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Urine drying with ash and lime at temperatures 20 - 60°C - nutrient recovery from source separated urine

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ABSTRACT

Utilization of source-separated urine in agricultural production has been practiced and different treatment options have been developed in last two decades. The purpose of this study was to develop a drying technique for untreated human urine added to ash and lime as drying agent at temperature 20-60°C. A solid urine fertilizer was expected as final product that will be easy to transport and apply on agricultural field.

In this study, a mixture of ash and lime (1:1 by weight) was used as drying agent and human urine was applied as undiluted and fresh. The reason behind using ash and lime was to maintain a $\text{pH} > 10$ during drying process, which should inhibit urea hydrolysis in urine, and thereby urea should be retained in drying agent.

The drying technique was developed and drying capacity was quantified. Concentration of ammonia nitrogen was measured photometrically. A mass balance for nitrogen was obtained. It was evident from the experiment that urea can be retained by maintaining a high $\text{pH} (> 10)$. Bad drying condition due to reduced evaporation rate, flooding of urine over drying agent, blockage in airflow have been identified as factors regulating nitrogen loss and the concentration of nitrogen in the final product.

According to the experiment, urine drying at 20°C is not a feasible option, since rate of evaporation is very low. The highest retention of nitrogen (in the form of urea) at 35 and 60°C were 74% and 54% respectively.

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INTRODUCTION

Separate collection and treatment of human urine has opened up a new path for safe and sustainable recycling of nutrients in the environment. Urine has a good fertilizer value and it contains substantial amounts of plant nutrients in a smaller volume compared to faeces (flush water is included) and grey water (Jönsson, 1997; Johansson et al., 2001; Vinnerås, 2002). Besides, it is easier to handle pathogens and other pollutants when urine is treated separately from faeces and other fractions of wastewater (Höglund, 2001). Source separated human urine is already in use for agricultural production (Jönsson and Vinnerås, 2007). Use of treated urine in field as fertilizer contributes in two ways, prevention of environmental pollution from human waste and reducing the application of chemical fertilizer, which leads to more sustainable agriculture (Esrey, et al., 1998).

In the last two decades, a number of experiments have been conducted and different procedures have been developed for the treatment of source-separated urine. Storage, which is the most simple treatment procedure, involves use of considerable space and time for storing and large-scale transportation facility for carrying urine to agricultural fields. Alternatively, high-tech systems are required for treatment procedures other than storage that are expensive to install and maintain.

This study was intended to develop a low-tech system for drying of fresh human urine added to drying agent- that is the combination of sieved ash and slaked lime at temperatures 20 to 60°C, and to produce a solid urine fertilizer containing sufficient amount of nitrogen in form of urea, which can be used as an alternative to commercially available chemical fertilizer.

Aim

The aim of this study was to produce a solid nitrogen rich fertilizer from fresh human urine in combination with ash and lime by using a low-tech drying system.

Main objectives were:

- ★ To develop a system for drying urine with ash and lime as drying agent at temperatures 20°C to 60°C as a low-tech approach to process source separated urine.
- ★ To evaluate drying capacity of the system at different temperatures (20°C to 60°C).
- ★ To obtain a mass balance for nitrogen (N) in the system.
- ★ To evaluate the potential of the system in terms of recovery of nitrogen (N), phosphorus (P) and potassium (K) in dried urine and to compare between different treatments.

Hypothesis

Urine dried with ash and lime produces a solid fertilizer in powder form, which retains a high proportion of the inflow of nitrogen, so that it can be used as an alternative to commercial chemical fertilizer. Around 20% of input nitrogen can be lost during drying process.

BACKGROUND

Flush-and-discharge as a conventional sanitation approach

Flush-and-discharge has been considered as a good sanitation system for urban and peri-urban areas over the past hundred years. This system can work well in terms of cleanliness and reduction of bad odor. On the other hand, it is not a feasible option for countries, which have water shortage, economic inefficiency and lack of institutional capacity. In a flush-and-discharge system, 400-500 liters of urine and 50 liters of faeces are flushed away with 15,000 liters of pure water per person per year (Esrey et al., 1998). Usually, this toilet water is collected together with water from kitchen, shower, and laundry and also with storm water and highly polluted water from industries in the same pipe. A treatment plant supposed to be present at the end of pipe that will treat the polluted water and afterwards dispose of the treated water in open water. In most of the cases, especially in countries lacking economic and institutional capacity, there is no treatment plant at the end of pipe and the polluted water is directly disposed into surface water. That practice leads to water pollution, eutrophication and disease breakouts (Esrey et al., 1998).

Ecological Sanitation approach

Ecological sanitation (eco-san) is a closed-loop system where human excreta are treated as a resource instead of disposing it to the environment through the waste water system (Esrey, et al., 1998). Treated excreta are free of disease causing organism but rich in plant nutrients that can be applied in agricultural field to improve soil structure and to increase crop yield.

Conventional sanitation approaches dispose of the nutrients from human excreta and break down the nutrient loop where, the eco-san approach helps us to prevent pollution from the disposal of human excreta and to return nutrients to the soil (Esrey et al., 1998). While Eco-san is a recent term discussed in modern society, people of different cultures have practiced the recovery and use of human excreta over thousands of years especially for agricultural production (Esrey et al., 1998; Bracken et al., 2007; Muskolus, 2008).

Human urine: chemical composition and fertilizer potential

Human urine is a liquid by-product of the body that is constituted of mainly water (95%), urea, cations (Na^+ , K^+ , NH_4^+ , Ca^{2+}) and anions (Cl^- , SO_4^{2-} , PO_4^{2-} and HCO_3^-), creatinine and organic compounds (Kirchmann & Pettersson, 1995). Urine accounts for approximately 1% by volume of the total domestic wastewater flow but it is the dominating source of main agricultural nutrients, nitrogen (80%), phosphorus (50-55%) and potassium (60%) (Johansson et al., 2001; Vinnerås, 2002). Urine contains 80-90% of the nitrogen, 50-80% of the phosphorus and 80-90% of the potassium in the total food consumption (Berger, 1960).

At an average, 400-500 liters of urine is excreted by an adult per year, which contains 4.0 kg of nitrogen, 0.4 kg of phosphorus and 0.9 kg of potassium (Jönsson, 1997; Esrey et al., 1998). The new Swedish design value for wet mass and nutrient content of urine proposed by Vinnerås et al., (2006) is given in Table 1.

Table 1. New Swedish default values for excreted mass and nutrients (NPK) in urine proposed by Vinnerås et al. (2006)

Parameters	Urine (kg/person, year)
Wet mass	550.0
Dry mass	21.0
Nitrogen (N)	4.0
Phosphorus (P)	0.36
Potassium (K)	1.0

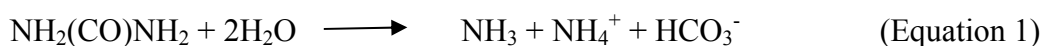
The main form of nitrogen in urine is urea (80%) and the remaining portion can be found as ammonia (7%), creatine (6%), shorter peptides and free amino acids (Lentner et al., 1981; Kirchmann & Pettersson, 1995). Phosphorus is mostly found as inorganic phosphates ($\text{PO}_4\text{-P}$) (>95%) and potassium mainly as free ions (K^+) (Berger, 1960; Lentner et al., 1981). Nutrients are found in highly plant available form and uptake by plant is essentially as good as chemical fertilizer (Kirchmann & Pettersson, 1995; Jönsson et al., 2004). On the other hand, the content of heavy metals (copper, zinc, chromium, nickel, lead and cadmium) in urine is very low compared to other categories of waste, for instance faeces, kitchen waste, farmyard manure and commercially available fertilizer and therefore, urine is considered a very clean fertilizer (Jönsson, 1997; Johansson et al., 2001; Vinnerås, 2002; Kirchmann & Pettersson, 1995).

Urine diversion

Urine diversion is the best way to recover resources in urine. In this system, urine is diverted away from faeces in a urine diversion toilet, then collected, and treated separately (Esrey et al., 1998). As urine is the most nutrient rich fraction of wastewater and contains only small amounts of heavy metals, it is possible to recover major proportion of nutrients from household wastewater if urine is collected separately (Vinnerås, 2002). Moreover, pathogen destruction is more efficient in source-separated urine than in mixed wastewater and the initial pathogen concentrations are lower, as pathogenic organisms are mostly excreted in faeces (Höglund, 2001).

Characteristics and degradation of source-separated urine

The pH of freshly excreted urine varies from 4.8 to 8.2 (Diem & Lentar, 1970; Lentner et al., 1981). The total nitrogen concentration in undiluted fresh urine ranges from 7-9 g/L (Guyton, 1986) which is mostly excreted as urea ($\text{CO}(\text{NH}_2)_2$). In a urine collection system, the urea is hydrolyzed by urease (urea amidohydrolase) producing bacteria and ammonia is produced (Jönsson et al., 2000; Udert et al., 2003). Consequently, the nitrogen in stored source-separated urine mostly exists as ammonia nitrogen (92-99%) and this process contributes to an alkaline pH (9-9.3) in urine (Eq.1) (Udert et al., 2003).



Due to this high pH in urine, phosphate, magnesium, calcium and ammonia become insoluble and precipitate as struvite (MgNH_3PO_4) and apatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) and form a sludge in the collection vessel (Jönsson et al., 2004).

Treatment options for source separated urine

Different techniques have been experimented and evaluated for processing and treatment of source-separated urine, particularly in laboratory scale.

Storage: the easiest way of sanitization

Though human urine is comparatively less affected by pathogens than faeces, it can contain some enteric pathogens like *Leptospira interrogans*, *Salmonella typhi*, *Salmonella paratyphi* and *Schistosoma haematobium* (Feachem *et al.* 1983). Moreover, there is risk of faecal contamination in source separating toilet. Therefore, handling and reuse of human urine involves hygiene risks and people can be affected by ingestion of urine and consumption of crops that have been fertilized with urine (Höglund, 2001).

Since storage of undiluted fresh urine leads to the production of ammonia and consequently raises the pH, it provides a hostile environment for microorganisms, which boosts the die-off of pathogens and thus reduces hygienic risks (Vinnerås, 2002; Schöning & Stenström, 2004). Urine can be used for fertilization directly when crop production is intended for household's own consumption, but in case of large-scale collection from different households and using it in agriculture, storage of urine is recommended for 1 to 6 months depending on the storage temperature and type of crop to be fertilized (Höglund, 2001). During storage of urine, volatile losses of nitrogen in form of $\text{NH}_3(\text{g})$ is a matter of concern but Jönsson *et al.* (2000) showed that the loss can be less than 0.5% in a well designed system. Acidification of urine was suggested by Hellström *et al.* (1999) to inhibit urea degradation.

Volume reduction of human urine

Since, nutrients in urine are very dilute compared to commercial fertilizer, huge volume of urine is needed to fertilize cropland and therefore long time storage and transportation become obstacles for management, energy efficiency and transportation facility (Jessen & Etnier, 1996; Hellström, 1998; Lind *et al.*, 2001). Therefore, concentrating of nutrients in human urine was suggested for ease of storage and transportation (Lind *et al.*, 2001; Behrendt *et al.*, 2002; Maurer *et al.*, 2006).

Different techniques have been used to reduce the volume and to concentrate urine. Dalhammar (1997) used reverse osmosis process, where maximum concentration factor of 5 could be achieved resulting in following nutrient recoveries in the retentate: ammonium 70%, phosphate 73% and potassium 71%. Lind *et al.* (2001) demonstrated that by freezing urine at a temperature of -14°C , approximately 80% of the nutrients could be concentrated in 25% of the original volume and the experiment was later validated by Gulyas *et al.* (2004). On a laboratory experiment, Mayer (2002) produced a viscous liquid by evaporating non-hydrolyzed urine at 200 mbar and 78°C temperature and the product contained 9.7% nitrogen by weight. In another laboratory experiment, Pahore *et al.* (2010) tested a volume reduction system based on water evaporation from a vertical gauge sheet and proposed a mathematical water transport model to evaluate the performance of the system. It was later applied to the dry climate in southern Pakistan, having an air temperature of $30\text{--}40^\circ\text{C}$ and air humidity of 20–40%, achieving an 80% volume reduction of 10 L of urine per day.

Other techniques for nutrient recovery

Udert et al. (2003a) performed batch experiments with precipitated solids and stored urine from a urine-collecting system to estimate the precipitation potential in the system and later simulated the result with a computer model and determined that struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) and hydroxyapatite (HAP, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) are the main precipitate compounds (Udert et al., 2003b). Kabdasli et al. (2006) conducted a laboratory experiment regarding struvite precipitation from enzyme hydrolyzed urine and proved it as an efficient process that provide up to 95% recovery of ammonia (complete urea hydrolysis was occurred by adding enzyme doses $25\text{--}49 \text{ mgL}^{-1}$). This process involved addition of magnesium salt and phosphate. Harada et al. (2006) developed an equilibrium model for estimating struvite precipitation and the model predicted that at $\text{pH} > 8.1$, 99% of phosphate in urine can be precipitated with 1.5 fold Mg concentration to $\text{PO}_4\text{-P}$.

Behrendt et al. (2002) showed that urea in urine can be converted to isobutylaldehyde-di-urea (IBDU) which is a slow release fertilizer; this process involves addition of large amount of chemical isobutyraldehyde (IBD) and extra processing for concentrating urine like evaporation or freeze-thaw prior to the actual production of IBDU. Pronk et al. (2006) conducted an experiment where electrodialysis with bipolar membranes was combined with an additional mass transfer unit in order to render a product containing ammonium and phosphate at a low pH.

Approaching to the intended experiment

Problems with huge space for storage and transportation of urine to agricultural land, high-tech processes and subsequent cost for recovery make it difficult to treat urine and recycle nutrients particularly in low and mid income countries. Hence, there is a need to develop such a treatment process, which requires minimal storage facility, little transportation and involves a cost-effective technology for processing of source-separated urine. Those reasons were the driving forces behind the intended experiment, which was to dry urine with ash and lime at temperatures of $20\text{--}60^\circ\text{C}$ together with maintaining a definite moisture level and airflow in drying chambers.

This experiment attempted to retain urea from urine in the drying agent by minimal hydrolysis occurring during application and drying of the fresh human urine in the drying chambers. The principle behind was the inactivation of enzymatic activity of urease by means of the high pH ($\text{pH} > 10$) of the drying agent (sieved ash and slaked lime). Kabdasli et al. (2006) reported in an experiment that no hydrolysis occurs in untreated human urine above $\text{pH} 10$. A solid fertilizer containing sufficient amount of nitrogen in the form of urea for agricultural application was expected as the final product of the experiment.

MATERIALS AND METHODS

Materials

Urine

Urine was collected every day from people aged from 25-35 years in sterile plastics bottles and applied in the drying box daily as undiluted and fresh. Urine from different donors was mixed before application and sample was taken from the mixed urine; sample represents 1% of the total applied urine in a specific day. Urine samples taken during 1st three weeks of the experiment were stored in one bottle and samples from rest of the experiment were stored in a separate bottle.

Apparatus and chemicals

Table 2. List of apparatus and chemicals used in the experiment

Apparatus/chemical	Specification	Producer/importer
Incubator	1. Nominal temperature- 100°C/212°F (temperature can be adjusted), 0.46 kW, 230 V, 2.0 A, 50/60 Hz	Binder, Germany
pH meter	pH electrode BlueLine 14 pH0-14/ -5..100°C/3mol/l KCl	Germany
pH stick	pH indicator strips (non-bleeding) pH 0-14	MerkKGaA, Germany
Spectrophotometer	Model- 4001/4, CAT- 4001-03	Thermo Electron Corporation, USA
Scale	Digital scale with 2 decimal Capacity-3000g	
Electronic stirrer	Analog vortex mixer 230 volt	VWR International AB, Sweden
Air pump	1. 60 l/h 2. 300 l/h 3. 108 l/h	China France China
Flow meter	1. 0.5-2.5 l/min 2. 5-30 l/min	Muurame, Finland Muurame, Finland
Hygro log	Internal temperature: -25°C to +85°C Relative humidity: 0-100% RH	Tinytag Gemini Data Loggers (UK) Ltd.
Beaker		
Pipette	10-100 µl, 1 ml, 5ml	VWR International AB, Sweden
Urine bottle		
Duran flask		
Sample bottle		
Test tube		
Drying box		
Syringe		
Pipe & joint	4 mm, 8 mm	Slangservice I Uppsala AB, Sweden
Table Salt	Salt (NaCl) min 99.8%	AB Hanson & Möhring, Sweden
Sulphuric acid (H ₂ SO ₄)		
De-ionized water		

Method

Experimental Design

Urine drying experiment was planned to run at three different temperatures, room temperature ~20°C, 35°C and 60°C with controlled airflow (1 L/min and 5 L/min) and controlled moisture content (relative humidity 70%). Desired amount of moisture was achieved by air pumped through a box containing sodium chloride (NaCl) solution (details are given below). A mixture of ash and lime was used as drying agent (drying agent preparation procedure is described in page 8). Exhaust air from urine drying box was passed through sulfuric acid solution that trapped ammonia (NH₃) from the air. Each treatment consisted of an air pump, a moisture control box, a urine-drying box and an acid trap. The experiment was planned to run for 6 weeks.

Rationale for choosing specific temperature and airflow

In this experiment, temperature and airflow were chosen as two important factors that can influence the urine drying system. Here, the selected temperatures were 20, 35 and 60°C and two different rates of airflow were used. We assumed that 20°C represents the average temperature for summer in Sweden or in many other countries in the northern hemisphere. The temperature 35°C represents tropical countries with hot and humid summer like Bangladesh, India etc. and 60°C represents the temperature that can be reached in solar toilets in tropical countries, where ambient air temperature assumed to be 35°C outside the toilet (Vinnerås, pers).

On the other hand, we assumed that in a dry toilet, the rate of airflow over the excreta drying bed is 1 L/min in general with typical ventilation, which is one of the specific airflow rates that we chose for the intended experiment. Alternatively, if the ventilation pipe in dry toilet is placed directly over the excreta drying bed, then it is expected that most of the incoming air would be directly passing over the drying bed. Then it is possible to have a higher rate of airflow, which can be five times higher compared to typical ventilation in dry toilet where ventilation pipe is not placed directly over the drying bed (Vinnerås, pers). That was the reason behind choosing 5 L/min as a rate of airflow in the intended experiment.

Preparation of moisture control box

The moisture control box contained solution of sodium chloride, which was prepared by adding table salt (NaCl) to de-ionized water to keep the air moisture content of 70%. According to Rockland (1960), saturated sodium chloride solution can maintain a relative humidity of 75% at 35°C temperature but the average value for relative humidity was 70% as it was measured during water loss test (pages 8-9).

The ratio of salt to water in the solution was 1:3 by weight; 250g of salt was mixed with 750g of de-ionized water (water and salt were measured by weight). All moisture control boxes were sealed with silicon and each box had an inlet for incoming air coming and an outlet for passing the air to urine drying box.

Preparation of drying agent

The drying agent was prepared by mixing ash and lime and the ratio was 1:1 by weight. Prior to use in the experiment, wood ash was sieved by using a sieve having 1 mm opening. Bigger particles were separated and fine ash was used in the experiment. The density and pH of sieved ash was measured. Slaked lime ($\text{Ca}(\text{OH})_2$) (Calcium hydroxide > 96%) produced by the company MerckKGaA, Germany(www.merck.de) was used in the experiment. (Detail specification is given in Appendix 1). The mixture was supposed to act as a buffer during the drying process and thereby to maintain the pH 10 or higher, since addition of fresh urine is likely to lower the pH every day.

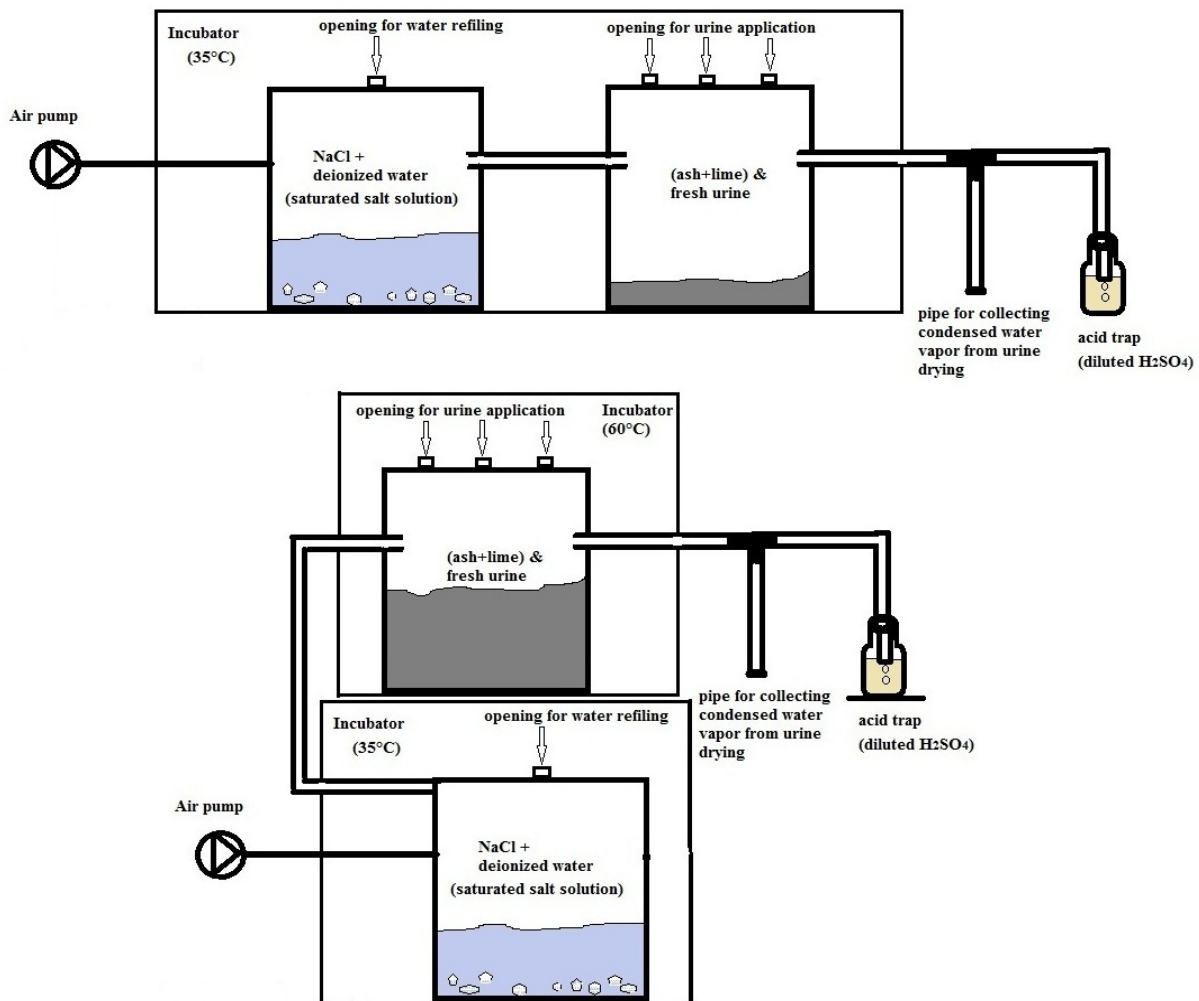


Figure 1. A diagram showing simplified system design for urine drying at 35 and 60°C (20°C version did look like 35°C treatment as it was implemented in the experiment).

Water loss test to measure urine drying capacity of the system

With intention to find out urine drying capacity of the system, a water loss test was carried out with water in the drying unit (box) without any drying agent and urine added. Two incubators were set at two different temperatures, 35°C and 60°C. Four treatments were set at three

different temperatures, one in room temperature (approximately 20°C), one treatment in 35°C and other two treatments in 60°C. During this test, all the treatments were set up with an air pump, a moisture control box and a drying box containing only water. In case of the two 60°C treatments, the moisture control boxes were placed in 35°C (Table 3). Water loss test was carried out for four weeks. Amount of water evaporated per day from the system was measured and that was considered as the urine drying capacity of the system. Relative humidity was measured by using a hygro log during the water loss test and it was 70% inside the drying box.

Table 3. System specification for water loss test (without drying agent and urine)

Treatment	Temperature for moisture control box (°C)	Temperature for drying box (°C)	Airflow (L/min)	Water loss (ml/day)
T-1	35	60	5	223
T-2	35	60	1	124
T-3	35	35	1	23
T-4	20	20	1	7

Urine drying

It was evident from the water loss test that drying capacity is too low for T-4 (7ml) (T-4 should be read as Treatment-4), where the drying box was in room temperature (20°C) and the rate of airflow was 1 L/min (specific water loss from T-4 is given in result section). Due to such a low drying capacity it would not be reasonable to run the urine drying experiment at 20°C. Therefore, T-4 was abandoned and did not carried out during the actual urine drying experiment.

The urine drying experiment was started with six drying boxes and three different treatments, one at 35°C and two others at 60°C (with different airflows) but in all cases moisture control boxes were set in 35°C temperature. Each treatment had two replicates. Each treatment was consisted of an air pump, a moisture control box, a urine-drying box and an acid trap (Fig.1). All the boxes were completely sealed with silicon except the inlets and the outlets.

Amount of urine applied in the system per day was based on drying capacity of the system that was measured by the water loss test. Amount of urine applied per day was 20% less than the drying capacity. During the experiment, no urine was applied in weekends; therefore, daily urine application rate was adjusted, so that the total amount of urine applied in 5 days equaled the total amount if urine applied per week. Amount of drying agent used in a treatment was five times by weight of the average daily application rate of urine over the week in that specific treatment, i.e. five times the weekly amount divided by seven (Table 4).

Table 4. System specification for urine drying experiment

Treatment	Temperature for moisture control box (°C)	Temperature for urine drying box (°C)	Airflow (L/min)	Urine application (5 days/week) (ml)	Amount of drying agent (ash + lime) (g)	Estimated exchange of air volume (times/hour)
T ₁	35	60	5	250	900	200
T ₂	35	60	1	140	500	30
T ₃	35	35	1	26	95	20

The system was designed in a way (Fig. 2) that it got a turbulent airflow and air passes over the largest possible space inside boxes. Based on free air space inside drying boxes and specific airflow rate, exchange of air volume in the drying box was estimated for treatments. As the volume of drying agent and rate of airflow (1 L/min) were low for T₃, it had large space for airflow inside the box, so it got only 20 exchanges of air volume. T₂ differs from T₁ in air space and that made the air exchange higher. For T₃, we had the lowest free air space inside the box and a very high rate of airflow (5 L/min); therefore, we got 200 exchanges of airflow for T₃ that was very turbulent.

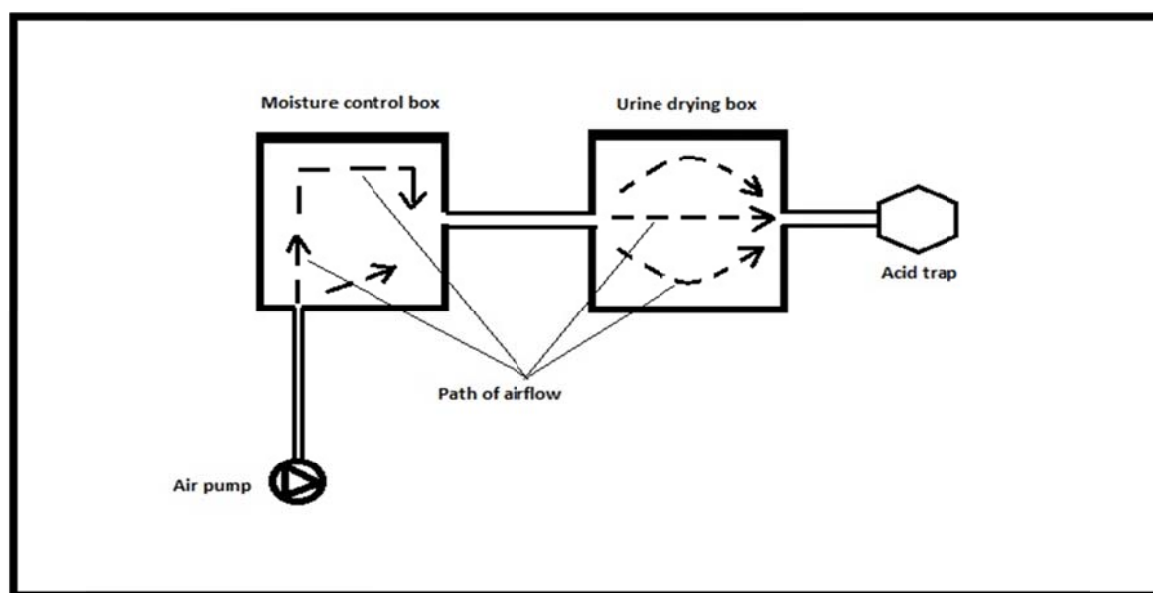


Figure 2. Schematic view from the top of the system showing path of airflow

Preparation of acid trap for NH₃

Sulphuric acid solution was made and placed into a duran flask. That was used as the acid trap to capture ammonia in outgoing air from drying box. Six acid traps were prepared for six boxes. The total volume of acid solution in a duran flask was 300 ml. A detail about acid trap preparation is given in Appendix 2.

Additional changes in the experiment

As the experiment was started, it was observed that all the acid traps were increasing by volume since they started to receive evaporated water from urine drying. There was a chance of spilling acid solution out of the bottle that contains acid. Therefore, T-connections were used to install pipes before the acid traps (Fig.1), which could retain a part of water vapor before it entered the acid trap. The pipes with T-connections were installed for all treatments, so that those could be removed every day for collecting condensate from urine drying and then connected back.

On third week of the experiment, T₂R₂ (it should be read as Treatment 2-Replicate 2) became moist and consequently urine flooded over the drying agent. It was stirred well, sometimes urine was not applied and it was observed if the treatment gets back good drying condition.

During the fourth week, the experiment was paused for whole week (for personal reason) that means the system was running but no urine was applied to any treatment. It is needed to mention that T₂R₂ did not recover; so, the box was cleaned and refilled with ash and lime that means T₂R₂ got a new start and afterwards urine was applied according to the capacity of system.

On the fifth week, T₁R₂ and both replicates of treatment 3 (T₃R₁ and T₃R₂) became moist and the material was mixed well to facilitate the drying process. As mixing did not help, some more drying agent was added to those treatments. For T₃R₁ and T₃R₂, the amount of drying agent was doubled, which means 95 g more (47.5 g ash and 47.5 g lime) were added to those two replicates and for T₁R₂, 300 g more (150 g ash and 150 g lime) was added in addition with the old material. Afterwards, urine loading was reduced by 50% for T₁R₂.

On the seventh week, all the six treatments became moist especially T₁R₂ and T₂R₂ and therefore, urine loading rate was reduced by 30% and materials were mixed well in all the boxes. Reduced loading rate and mixing did help to improve the drying condition of treatments except for T₁R₂, T₂R₁ and T₂R₂, so, 100g, 150g and 150g more drying agent was added to T₁R₂, T₂R₁ and T₂R₂ respectively.

At the end of seventh week, it was decided to run the experiment for 1 week more. On the eighth week, 50g drying agent was added to T₃R₁ and T₃R₂. So, the experiment was run for 8 weeks in total including 1-week break (urine was applied for 7 weeks).

Table 5. Summary of additional changes during the experiment

Treatment repetition	Changes occurred during the experiment										
	Day-14	Day-15	Day-22 to 28	Day-29	Day-31	Day-33	Day-38 to 40	Day-43	Day-45	Day-48	Day-39
T ₁ R ₁			The experiment was paused that means the system was running but no urine was added to any treatment					All the treatments became moist and urine loading was reduced by 30% for all treatments			
T ₁ R ₂				Became moist	300g drying agent was added	Urine loading was reduced by 50%	No urine applied		100g drying agent was added		
T ₂ R ₁										150g drying agent was added	
T ₂ R ₂	Became moist	No urine applied		Replaced with new drying agents							
T ₃ R ₁		Became moist			95g drying agent was added						50g drying agent was added
T ₃ R ₂		Became moist									

Analytical procedure

Measurement of pH

The pH of fresh urine and dried urine were measured throughout the experiment. In case of fresh urine, pH was measured every day before application, and for dried urine pH was measured every second week during the experiment.

Measurement of $\text{NH}_4^+ - \text{N}$

Ammonium test kit was used to prepare samples for measuring ammonium nitrogen ($\text{NH}_4\text{-N}$) in urine, dried urine fertilizer, acid solution and in condensed water vapor (from urine drying) by using a spectrophotometer. The method is analogous to EPA 350.1, US Standard Methods 4500- $\text{NH}_3\text{ D}$, and ISO 7150/1.

Fresh urine samples were diluted with deionized water to be compatible with the measuring range of ammonium test kit. The pH of the diluted samples was checked and it was within the range (according to ammonia test kit it should be within the range of 4-13). The amount of urea in diluted samples was estimated and 5000 units of urease enzyme was added per gram of urea to those samples (urease was in the form of powder and was mixed with deionized water to make urease solution). Prepared samples were shaken overnight for complete breakdown of urea by urease. After that ammonia test kit and spectrophotometer were used to measure $\text{NH}_4\text{-N}$ in samples. For dried urine fertilizer, the procedure was the same as for urine, except that the pH of dried urine samples was very alkaline ($\text{pH} \geq 13$) and so, it was adjusted by adding diluted hydrochloric acid (HCl). Samples from acid trap and condensate were also analyzed by ammonia test kit. It is important to note that the pH of acid solution sample was very low ($\text{pH} \leq 2$) and therefore, it was adjusted by adding diluted sodium hydroxide (NaOH).

Sampling

Samples were taken from fresh urine, dried urine, condensate and acid trap for Total Ammonium Nitrogen analysis; it is needed to mention that for fresh and dried urine samples were taken after urea hydrolyzation (TAN+u). For dried urine, samples were taken randomly to ensure unintentional sampling from treatments; each drying box was divided in 16 imaginary plots and 4 of those were chosen by using simple random sampling (lottery) to take samples for analysis.

Table 6. Sampling and number of measurement throughout the experiment

Type of analysis	Number of sampling occasion	Number of sample in each occasion	Total number of measurement throughout the experiment
pH- fresh urine	35	1	35
pH- dried urine	3	2/box, in total 12 for 6 boxes	36
TAN+u- fresh urine	3	5/bottle, in total 10 for 2 bottles	30
TAN+u- dried urine	3	4/box, in total 24 for 6 boxes	72
TAN- acid trap	3	2/bottle, in total 12 for 6 bottles	36
TAN-condensate	3	2/bottle, in total 12 for 6 bottles	36

Calibration of TAN values by using acid and drying agent

As there were some strange results in the mass balance for some treatments (more output than input), it was decided to conduct photometric analysis only with acid solution (with no added ammonia from urine drying) and another photometric analysis only with ash and lime dissolved in water (no urine added) by using ammonia test kit. The calibration was done to determine if there was any influence of acid and drying agent on photometric analysis of acid trap samples and dried urine samples.

Acid solutions were prepared according to the concentration of acid that was contained in the respective acid traps in different treatments after the experiment was completed. In both cases (acid and ash+lime) the solutions gave some absorbance in the spectrophotometer (Tables 15 and 16). In case of acid solution, the samples were diluted 100 times and the mixture of ash and lime was diluted 1000 times before analysis.

Errors associated with dilutions

Photometric analysis of dried urine samples gave higher values for TAN concentration when the samples were diluted 1:1400 but the value was lower when the samples were analyzed after diluting 1:800; it also happened for acid trap samples and condensate samples. We assumed that high possibility of errors, uncertainties are associated with high level of dilution (considering human errors and uncertain distribution of particles & ions in the solution), and false high values can make our calculation wrong. Therefore, results obtained from analysis at lower dilution level were, when possible, used in this study. It was followed for all kind of samples and dilutions were in every case kept low as much as possible within the range of ammonia test kit.

RESULTS

Water loss test & estimated urine loading rate

Here is the data measured during water loss test (Table 7) for four treatments representing four different drying conditions. Total daily water loss (from the whole area of a box) was highest in T-1 (223 ml), where the drying box was placed in an incubator with 60°C temperature and provided with 5 L/min airflow and the loss was lowest for T-4, where the drying box was in room temperature (20°C) and airflow was 1 L/min.

The daily urine-loading rate to be used was estimated from the water loss test. Aimed at the actual urine drying experiment, it was decided to keep the daily urine loading rate as 20% less than the actual daily drying capacity of the system, so that there would not be any risk of urine stored in the system and it was important to produce a completely dried urine fertilizer. As mentioned in the materials and method, total urine load per week was divided by 5 to get daily urine load, because urine was applied 5 days per week. Daily load (5 days/week) was 250 ml for T-1, 140 ml for T-2 and 26 ml for T-3.

Table 7. Estimated daily urine loading rate in different treatments

Treatment	Temperature of moisture control box	Temperature of drying box	Airflow	Water loss	Total water loss from the drying box	80% of average daily water loss	Total urine load in a week	Urine load (when applied 5days/week)
	(°C)	(°C)	(L/min)	(L/m ² ,day)	(ml/day)	(ml)	(ml)	(ml/day)
T-1	35	60	5	6.20	223	179	1250	250
T-2	35	60	1	3.40	124	99	695	140
T-3	35	35	1	0.64	23	19	130	26
T-4	20	20	1	0.20	7	*	*	*

Urine drying experiment

Characteristics of collected fresh urine

The average pH of urine (mixed urine from different donors) applied to the system was 6.1 (Table 8). The range of pH of every day urine mixture was 5 to 7.5. Fresh urine samples were collected in different bottles for 1st half and 2nd half of the experiment and total ammonia (TAN) was measured after enzymatic degradation with urease (TAN+u) and it was 6.84±0.02 g/L and 8.24±0.09 g/L of wet mass of urine (Table 8) respectively.

Table 8. Measured characteristics of collected urine

Parameter	Value
Average pH	6.1 (range: 5 to 7.5)
TAN+u	6.84 g/L (week 1-3.5, 1 st half of the experiment)
	8.24 g/L (week 3.5-7, 2 nd half of the experiment)

Nitrogen loading through fresh urine application

Volume of fresh urine applied to every single treatment was recorded everyday and summed up at the end of experiment to get the total volume applied throughout 7 weeks. Nitrogen (in form of urea) input in the system is directly related to the nitrogen concentration in fresh urine and the total volume of urine applied in the system. Total volume of urine applied for 1st and 2nd half of the experiment was multiplied with respective TAN+u values and added together to get the TAN load for every treatment. TAN+u load was highest for T₁R₁ (63.51g) as maximum amount of urine was applied in that treatment and was lowest for T₃R₁ (6.22g) (Table 9).

Table 9. Total TAN+u load in different treatments due to fresh urine application

Treatment	Expected volume of urine to be applied (L)	Total volume of urine applied (L)	Volume of urine applied	TAN+u	Volume of urine applied	TAN+u	TAN+u input to the system (g)
			(1st half)		(2nd half)		
			(L)	(g/L)	(L)	(g/L)	
T ₁ R ₁	8.75	8.43	4.25	6.84	4.18	8.24	63.51
T ₁ R ₂	8.75	6.71	4.10		2.61		49.55
T ₂ R ₁	4.90	4.90	2.80		2.10		36.46
T ₂ R ₂ *	3.21	3.21	0.70		2.51		25.47
T ₃ R ₁	0.91	0.84	0.52		0.32		6.22
T ₃ R ₂	0.91	0.93	0.52		0.41		6.89

*T₂R₂ was replaced with new material after 3rd week

TAN captured by acid trap

Samples from acid trap were analyzed to get the amount of trapped TAN (Table 10). As volume of acid trap (initial volume was 300ml) was increasing due to added water vapor from urine drying, the total volume of acid traps differed between the treatments. The largest amount of TAN captured by the acid trap was for T₁R₂ (20.97g), and the smallest amount was captured by the acid trap for T₃R₁ (1.01g).

Table 10. TAN captured by acid traps in different treatments

Treatment	Total volume (L)	TAN conc. (g/L)	Total captured TAN (g)
T ₁ R ₁	2.8	3.20	8.97
T ₁ R ₂	3.1	6.76	20.97
T ₂ R ₁	4.0	3.54	14.15
T ₂ R ₂ *	2.3	3.80	8.75
T ₃ R ₁	1.0	1.01	1.01
T ₃ R ₂	1.2	0.99	1.19

*T₂R₂ was replaced with new material after 3rd week.

TAN in condensate

Pipes for collecting condensate were set up before the acid trap, so it was expected that the condensate must contain some TAN since it was directly coming from urine drying box. Samples from condensate were analyzed and amount of TAN was calculated similarly as it was done for acid traps. The concentration of TAN was found highest for T₁R₂- 0.3g/L for 1st

half of the experiment and it was highest for T₂R₁- 0.4 g/L in the 2nd half of the experiment (Table 11). On the other hand, it was lowest for both T₃R₁ and T₃R₂- 0.07 g/L in the 1st half and in second half it was lowest for T₁R₂- 0.08 g/L.

Table 11. TAN in total amount of condensate

Treatment	Total volume (1st half) (L)	TAN conc. (1st half) (g/L)	Total volume (l) (2nd half) (L)	TAN conc. (2nd half) (g/L)	TAN in the total volume (g)
T ₁ R ₁	0.5	0.09	0.75	0.16	0.17
T ₁ R ₂	0.4	0.30	0.80	0.35	0.40
T ₂ R ₁	0.4	0.21	0.25	0.40	0.18
T ₂ R ₂ *	*	*	0.30	0.34	0.10
T ₃ R ₁	0.07	0.07	0.09	0.10	0.01
T ₃ R ₂	0.05	0.07	0.08	0.08	0.01

*T₂R₂ was replaced with new material after 3rd week and afterwards no condensate stored in the T-connection until 4th week

Total solids in dried urine

Total solids in dried urine (urine+ash+lime) were different for different treatments. T₂R₁ was the one which contained highest percentage of total solid (82%) and it was lowest for T₃R₁ (56%) (Table 12).

Table 12. Total solid in dried urine

Treatment	Total solid (%)
T ₁ R ₁	74
T ₁ R ₂	76
T ₂ R ₁	82
T ₂ R ₂	73
T ₃ R ₁	56
T ₃ R ₂	67

The pH and stored TAN+u in dried urine

The average pH of dried urine was 12.1 as measured in the experiment. Dry weight of dried urine was calculated by using the values of wet weight and percentage of total solid. TAN+u concentration was measured based on wet weight and then transferred into gram of TAN+u per kilogram of dry weight. After that, dry weight of dried urine was multiplied with TAN+u concentration (g/kg of dry weight) and total amount of stored TAN+u in dried urine was calculated. T₁R₁ was the treatment with highest concentration of TAN+u (30.40g/kg of dry weight) and highest amount of TAN+u stored in dried urine, where replicate of the same treatment (Treatment 1) T₁R₂ contained only 9.7 g of TAN+u per kg of dried urine (dry weight) (Table 13). For Treatment 2, replicates T₂R₁ and T₂R₂ contained 20.06 and 17.42 g TAN+u/kg of dry weight. T₂R₂ was fed with urine for 4.5 weeks and it contained 14.42 g of TAN+u, where T₂R₁ contained 18.65 g of TAN+u after 7 weeks of urine application.

Table 13. TAN+u stored in dried urine

Treatment	Wet weight (kg)	Dry weight (kg)	TAN+u conc. (g/kg of dry weight)	TAN+u conc. (g/g of dry weight)	TAN+u stored (g)
T ₁ R ₁	1.81	1.35	30.40	0.03	40.90
T ₁ R ₂	2.33	1.76	9.70	0.01	17.03
T ₂ R ₁	1.13	0.93	20.06	0.02	18.65
T ₂ R ₂ *	1.14	0.83	17.42	0.017	14.42
T ₃ R ₁	0.50	0.28	21.65	0.022	6.09
T ₃ R ₂	0.48	0.32	21.66	0.022	6.90

Mass balance for TAN+u

After calculating the total amount of TAN+u in fresh urine, dried urine, condensate and acid trap, all the values are put together to make a mass balance of TAN+u in the whole urine drying system (Table 14). Here, we consider fresh urine application as the input of TAN+u, the output includes two parts- one is storage in the dried urine, and the other part is loss from the system, which represents the TAN found in condensate and acid trap.

Table 14. Mass balance of TAN+U in the urine drying system

Treatment	Input (TAN+u)	Output (TAN+u)			Total output (storage+ loss) (g)	Unaccounted		Stored (%)	Lost (%)
	Fresh urine (g)	Storage	Loss			Input- Output (g)	Input- output (%)		
		Dried urine (g)	Conden -sate (g)	Acid trap (g)					
T ₁ R ₁	63.51	40.90	0.17	8.97	50.03	13.48	21.22	64	14
T ₁ R ₂	49.55	17.03	0.40	20.97	38.40	11.15	22.51	34	43
T ₂ R ₁	36.46	18.65	0.18	14.15	32.98	3.47	9.53	51	39
T ₂ R ₂ *	25.47	14.42	0.10	8.74	23.27	2.20	8.65	57	35
T ₃ R ₁	6.22	6.09	0.01	1.01	7.11	-0.89	-14.30	98	16
T ₃ R ₂	6.89	6.90	0.01	1.19	8.10	-1.21	-17.50	100	17

*T₂R₂ was replaced with new material after 3rd week.

For T₁R₁, total input is 63.51g and total output (storage+loss) is 50.03g where 40.90g is stored in dried urine (Table 14). For this treatment, the difference between total input and total output is 13.48g, 64% of the input TAN+u was stored in dried urine meanwhile 14% of TAN+u was found in condensate and in acid trap. For T₁R₂, storage was 34%, loss is 43%, and in this case, loss is higher than storage. For T₂R₁ and T₂R₂, 51% and 57% of input TAN+u was stored in dried urine and loss was 39% and 35% respectively; for T₃R₁ and T₃R₂, storage was 98% and 100% respectively where also 16 & 17% of loss was found.

Calibration of TAN values

Photometric analysis of acid samples gave some absorbance values. After converting those absorbance values in TAN concentration, it was apparent that TAN concentration in samples (Table 15) were below the detection limit of ammonia test kit (the lowest detection limit of ammonia test kit is 0.005g/L) except for T₃R₁. The samples were 100 times diluted than the actual acid solution as it was done for during TAN measurement of acid trap.

In case of acid solution, measured concentration of TAN ranged from 0.34 to 0.48 g/L when the TAN concentration was calculated for acid solutions (Table 15). On the other hand, the mixture of ash and lime was diluted 1000 times (according to dried urine analysis) before analysis and the average concentration of TAN was 3.74 g/kg (Table 16).

Table 15. TAN reading in acid solution without TAN according to spectrophotometer reading

Treatment	abs	TAN reading(in sample) (g/L)	TAN read (in acid solution) (g/L)
T ₁ R ₁	0.067	0.004	0.40
T ₁ R ₂	0.061	0.004	0.37
T ₂ R ₁	0.057	0.003	0.34
T ₂ R ₂	0.062	0.004	0.37
T ₃ R ₁	0.08	0.005	0.48
T ₃ R ₂	0.068	0.004	0.41

Table 16. TAN reading in drying agent solution without TAN according to spectrophotometer reading

Sample	abs	TAN reading (g/kg)	Average TAN reading (g/Kg)
1	0.056	3.37	
2	0.065	3.91	
3	0.058	3.49	3.74
4	0.057	3.47	
5	0.059	3.55	
6	0.078	4.69	

New mass balance for TAN+u after considering calibration values

A new mass balance for TAN+u was obtained (Table 17) after considering the influence of calibration values on photometric analysis of acid trap samples and dried urine samples. In case of acid traps, values from TAN+u calibration (Table 15) were deducted from respective TAN+u values measured for different treatments. For dried urine, average value from TAN+u calibration (Table 16) was deducted from measured TAN+u values of all dried urine samples. Output of TAN+u in different treatments were calculated according to new values for dried urine samples and acid trap samples and eventually percentages of stored and lost TAN+u were calculated to obtain the corrected mass balance.

Table 17. New mass balance for TAN+u after correcting the influence of acid and drying agent

Treatment	Input (TAN+u)	Output (TAN+u)			Unaccounted				
	Fresh urine (g)	Storage	Loss		Total output (storage+loss) (g)	Input-Output (g)	Input-output (%)	Stored (%)	Lost (%)
		Dried urine (g)	Condensate (g)	Acid trap (g)					
T ₁ R ₁	63.51	34.13	0.17	7.83	42.13	21.38	33.66	54	13
T ₁ R ₂	49.55	8.33	0.40	19.82	28.54	21.01	42.39	17	41
T ₂ R ₁	36.46	14.41	0.18	12.79	27.38	9.08	24.89	40	36
T ₂ R ₂ *	25.47	10.16	0.10	7.88	18.14	7.33	28.76	40	31
T ₃ R ₁	6.22	4.20	0.01	0.53	4.75	1.47	23.67	68	9
T ₃ R ₂	6.89	5.11	0.01	0.70	5.82	1.07	15.53	74	10

*T₂R₂ was replaced with new material after 3rd week.

TAN+u distribution between stored and lost (measured) fraction

Here, amount of TAN+u measured in stored and lost fractions (only measured amount from Table 17 were considered) were added up and taken as 100% and distribution of TAN+u between stored and lost fraction was calculated. N distribution for different treatments were plotted in a bar diagram (Fig.3).

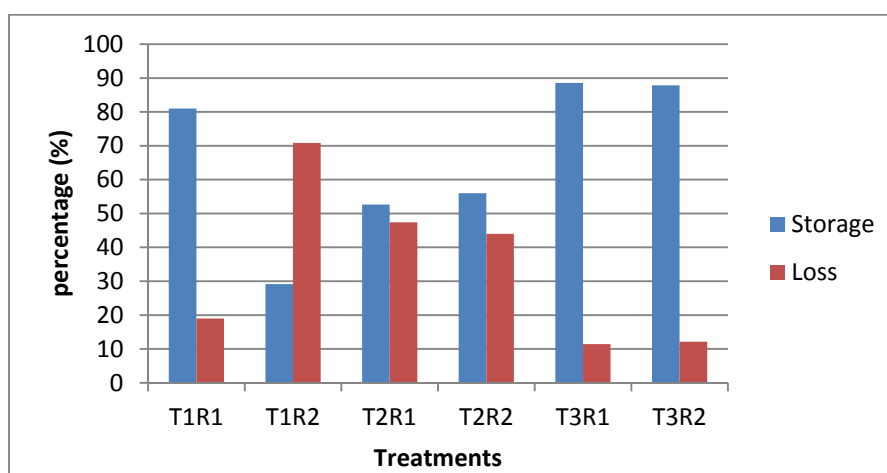


Figure 3. TAN+u distribution between stored and lost (measured) fraction

DISCUSSION

Drying capacity of the system

It was evident from the water loss test that temperature and airflow both were important factors influencing the amount of water lost from the system. Average amount of water lost per day was highest at 60°C temperature (T 1 & T 2) and it was lower at 35°C (T 3) and lowest at room temperature (T 4). The rate of airflow in the system is also important as a factor controlling daily water loss when if we compare between T 1 and T 2, where in both treatments had the temperature in the drying box 60°C but the rate of airflow was five times more in T 1 compared to T 2. This difference in airflow rate resulted in 45% more water lost per day in T 1 where amount of daily water lost in T 1 and T 2 were 223 ml 124 ml respectively.

Urine drying experiment

Urine application and TAN+u input into the system

Measured average values of pH and TAN+u in urine in this experiment were found compatible with the values mentioned in literatures (Diem & Lentar, 1970; Lentner et al., 1981; Nordin, 2010). The difference in TAN+u values between 1st half and 2nd half of the experiment was because of time of urine collection from donors. During the 1st half of experiment, urine was collected between 10 am to 2 pm but in the second half “first pee in the morning” was collected from donors which resulted in somewhat higher TAN+u concentration in fresh urine input.

For treatment T₁R₁ and T₁R₂, the theoretical volume of urine application was expected to be 8.75 L but the actual volumes applied to those two treatments were 8.43 L and 6.71 L respectively. The difference between actual and expected volume of urine application was not big for T₁R₁, but the difference is big for T₁R₂. This occurred because of unexpected drying condition throughout the experiment. The drying capacity of the system was measured during water loss test and urine application rate was 20% less than the drying capacity. Therefore, it was expected that the drying agent (ash+lime) added with urine would be completely dry at the end of every week but the reality was different.

A general observation about the experiment was that all treatments were drying very well during 1st and 2nd week and at the end of 2nd week all treatments became slightly moist. Besides, different treatments became moist at different periods of the experiment and the degree of wetness was different for different treatments, some were less moist and some were more. Some amendments were done to recover drying condition and those were described in materials and method section. While searching for reasons behind such moist condition, it was found that sometimes airflow was obstructed in the pipe because of water vapor condensed and remained in pipes, both in between moisture control box & drying box and drying box & acid trap, so air could not pass to drying box. Throughout the experiment, it was observed that maintaining a good airflow in the system was necessary to keep good drying condition.

TAN+u captured by acid trap and in condensate

Treatment 1 replicates behaved very differently from each other. In case of T₁R₁, the acid trap captured 7.83 g of TAN+u in total (Table 17) where the total TAN+u input was 63.51g (Table 9) and for T₁R₂ the acid trap captured 19.82 g (Table 17) of TAN+u in total where the total input was 49.55g (Table 9). It is evident from the study that the loss of TAN+u was significantly higher in T₁R₂. In case of Treatment 2, replicate T₂R₁ had higher losses compared to T₂R₂ where in case of Treatment 3, ammonia loss was similar for both replicates.

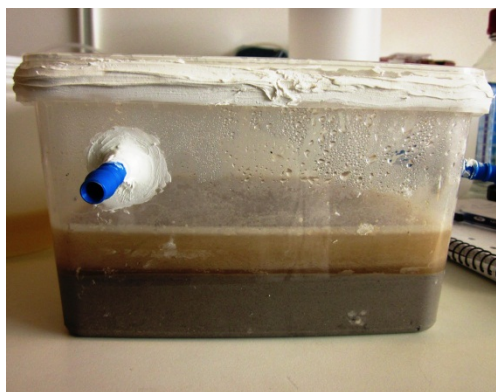


Figure 4. Urine flooding over drying agent
Photo: Shanta Dutta

As observed during the experiment, T₁R₂ showed very bad drying condition compared to T₁R₁. For T₁R₂ urine was flooding over the drying agent (ash+lime) for a long time. For T₂R₁, drying condition was comparatively better for first five weeks and urine started to flood over the drying agent on the 6th week. It was mentioned earlier in this document that T₂R₂ was replaced after 3rd week of the experiment, since it could not recover urine flooding, which was the result of bad drying condition. *Figure 4* shows the actual condition of T₂R₂. Treatment T₃R₁ and T₃R₂ were also flooded with urine for a few days.

As airflow was obstructed due to condensing water in pipes, the system could not run at its full potential that means evaporation was less. That condition made the drying agent saturated with urine and consequently urine flooded on top of drying agent since urine was applied regularly (except for 2nd half of the experiment when urine application was reduced by some percent for a period and even sometimes urine was not applied at all to some treatments). Urine flooding might reduce the rate of evaporation in drying box. There are some peptides and other shorter proteins in the urine upon excretion and when urine flooded over drying agent, those substances end up on top resulting in a thin lipid layer that obstructs the water from evaporating. In this case, very high temperature is needed for urine drying and 35 and 60°C temperature is not adequate to evaporate the amount of water from flooded urine (*Figure 2*) as it does when urine is absorbed in drying agent (Vinnerås, pers).

The aim of using the mixture of ash and lime as drying agent was to maintain the pH>10 in the drying box and thereby to inhibit the activity of urease enzyme even though addition of fresh urine acts to lower the pH every day. When the drying agent was totally saturated and urine flooded over it, then the pH might get down below 10 in the flooded urine, although pH

of dried urine (drying agent+urine) was 12 as measured during the experiment. Hence, urease enzyme might become active in flooded urine and urea be hydrolyzed. As mentioned before, urine started to flood over T₂R₁ from the 6th week and the data from condensate analysis (Table 11) also shows that the loss was higher in the 2nd half of the experiment, it is evident that moist material in the drying box contributed to TAN+u loss.

Total solids in dried urine

Measurement of total solids in dried urine shows that the final product, the urine fertilizer, was not completely dry as expected. In general, for Treatments 1 and 2, where drying temperature was 60°C, all replicates had 25% moisture on an average but in case of Treatment 3, moisture was 44% and 33% respectively for T₃R₁ and T₃R₂, which are high. It was mainly because of those two treatments were not drying well in the 6th and 7th week of the experiment due to urine flooding over drying agent.

Stored TAN+u in dried urine

Data from TAN+u concentration in dried urine (Table 17) showed that TAN+u was actually stored in drying agent (in the form of urea), so our hypothesis was accepted. TAN+u concentration was higher in some treatments compared to others, but even so it is evident that the idea behind using the mixture of ash and lime as drying agent and thereby, inactivating enzymatic activity of urease by maintaining high pH was correct.

Mass balance for TAN+u

TAN calibration values indicate that there might be some influence of acidic ions during the photometric analysis of acid trap samples as the calibration values indicated that the read TAN concentration during photometric analysis were increased. The condition was similar for dried urine analysis, where we may expect influence from wood ash and lime. As we see in the (Table 14), for T₃R₁ and T₃R₂, we have 14 and 17% more output in the system compared to input; where (Table 17) shows that the output is less than input after correcting for the calibration values. We expected the degradation of organic nitrogen in urine (which were in the form of peptides and amino acids) throughout the experiment which might also end up in the dried urine, however, this degradation does not seem to be large, as nitrogen was unaccounted for all treatments (Table 17).

According to the corrected mass balance (Table 17), T₁R₁ showed higher TAN+u storing capacity (54%) compared to the replicate of same treatment T₁R₂ (17%) that means T₁R₁ retained a good proportion of the input TAN+u but T₁R₂ lost the major part of input TAN+u (41%). This difference between replicates can be explained by the drying condition occurred throughout the experiment. As observed during the experiment, T₁R₁ was drying well throughout the experiment except for 6th and 7th week. On the other hand, T₁R₂ was not drying well most of the time. Urease-producing bacteria might be active in flooded urine when pH was below 10, and a substantial amount of ammonia was lost that was captured by the acid trap (19.82 g). Similar explanation can be given for T₂R₁ and T₂R₂ where both replicates retained only 40% of input TAN+u.

If we consider all replicates from all the treatments, both replicates in Treatment 3, T₃R₁ and T₃R₂ showed higher TAN+u storing efficiency (68% and 74% respectively, Table 17) among all replicates of other treatments, though those became moist several times throughout the experiment. It can be said that those two treatments T₃R₁ and T₃R₂, which were provided with 35°C drying temperature and 1 L/min airflow rate, had the best TAN+u retaining capacity and smallest losses compared to all the treatments set up in 60°C drying temperature. High temperature might favor ammonia volatilization as the ammonia equilibrium ($\text{NH}_3\text{-NH}_4^+$) gives a higher percentage of NH_3 (gas) at higher temperature, so ammonia volatilization risk increases with increasing temperature.

As measured in the experiment, the highest TAN+u retention in this experiment was 54% at 60°C, and at 35°C it was 74% (Table 17). Kabdasli et al. (2006) reported a TAN+u recovery of 95% through struvite precipitation conducted on enzyme hydrolyzed samples at 20±1°C; it needs to be noted that the precipitation of 1 kg TAN requires between 9.5 kg to 7.1 kg H_3PO_4 , 4.0 kg MgO and 1.2 kg NaOH (Siegrist, 1996; Munch and Barr, 2001). In another experiment, 86% recovery by using clinoptilolite was reported by Beler-baykal et al. (2011). TAN+u retention rate in this urine drying experiment is lower than those more resource demanding concentration methods. In another experiment, Antonini et al. (2012) reported 70% recovery of input nitrogen by solar thermal evaporation, which is close to the value that we got for the treatment in 35°C.

TAN+u distribution between stored and lost (measured) fraction

It is apparent from Figure 3 that TAN+u distribution between stored and lost fraction (measured) was different for different treatments and it differed also between replicates. For T₁R₁ and T₁R₂, we got completely different results; for T₁R₁, 81% of all recovered TAN+u was found in stored fraction and only 19% was found in lost fraction. On the other hand, T₁R₂ contained only 29% in the stored fraction, so most part of recovered TAN+u was in fractions that would be lost. T₂R₁ and T₂R₂ behaved similarly; stored fractions of TAN+u were 53% and 56% respectively while 47% and 44% respectively were found in fractions that would be lost. T₃R₁ and T₃R₂ were similar in case of accounted TAN+u distribution between stored and lost fractions; respectively stored fraction contained 89% and 88% of recovered TAN+u and lost fractions contained 11% and 12%, respectively. We can see that T₃R₁ and T₃R₂ functioned well for TAN+u retention.

The final product

The final product- the dried urine was a soil like material; it was dark in color and smelt like a mixture of urine and lime and the smell was not the same as fresh or stored urine. The fertilizer was not in powder form, since it contained a considerable amount of moisture and went through consecutive dry and moist phases during the experiment but could be dried to close to 100%. The pH of the fertilizer was measured and it was 12.



Figure 5. A snap of dried urine fertilizer at the end of experiment
Photo: Shanta Dutta

The amount of urine treated per gram of drying agent was calculated after the experiment was finished. Total amount of urine applied was divided by total amount of drying agent that gives amount of urine treated per gram of drying agent. A concentration factor was also calculated that indicate the proportion of weight of urine treated and the final dry weight of the fertilizer.

Table 18. Concentration factor for different treatments

Treatment	Total amount of urine applied (ml)	Total amount of drying agent (g)	Urine treated per gram of drying agent (ml)	Final dry weight (kg)	Concentration factor (weight of urine treated, kg /final dry weight, kg)
T ₁ R ₁	8430	900	9.4	1.35	6.2
T ₁ R ₂	6710	1300	5.2	1.76	3.8
T ₂ R ₁	4900	650	7.5	0.93	5.3
T ₂ R ₂	3210	650	4.9	0.83	3.9
T ₃ R ₁	840	240	3.5	0.28	3.0
T ₃ R ₂	930	240	3.9	0.32	2.9

Table 18 shows that T₁R₁ is the treatment which treated the largest amount of urine per gram of drying agent (9.4 ml) and that treatment represents the highest concentration factor (6.2) that means it was the most efficient treatment in terms of urine concentration. T₂R₁ had higher concentration factor compared to T₂R₂ since T₂R₂ was replaced with new material after the 3rd week. On the other hand, T₃R₁ and T₃R₂ has similar concentration factors that goes with their similar condition throughout the experiment.

Additional plant nutrients in the final product

Concentration of TAN+u in dried urine has been estimated and discussed in detail in this report, which was the main theme of the experiment. Other plant nutrients like phosphorus (P), potassium (K), calcium (Ca) should also be present in the final product, as raw materials used in the experiment contained those elements. Concentration in the final product can be estimated by using the values of those plant nutrients in urine and in drying agent, found in literature and by using the information provided with the product.

Since amount of urine applied differs for different replicates in the same treatment and they even do not represent the initial mass of drying agent, it is difficult to generalize the concentration of those nutrients in the final product in this particular study. T₁R₁ is the only

replicate, which contains the initial mass of drying agent and amount of urine applied is almost the same as expected amount of urine application after 7 weeks of experiment. The following estimation shows the concentration of plant nutrients in the final product from T₁R₁, and it is based on the assumption that no loss occurred during drying process.

Table 19. Estimated concentration of plant nutrient

Nutrient	Source of input			Total (g/kg of fertilizer)
	Urine ¹ (g/kg of fertilizer)	Ash ² (g/kg of fertilizer)	Lime ³ (g/kg of fertilizer)	
TAN+u (N)	25.3	0.2	-	25.5
Calcium (Ca)	-	60.0	173.3	233.3
Potassium (K)	11.4	7.5	-	18.9
Magnesium (Mg)	-	3.3	-	3.3
Phosphorus (P)	4.1	1.4	-	5.5
Sulfur (S)	3.4	-	0.7	4.1

1 Concentration values of K and P are calculated according to the nutrient values in urine proposed by Vinnerås et al. (2006), except for TAN+u, and the concentration factor (Table 18); concentration value of S was calculated from Jönsson et al., 2005

2 Values estimated according to Vance, (1996)

3 Values estimated from atomic mass and information provided with the product (Appendix 1)

Here, TAN+u input coming from urine is given as measured in the experiment and after correction for the calibration value. Dry weight of the fertilizer was considered for calculation of nutrient values. If all the replicates would contain the initial mass of drying agent until the end of experiment and expected amount of urine would be applied, then concentration of nutrient in the final product would be same for all treatments, since the proportion of initial mass of drying agent and amount of urine applied is same for all treatments. Assuming that there was no nutrient loss during urine drying, above estimation (Table 19) shows that calcium (Ca) is the dominant nutrient (233.3g/kg of dry weight) in the final product since it is the main constituent of drying agent. The fertilizer contains high concentrations of TAN+u, potassium, magnesium, phosphorus and sulfur that all are necessary plant nutrient.

Probable effect on pathogens

Temperature, pH, desiccation, and concentration of uncharged ammonia have been discussed and proved as important factors for pathogen inactivation in excreta (Höglund, 2001; Vinnerås, 2002; Nordin, 2010). Here, in this experiment, pathogen inactivation is not possible by means of high ammonia concentration, since urease enzyme was inactivated by high pH of drying agent. Therefore, temperature, pH and desiccation are probable driving forces behind pathogen inactivation.

High temperature (>40°C) can quickly kill most microorganisms despite of various types of media including water, soil, sewage and crops (Feachem et al., 1983). In case of composting process, temperature around 55-65°C can kill all types of pathogens (except bacterial spores) within hours (Burge et al., 1981; Haug, 1993). On the other hand, very acidic or very alkaline conditions can have an inactivating effect on most microorganisms by the hydrolysis of cell components or denaturation of enzymes (Atlas & Bartha, 1998). Eriksen et al. (1995) recommended a pH of 12 to inactivate *Ascaris* eggs, after 3 months of storage, where eggs of *Ascaris spp.* are regarded as being very resistant in the environment and to treatment.

Moisture favors the survival of pathogens, so drying can be an effective method for inactivation of pathogens (Esrey et al., 1998). All biological activity stops at moisture level below 12% and moisture level below 30-40% can become a limiting factor in biological activity (Austin, 2002). Therefore, desiccation is an option for pathogen removal in our experiment, particularly at 60°C, where average moisture level is 25%. Moreover, wood ash is considered as a good desiccant and that is one of the drying materials used in the experiment.

In this particular experiment, we have treatment at two different temperatures, 35 and 60°C and pH of dried urine was 12 throughout the experiment as measured. In case of 60°C, temperature can be the single inactivation factor or the combined effect of 60°C temperature and pH 12 must be very effective for pathogens inactivation as discussed above. In case of 35°C, only temperature may not be efficient for pathogen inactivation but sufficient result can be expected from the combined effect of temperature, low moisture content and high pH.

An estimation for practical implementation

This urine drying experiment was done in laboratory scale and there was no scope to run it in a practical situation. Here is estimation for urine drying system in a household level. For this estimation, we assume that a family consisting of four members excrete approximately 6 liters of urine per day (1.5 L/person, day). We assume that they live in a tropical country and have a solar toilet where in tropical summer temperature should reach at 60°C in the drying bed and relative humidity in ambient air is approximately 70%. We assume that the ventilation pipe in toilet is directly placed on the drying bed, so we can expect 5 L/min airflow over the drying bed.

According to the calculation in our laboratory experiment, average water loss in above stated situation (60°C temperature, 70% relative humidity & 5 L/min airflow) is 6.2 L/m², day.

Therefore, it can be said that the family will need a drying chamber, which should have an area of 1m² to dry 6 liter of urine that they excrete every day. In this case, they need to apply 30 kg of drying agent (15kg ash and 15 kg of lime) in the drying chamber, which is five times of daily urine excretion rate of the family similar to the procedure used in our laboratory scale experiment.

As done in the experiment, once drying agent is applied in drying chamber, it can be used for seven weeks or longer and by this time, it will retain a good amount of TAN+u; after seven weeks or longer, it can be removed and used as fertilizer and the drying chamber needed to be refilled with fresh drying agent. There is a possibility to use the same drying material for more than seven weeks, so, research is needed to determine the optimum time after which the drying agent should be replaced. It is important to consider that evaporation rate in the drying chamber is particularly dependent on existing temperature & airflow inside the chamber and relative humidity in ambient air. The study shows that this particular system is vulnerable to failure and significant TAN+u loss can occur if the drying material get saturated and urine is flooding above it, which ultimately will reduce the nitrogen fertilizer value of dried urine.

CONCLUSIONS

This study was helpful to develop a urine drying system at definite temperatures and rates of airflow by using a mixture of ash and lime as drying agent. The system proved to work, but was sensitive to disturbances. It was recognized that temperature and airflow are important factors for this particular process and urine drying at 20°C temperature is not feasible according to water loss test, therefore it was not a part of actual experiment.

The final product (dried urine fertilizer) was partially dried, not completely dried as expected in the experiment. The experiment showed that it is possible to retain nitrogen from the urine in form of urea by maintaining a high pH (>10) that means the hypothesis was correct. The concentration of nitrogen in input urine, airflow and drying condition regulates the concentration of nitrogen in the final product.

Approximate mass balances for nitrogen were obtained for the investigated systems system. Obstruction in airflow to the drying chamber and following bad drying condition result in urine flooding over drying agent; urea might be hydrolyzed and caused the loss of input nitrogen. The experiment showed that nitrogen retention rate was higher in treatment at low temperature (35°C), compared to treatment at high temperature (60°C); the highest nitrogen retention was 54% of the inflow at 60°C and it was 74% at 35°C. More replicates are needed in order to generalize the result in specific temperature and airflow.

It was observed that technical errors during measurement could highly influence the result,. Moreover, difficulty in sampling of dried urine and influence of acid and drying agent on photometric analysis made it complicated to get actual values from measurement. Further research is needed to optimize the system in terms of temperature and airflow and to obtain specific results.

SUGGESTION FOR FURTHER STUDIES

The scope of this urine drying experiment was limited. There was only three different drying temperatures (20, 35 and 60°C) and two different rate of airflow were used; so, it was not possible to optimize the drying capacity of the system in terms of temperature and airflow. Different combinations of drying temperature within the range of 35-60°C and rates of airflow (e.g., 1, 2, 3, 4, 5) would be of interest to find out the optimum temperature and airflow for effective urine drying process.

Here, we had only two replicates for each treatment, which was not sufficient to establish an average concentration of nitrogen in the final product since different replicates behaved differently. More replicates for each treatment can be beneficial in this case. On the other hand, concentration of nitrogen in the final product was measured, and a mass balance was obtained, and that was the main objective in this study. Concentration of other plant nutrients like potassium and phosphorus can be measured in the final product and mass balance can be obtained for those nutrients. There is another possibility to investigate if the same drying material can be used for a period longer than 7 weeks and determining an optimum replacement time for drying material would be of interest.

In addition, laboratory test can be conducted to prove the pathogen inactivation capacity of this system. Practical implementation of this urine drying process and testing the effectiveness of dried urine fertilizer would be of interest for further research.

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APPENDICES

Appendix-1

Detail information about slaked lime (provided with the product):

Chemical name - Calcium hydroxide

Chemical formula - Ca(OH)_2

M = 74.10 g/mol

Specification:

Assay (acidimetric)	min	96%
Substances insoluble in hydrochloric acid	max	0.1%
Carbonate (as CaCO_3)	max	3.0%
Chloride (Cl)	max	0.005%
Sulphate (SO_4)	max	0.2 %
Heavy metals (as Pb)	max	0.005%
Iron (Fe)	max	0.05%
Substances not precipitated by ammonium oxalate (as sulphate)	max	2.5%

Appendix-2

Ammonia emission from urine drying:

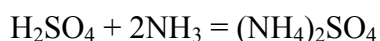
For estimating total NH_3 emission for 6 weeks the value for NH_4^+ -N concentration in urine was taken as 8 g/L. It was assumed that 20% of NH_4^+ -N would be emitted as NH_3 from the system.

Table.1 Estimated emission of NH_3 from urine drying

Treatment	Total amount of urine applied in 6 weeks (L)	Total amount of NH_4^+ -N in urine applied for 6 weeks (g)	Estimated total emission from 6 weeks (20% of NH_4^+ -N) (g)
T-1	7.56	60.48	12.1
T-2	4.2	33.6	6.72
T-3	0.781	6.24	1.25

Estimated total emission (Table-1) showed that T-1 would emit approximately 0.9 mole of nitrogen (molar mass of mono-atomic nitrogen is 14 g) and 0.9 mole of mono-atomic nitrogen forms 0.9 mole of NH_3 .

Stoichiometrically, 1 mole of H_2SO_4 can completely trap 2 moles of NH_3 .



For T-1, 0.45 mole of H_2SO_4 was needed to trap 0.9 mole of NH_3 . As a precautionary principle 0.75 mole of H_2SO_4 was used to trap the emitted ammonia from T-1 and 0.5 mole of H_2SO_4 was used for T-2 and T-3, which was more than required.

It is needed to mention that, more acid was added to acid traps later on while it was decided to carry out the experiment for 7 weeks.

Appendix-3

Pictures:

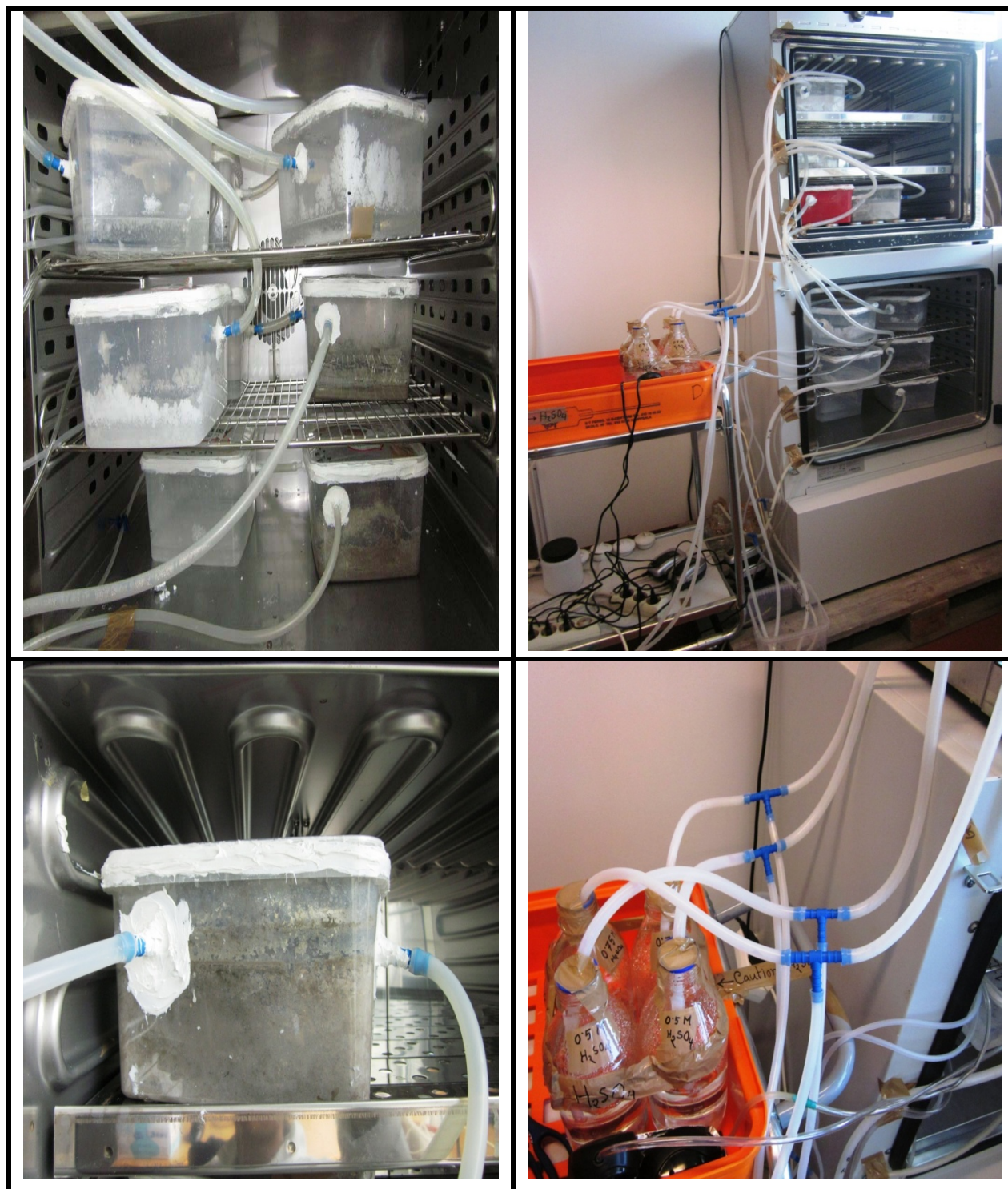


Photo: Shanta Dutta

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