required BPA for total (section A & B)
  - =  $0.225 + 0.9 = 1.125 \text{ m}^3$  of urine
  - =  $22.5 \text{ kg (for } 10 \text{ g/500 mL) / } 6.336 = 3.55 \text{ (} \approx 4 \text{ bags)}$

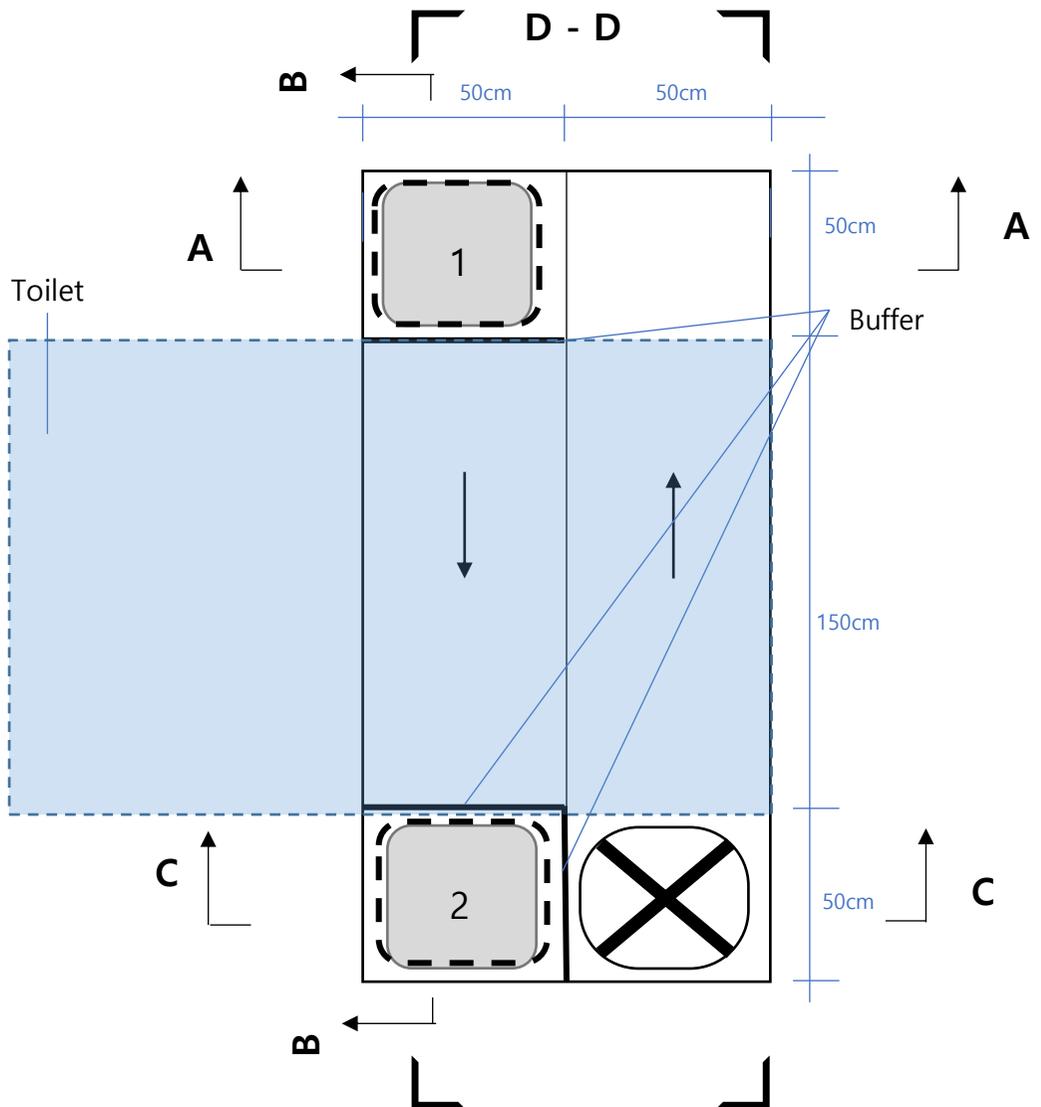


Figure 4. 1 Top view of urine tank under UDDT

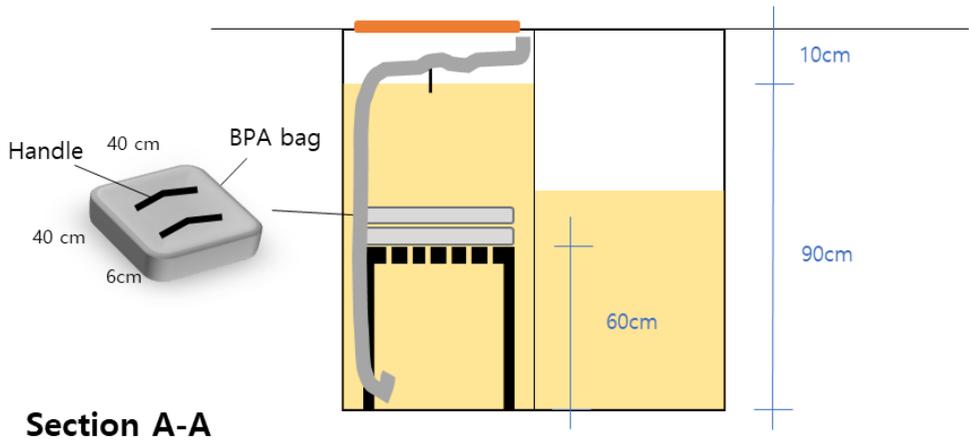


Figure 4. 2 Section A-A of urine tank under UDDT

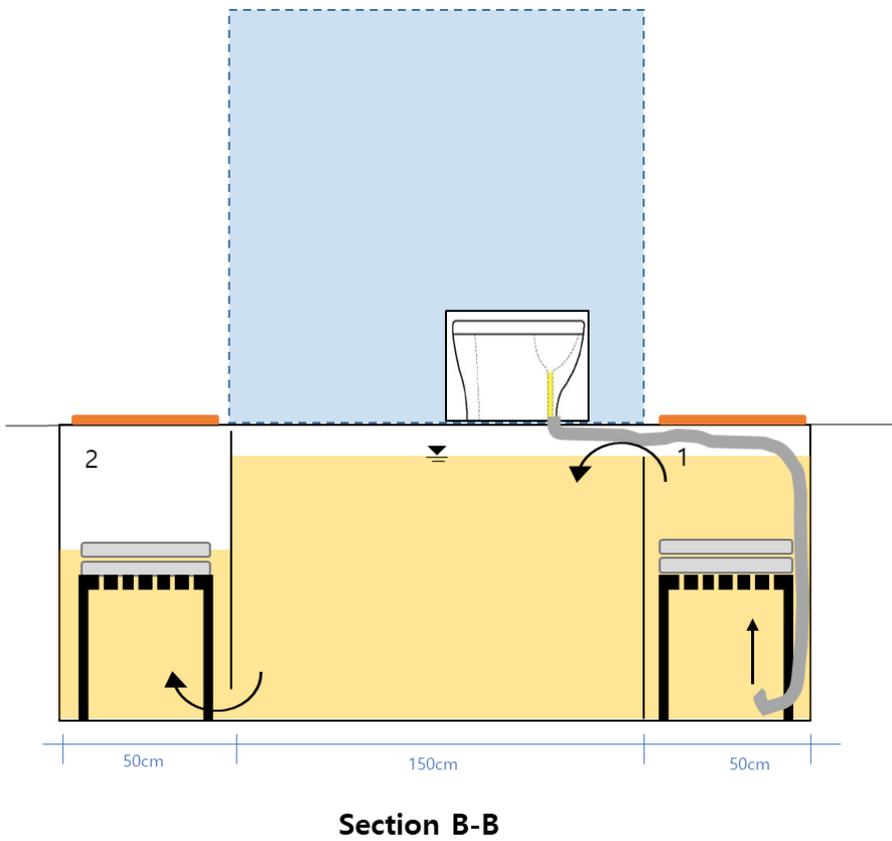
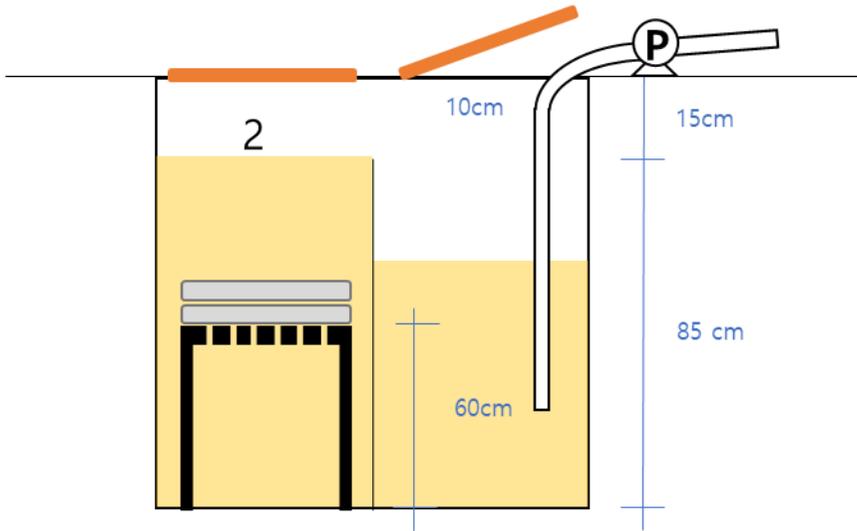


Figure 4. 3 Section B-B of urine tank under UDDT



**Section C-C**

Figure 4. 4 Section C-C of urine tank under UDDT

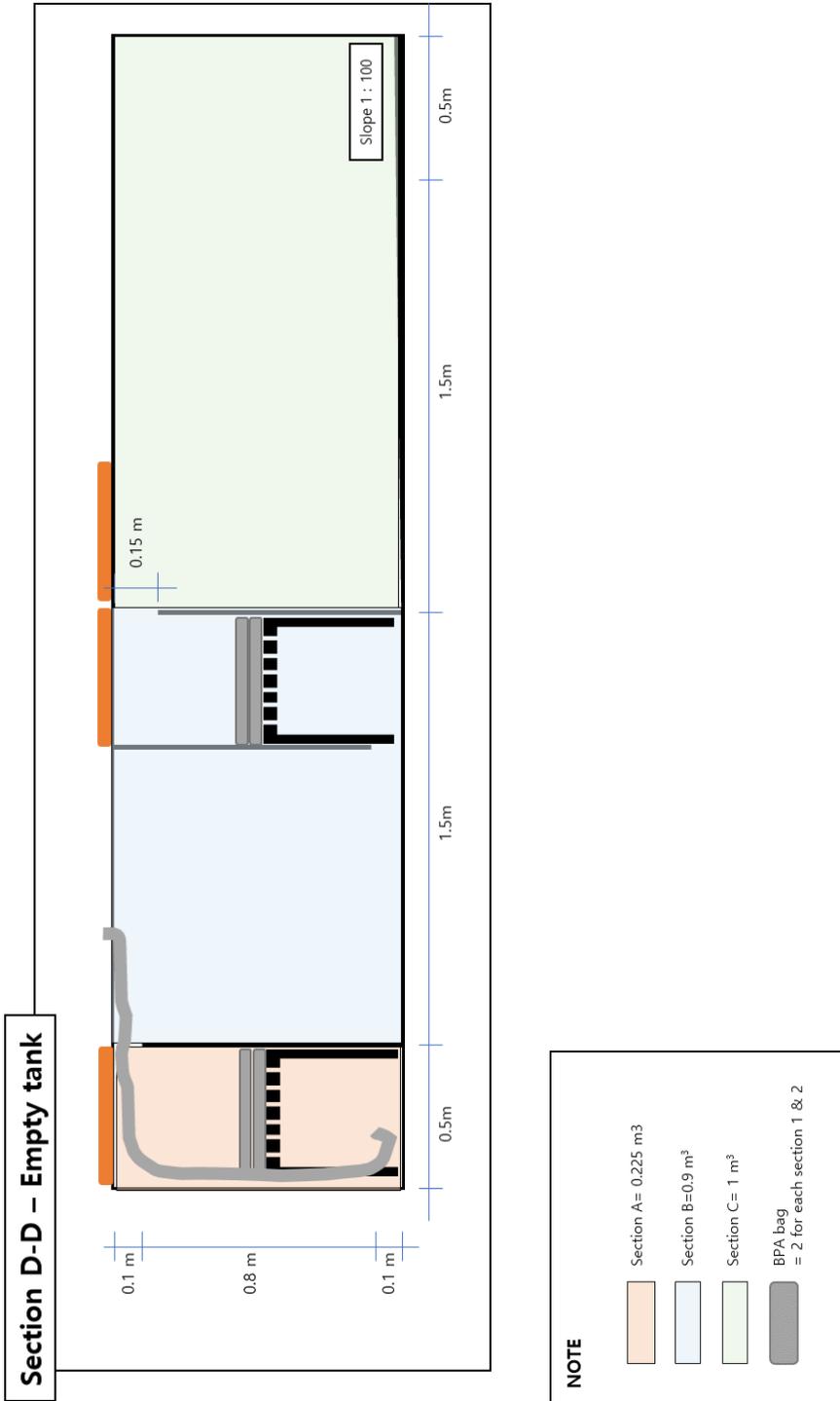


Figure 4. 5 Section D-D empty urine tank and each section

#### ***4.1.2. Simulation of system***

##### **Part 1. Inhibit urea hydrolysis**

In previous chapters we demonstrated that BPA could increase the pH of fresh urine and consequently, prevent urea hydrolysis. A dose of 10 g/500 mL was selected as the optimum dosage to delay the urea hydrolysis process for a period of around 1 month. In this chapter, we focused a 4-member family that produce 6 L of urine per day. For one month section A need a volume of 0.225 m<sup>3</sup> and in the first compartment, fresh urine will pass through BPA and the pH will increase. The high pH conditions instilled by BPA will prevent ammonia production thus, the odor generation can be minimized. After a month, urine start to flow over 0.9 m height buffer to the next section (section B).

Calculation and drawing is provided below:

- Section A =  $0.225 \text{ m}^3 / 0.006 \text{ m}^3 = 37.5 \text{ days (} \approx 1 \text{ month)}$
- Required BPA  
 $= 0.45 \text{ kg (for } 0.225 \text{ m}^3) < 6.336 * 2 = 12.672 \text{ ....(ok)}$

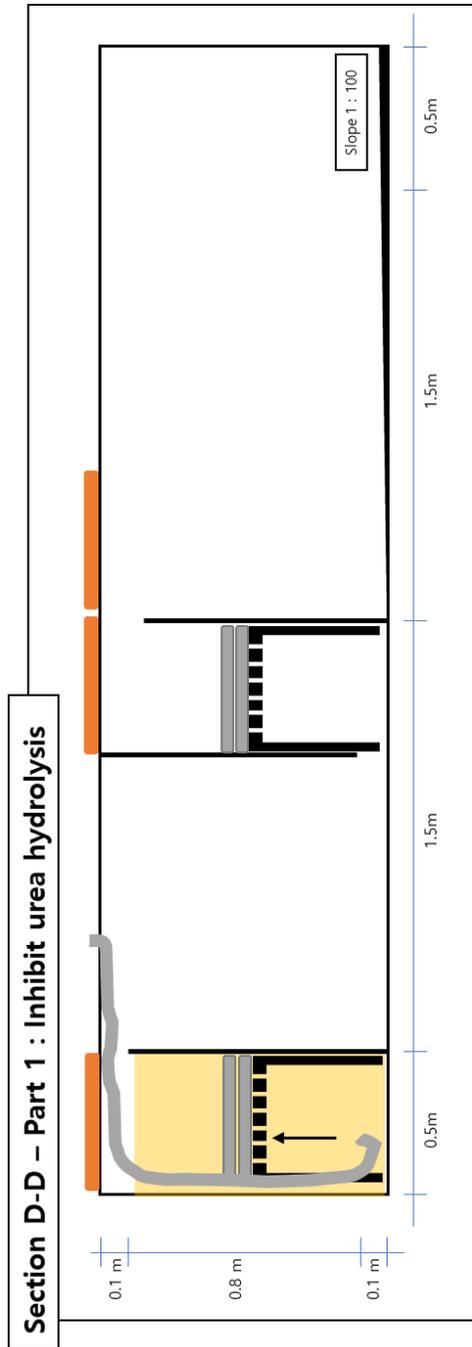


Figure 4. 6 Section D-D of urine tank (Part 1: Inhibit urea hydrolysis)

## **Part 2. Urea hydrolysis due to decreasing pH**

After urine is separated by the barrier between section A and B, urine level gets filled up to 0.18 m / month. During a period of three months, the pH of urine decrease below 10.5 and start the urea hydrolysis process again. This step conditions the composition of urine preferable for struvite precipitation.

Calculations and drawing are given below:

- Structure height / urine flowrate = time  
=  $0.6 / 0.18 = 3.3333\dots$  months

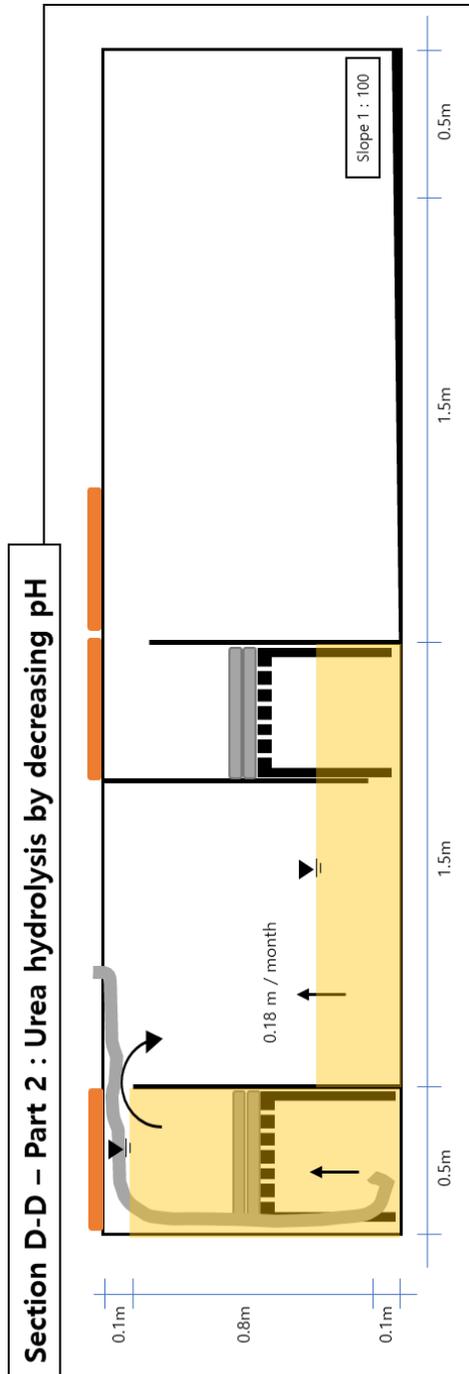


Figure 4. 7 Section D-D of urine tank (Part 2: Urea hydrolysis by decreasing pH)

### **Part 3. Struvite production**

In section B, if urine gets accumulated above 0.6 m, urine flow down to up through the BPA bags. Part of Mg and P dissolved in urine already so, once Mg, P, and N in urine react, struvite precipitate and gets attached on BPA. After urine in section B reach more than 0.8 m, it flows over the barrier to the next section (section C).

Calculation and drawing are as follows:

- Required BPA  
=  $0.225 + 0.9 = 1.125 \text{ m}^3$  (section A&B are considered)  
= 22.5 kg (for 10 g/500 mL)

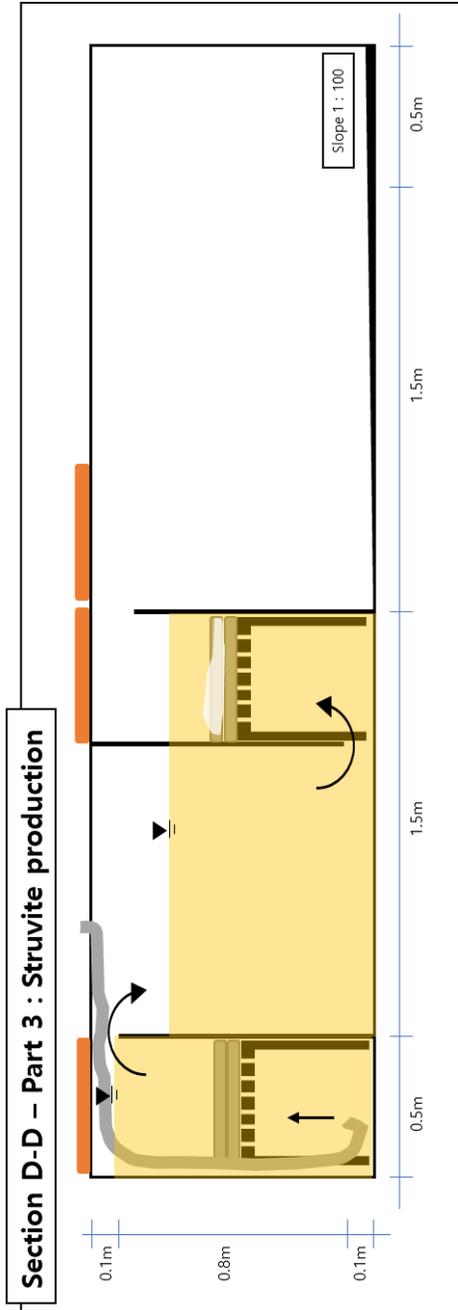


Figure 4. 8 Section D-D of urine tank (Part 3: Struvite production)

#### **Part 4. Storage**

After urine react with BPA, remaining urine contains high levels of N in the form of  $\text{NH}_4^+$  and additional K gets dissolved because of BPA. This is beneficial for chlorophyll in leaf of plants as Nitrogen and Potassium foster their growth. (Nations, 1984). This can be used as fertilizer and it is stored in section C.

Drawing of this section is provided below:

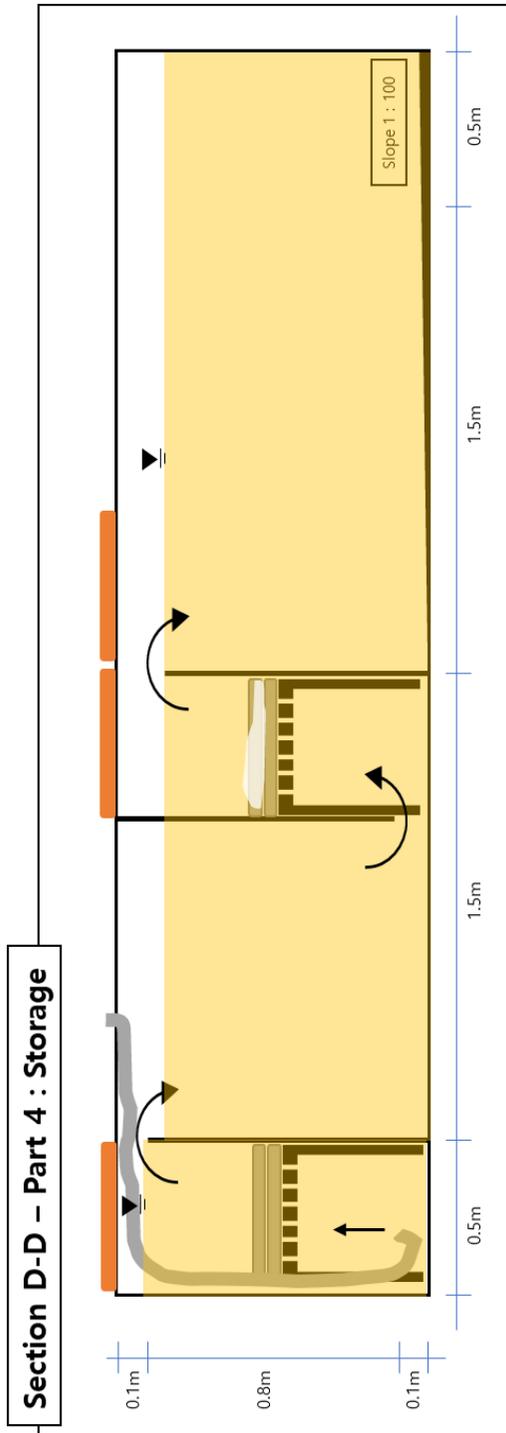


Figure 4. 9 Section D-D of urine tank (Part 4: Storage)

## **Collect fertilizer**

Two type of fertilizer can be obtained from this system namely, struvite fertilizer and N based urine fertilizer.

Firstly, 1 m<sup>3</sup> of N based urine fertilizer can be collected regularly every 6 months. Slope provided at the bottom assist the collection of urine.

Secondly, BPA from section B, around 12.67 kg of BPA and struvite fertilizer can be obtained along with urine fertilizer after 6 months. This can be done using local labor because of support structure under the BPA bag. BPA bag in Section A can be replaced by a new one and the old BPA bag can be re-used in section B for struvite production. Calculation and drawing is given below:

- Section C =  $1 \text{ m}^3 / 0.006 \text{ m}^3 = 160.67 \text{ days (} \approx 6 \text{ month)}$

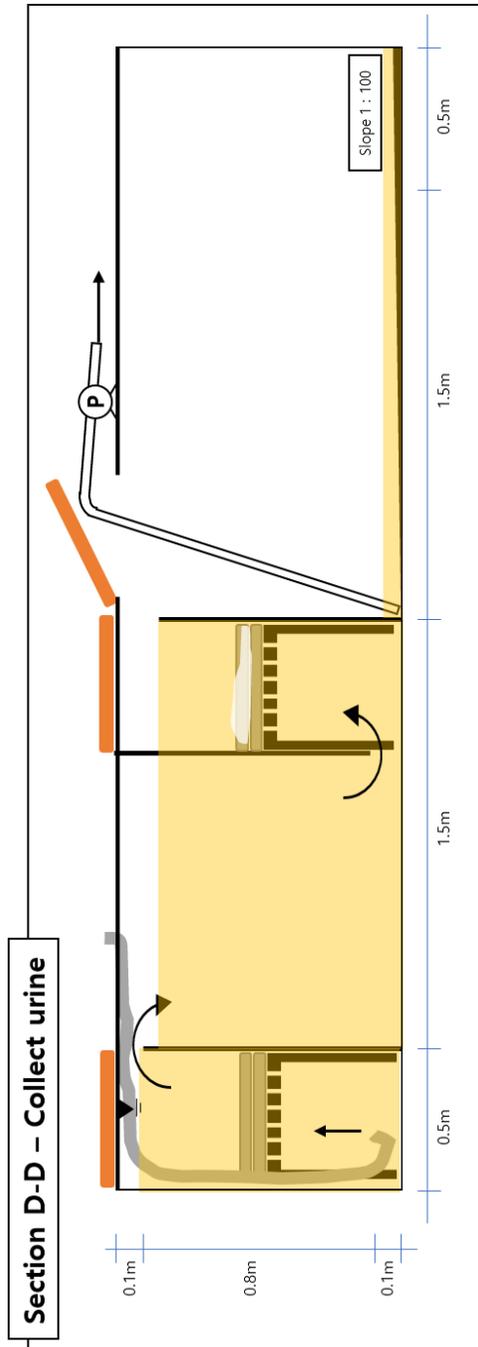


Figure 4. 10 Section D-D of urine tank (Collect urine)

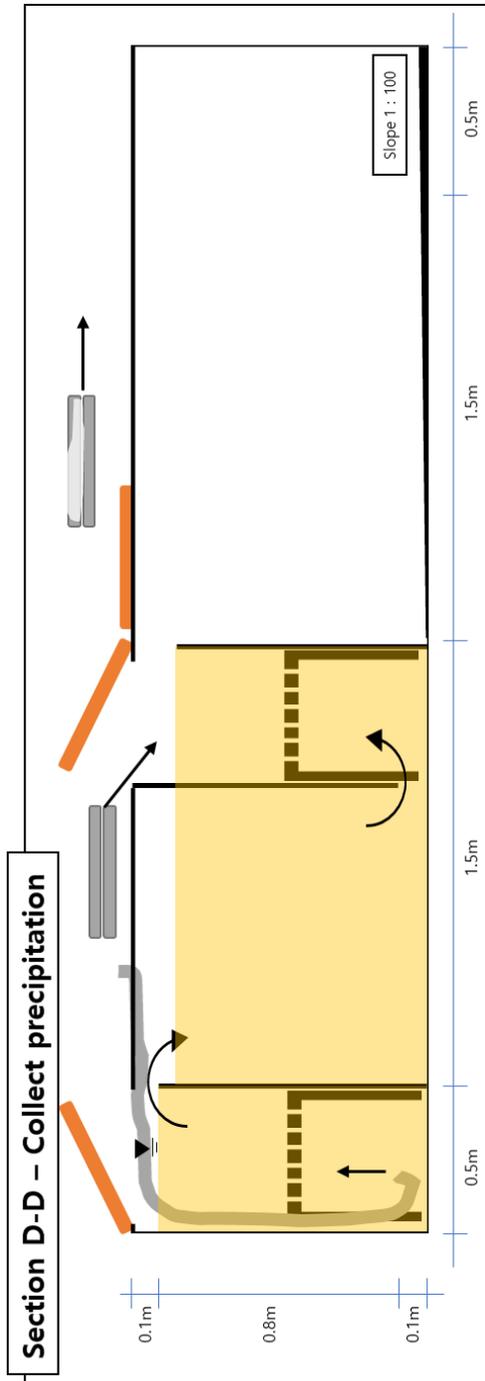


Figure 4. 11 Section D-D of urine tank (Collect precipitation)

## CHAPTER 5. CONCLUSIONS AND RECOMMENDATIONS

### 5.1 Conclusions

Human urine contains high quantity of nutrients such as Nitrogen and phosphorous. By adding BPA we can prevent the urea hydrolysis thus, minimizing the nutrient loss of fresh urine. When BPA was added to synthetic wastewater,  $\text{PO}_4^{3-}$  and  $\text{NH}_4^+$  levels reduced as Mg present in BPA reacted with  $\text{PO}_4^{3-}$  and  $\text{NH}_4^+$  to produce struvite. Further, similar results were obtained for stored urine as well. XRD analysis confirmed that the precipitate on BPA had a similar band pattern of struvite. Dosages above 10 g/500 ml, showed no significant improvement in nutrient recovery thus, a dose of 10 g/ 500 ml of urine is recommended for stabilizing stored urine. As BPA contained no potentially toxic metals, so, BPA treated urine can be directly used as soil fertilizer.

### 5.2 Recommendation for further study

Chemical composition of BPA may vary depending on banana species thus, performance of banana peel ash should be investigated for many species prior to generalizing the results for all banana species. Further, the mechanism of precipitation needs investigating as BPA may adsorb chemicals to the surface as well. The performance of the proposed system needs to be investigated after implementation for smooth operation. Finally, as only lab-scale experiments were performed, large-scale application should be investigated as well. Product also need further investigation prior to

administering. Further economic benefit associate with this system needs analysis.

## 국문초록

전 세계 인구의 30%가 물 공급을 제공받지 못하기 때문에 기본적인 화장실 시설을 이용하지 못하고 있다. UDDT(분뇨분리식 화장실)은 이러한 어려움을 극복하기 위해 도입된 화장실로 소변과 대변을 따로 모으는 건식 화장실의 일종이다. 사람의 소변은 농업에서 질소와 인의 좋은 공급원으로 사용될 수 있지만, 사용 전에 반드시 처리과정을 거쳐야 한다.

소변을 퇴비로 안정화 시킬수 있는 첨가제들이 개발되어왔지만, 개발도상국에서는 이러한 복잡한 과정과 정제된 물질의 활용이 현실적으로 어려운 환경이다. 이를 개선하기 위해 자연물을 활용한 개선책으로 나무재와 같은 물질이 연구 되었다. 나무재는 마그네슘 공급원으로 첨가되어 인산 회수를 위한 스트루바이트 생성에 활용할 수 있으나 침전물의 높은 중금속 농도로 인하여 비료로써 사용할 수 없다는 결론을 내렸다. 바나나는 값이 싸고 널리 사용가능한 자연 폐기물로 바나나 껍질 재(BPA)는 좋은 대안이 될 수 있다.

먼저 BPA 는 갖 배출된 소변을 안정화 시키는 역할로 조사되었다. 소변 10 g/500 ml 이상 용량의 BPA 를 첨가하면

산성도가 11 이상으로 상승하여 요소 가수분해가 억제되는것되었고 시간에 지남에 따라 소변내의 산성도가 10.5 미만으로 감소함으로 초기에 억제시킨 가수분해를 재개 시킬수 있다.

또한, 이 연구는 바나나 껍질 재가 인공 하수와 저장된 소변에서 스트루바이트 생성을 통해 질소 및 인산 회수에 미치는 영향조사에 초점을 맞췄다. BPA 농도를 10g / 500 ml 이상 초과하여 인공하수에 첨가하게 되면 10g / 500 ml 보다 질소 및 인산염 회수율에서 큰 개선을 보이지 않았다. 질소 및 인산염은 스트루바이트 생성으로 인해 대조군에 비해 현저히 감소되었다. 소변에 BPA 를 넣고 반응시킨 침전물의 XRD 분석 결과는 표준 스트루바이트 패턴과 유사한 패턴을 보였다. 따라서, BPA 가 소변의 영양분을 회수하는 데 사용될 수 있고 10 g/500 ml 이 가장 최적이라고 결론 내렸다.

저자는 이 연구의 결과에 기초한 혁신적인 화장실 설계를 제안하였다.

**주요어:** 분뇨분리식화장실; 소변처리; 영양회수; 스트루바이트;

바나나 껍질 재; 요소 가수분해

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