Anastasija Vasiljev

SAFE NUTRIENT RECYCLING OF HUMAN URINE AS A CROP FERTILIZER

Capture of the free ammonium nitrogen during alkaline urine dehydration

Bachelor's thesis

Environmental Engineering

2020

South-Eastern Finland
University of Applied Sciences

South-Eastern Finland University of Applied Sciences

Author	Degree title	Time
Anastasija Vasiljev	Bachelor of Engineering	May 2020

Thesis title

Safe nutrient recycling of human urine as a crop fertilizer; Capture of free ammonium nitrogen during alkaline urine dehydration

44 pages
7 pages of appendices

Commissioned by

Swedish University of Agricultural Sciences (SLU), Department of Energy and Technology

Supervisors

Björn Vinnerås, Head of the Department of Energy and Technology (SLU)
Hannu Poutainen, Senior lecturer (XAMK)

Abstract

The symbiosis between agriculture and sanitation has long been one of the hot topics for environmentalists. Human urine contains all of the essential nutrients needed for plant growth and can potentially be used as a balanced fertilizer. One of the technologies used to produce such fertilizer is alkaline urine dehydration. The objective of this thesis was to examine whether free ammonium nitrogen lost during alkaline urine dehydration can be captured by struvite precipitation. Qualitative methods were used to examine the recovery rate of urine nutrients (primarily N), through continuous additions of alkalized urine to three different dehydration media (MgO, $Mg(OH)_2$, $Ca(OH)_2$) and subsequent drying at 40 °C. The urine samples (before and after drying) were analyzed, and recovery rates and fertilizer value of the end products were assessed. The study showed that all three drying media managed to recover almost all the nutrients, thus creating a high-value dry fertilizer. While the free ammonium N may have been captured as magnesium ammonium phosphate, the results were inconclusive because the urine contained very little ammonium (about 2% of the total N), which was less than the standard deviation of the analytical methods used to measure the N content of urine and the end-products. This thesis also provides recommendations for future research by describing which further adjustments to the experimental setup are needed for quantifying the rate of free ammonium nitrogen capture from fresh human urine.

Keywords

nutrient recycling, urine, fertilizer, ecological sanitation, sustainability

CONTENTS

1 INTRODUCTION

Human excreta are still poorly managed on the global scale, with over four billion people still discharging untreated human excreta into the environment, causing eutrophication and spreading disease (WWAP, 2017). Human urine is the major contributor of nutrients and organic matter to household wastewater streams and contains the same nitrogen, phosphorous and potassium as the fertilizers used to produce the food consumed (Senecal & Vinnerås, 2017). More and more fertilizers are harmfully produced in order to replace the nutrients removed from the fields during harvest (Senecal & Vinnerås, 2017). However, by recycling human urine, approximately 20% of N, 25% of P and 35% of K used in Swedish farms can be replaced. Human urine-based fertilizers thus have potential as a lower environmental impact alternative to chemical fertilizers.

Source-separated and treated urine is already being used in agriculture (Langergraber & Muellegger, 2005). There are several technologies that treat and transform the urine into an effective fertilizer, and one of them is alkaline urine dehydration, which has been developed by the environmental engineering research group (Kretsloppsteknik) at the Swedish University of Agricultural Sciences (SLU). This method first alkalizes urine by increasing its pH and then dehydrates it, thus producing dry, nutrient-rich fertilizer. The benefits of this method lie in its simplicity and minimal capital costs compared to the conventional sanitation system and that it can be adapted almost anywhere in the world.

However, the system still needs further research, especially optimization. My thesis focuses on free ammonium nitrogen ($N\text{-}NH_4^+$), which is N that is potentially lost during the alkaline urine dehydration process (due to high pH of >10), but which can perhaps be captured by using a magnesium-based medium to produce struvite precipitate. The goal is to determine which magnesium media trap ammonium nitrogen in the most efficient way during the urine dehydration process, as well as to understand which factors influence its retention. The research for this thesis was conducted at the *Department of Energy and Technology (SLU)* over two months.

The thesis begins with the *Theoretical Background* of wastewater and sanitation, urine treatment methods and a setup of the alkaline urine dehydration unit installed at SLU. Then, in the *Materials and Methods*, the theoretical basis of my project, as well as the technical explanation of the system setup, is given. The results are shown immediately after, with reference to appendices for detailed calculations. Within the *Discussion* chapter, the results are discussed, and explanations for certain trends and irregularities are given. The whole thesis is then concluded with the chapter titled *Conclusion.*

2 THEORETICAL BACKGROUND

Human excreta are a valuable resource with a long history of usage yet are underestimated and under-utilized today (Andersson, 2015). Urine has been especially important to scientific and industrial advancement – urea was the first organic substance created from inorganic compounds, a development which led to a new field of organic chemistry and the discovery of new medicines, new materials and synthetically derived compounds (Kumar, 2013).

In the past, throughout the world, urine has been used variously as a detergent, leather softener, dying agent, teeth whitener, an ingredient in gunpowder production, drink therapy and to smear wounds (Kumar, 2013; Schönning, 2006). It has also been used as a fertilizer and organic soil amendment (Simha, et al., 2017). One of the examples for a fertilizer would be a product called urat which consisted of urine mixed with peat litter (Schönning, 2006).

Today, urbanization, the introduction of water flush toilets and sewer networks and the widespread use of synthetic fertilizers have pulled society away from these old methods and practices for use of human urine. However, there is a rising concern about the future of fertilizer availability, which has led to a re-evaluation of the need for a more sustainable nutrient management, with one suggested approach being the recycling of nutrients from human excreta to agriculture (Harder, et al., 2019).

One major setback, however, is the prejudice surrounding the handling of human waste, which is often associated with cultural norms and taboos that restrict its use across the world (Drangert, 1998; Simha, et al., 2017). Another hurdle with such recycling approaches is the development and market readiness of new technologies that can safely harness and recycle nutrients present in human excreta (Simha & Ganesapillai, 2017). Hopefully the need for, education about and understanding of the benefits of human excreta recycling will transcend such socio-technical challenges and bring us closer to safe, sustainable and culturally accepted sanitation systems (WWAP, 2017).

2.1 Sanitation

The management of human excreta is still very poor on a global scale. In 2017, it was estimated that around 2.0 billion people still did not have access to basic sanitation facilities (WWAP, 2017). To make matters worse, human excreta from around 4.5 billion people is still being discharged into the environment without any treatment (WWAP, 2017). Improper sanitation is linked to transmission of diseases such as cholera, diarrhoea, hepatitis A and polio. Moreover, the discharge of the unwanted excreta leads to negative effects on the environment, such as eutrophication. Therefore, there is a strong need for sanitation systems that protect the user by providing safe toilets and for a safe way of removing, transporting and processing the excreta into value-added end products (Opel, 2012).

2.1.1 Conventional sanitation systems

When it comes to conventional water and sanitation systems, what we have today is essentially an end-of-the-pipe concept that produces linear mass flows of resources (*Figure 1*). Our current systems are designed and operated from a perspective that places human excreta in a category of ‘wastes’ (Ersay, 2001).

It all starts with nutrient extraction (mining of P and K, which are limited resources, and extraction of N from the atmosphere) for fertilizer production that requires large amounts of energy (Tildåker, et al., 2007); the fertilizer is then used in agriculture. Even when applied to agricultural lands, very little of these nutrients gets used by plants, as they often leach off the fields during irrigation or rain events (Bergström, et al., 2009). After fertilizer production and uptake by plants, these nutrients end up in households in the form of food, which upon consumption is converted into human excreta and kitchen waste.

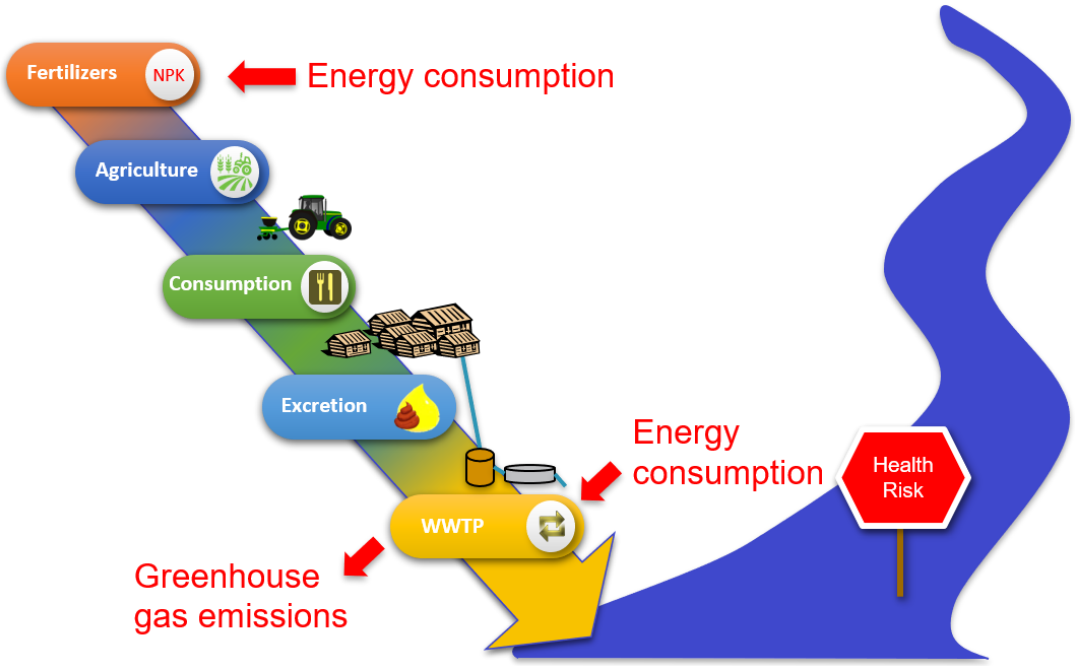

Figure 1. Linear mass flow of the nutrients from their extraction to release into the environment (modified with permission from McConville, 2020).

In urban areas of most countries, large volumes of clean water are used to dilute and transport small volumes of human excreta through sewer networks connecting household toilets with centralised wastewater treatment plants (Simha, et al., 2016). The transportation method itself also poses an issue, since it magnifies the volume of waste that needs to be treated at the end of the pipe (Simha, et al., 2016).

Generated wastewater that is transported by the sewage network to a wastewater treatment plant, also requires large amounts of energy to be treated; e.g. 45 $MJ \cdot kg_N^{-1}$ and 49 $MJ \cdot kg_P^{-1}$ (Maurer, et al., 2003). Treated effluent, greenhouse gases and sewage sludge are the outputs from the treatment plants (Daelman, et al., 2013). A large part of the sludge is then incinerated due to health concerns such as pathogens, organic pollutants and heavy metals (Harder, et al., 2019). Nutrients are thus not recovered but are wasted and lost.

Furthermore, there is a question of adequacy and long-term sustainability of the conventional urban water and sanitation systems. The biggest challenge for low-income countries is high infrastructure costs, whereas high-income countries

struggle with large energy and water consumption issues and problems with sludge disposal, which also limit nutrient recycling (Harder, et al., 2019).

2.1.2 Sustainable sanitation

A need for more sustainable systems has led scientists to slowly shift focus onto recovery and reuse of resources from wastewater and sanitation systems by following the concept of circular economy (*Figure 2*) (Cossio, et al., 2020). The aim of the alternative conventional systems is often to reuse the plant nutrients from excreta as a fertilizer (Schönning, 2006).

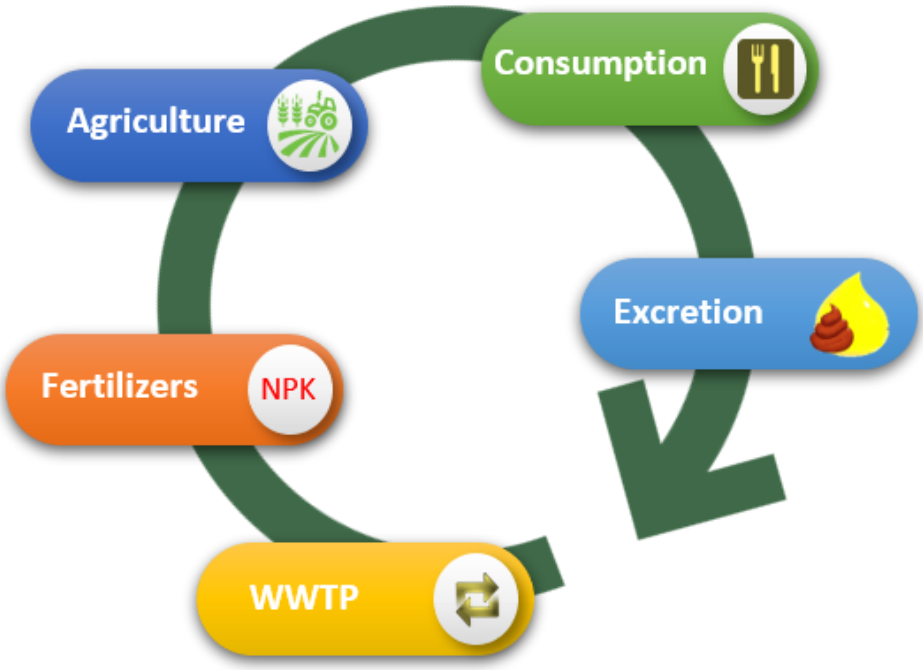

Figure 2. Circular approach to wastewater and sanitation systems – creation of a sustainable cycle (modified with permission from McConville, 2020)

Modern household wastewater is made of three major waste streams, greywater, urine and faeces, which are usually mixed and transported together into the sewage system (Vinnerås & Jönsson, 2002). These wastewater fractions have different characteristics and carry many different potential benefits (and risks), leading to the conclusion that they should be managed separately (Simha, et al., 2020).

One of the most popular sustainable concepts for wastewater and sanitation systems is source separation. Source separation systems include blackwater systems (toilet wastewater treated separately), urine-diverting systems (separate treatment of urine) and several types of dry systems (human excreta handled without the use of flush water) (Schönning, 2006).

Several environmental system analyses of wastewater systems place urine separation as one of the best alternatives to conventional sanitation systems (Tildåker, et al., 2007; Kvarnström, et al., 2006). Collection of urine at the source can bring many positive impacts, including improved performance of existing centralised wastewater treatment plants, lower rates of fresh water and energy consumption, and reduction of reliance on synthetic fertilizers, while simultaneously mitigating environmental pollution (Simha, et al., 2020). Moreover, separating faeces, which contain the most pathogens, from urine reduces the risk of potential transmission of water-borne diseases (Senecal, et al., 2018).

2.1.3 Urine-diverting toilet

For source separation of urine, urine-diverting toilets take advantage of human physiology, since the body separately excretes faeces and urine (Simha & Ganesapillai, 2017). The toilets are designed so that a divider directs the urine away from the faeces; the urine is then collected in the front-end bowl, whereas the rest of the excreta are deposited in the rear-end bowl (*Figure 3*) (Langergraber & Muellegger, 2005).

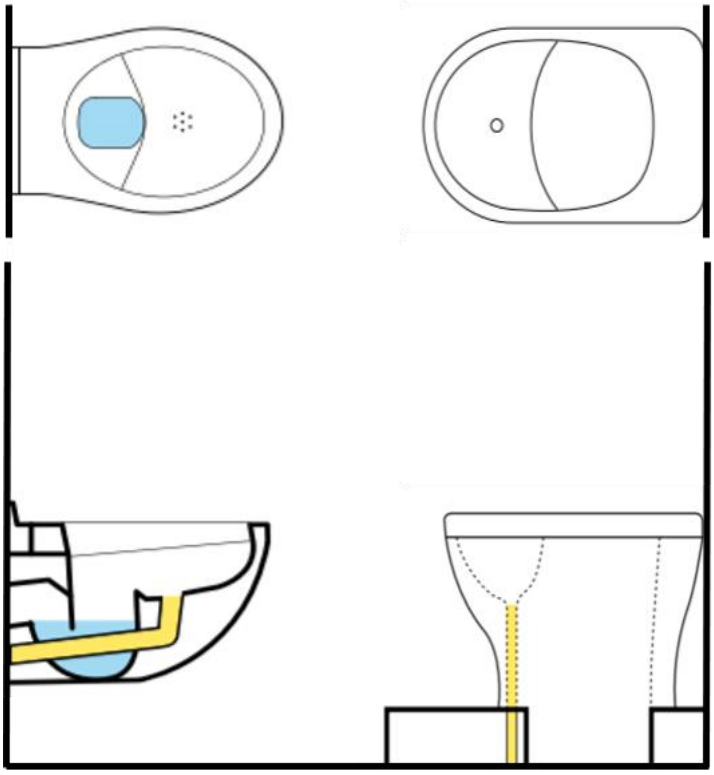

Figure 3. Schematic images of urine diverting flush toilet (left) and urine diverting dry toilet (right) (modified from Tilley, et al.,2014)

In some types of urine-diverting toilets, flush water is used for one or for both bowls (Langergraber & Muellegger, 2005). If flush water is absent by design, urine-diverting toilets are categorized as urine-diverting dry toilets (Simha & Ganesapillai, 2017). These types of toilets are already in use across the world, and many cities are considering implementing them at a larger scale (Kvarnström, et al., 2006).

2.2 Urine as a source of nutrients

Human urine is the major contributor of nutrients and organic matter to household wastewater streams (Tildåker, et al., 2007). The products created by treating human urine and/or human excreta can be in the form of fertilizers, feed or energy carriers, or used in production of biopolymers, biofuels or other valuable chemicals (Harder, et al., 2019).

In Sweden alone, with approximately 10 000 000 inhabitants, more than 40 000 tonnes of nitrogen, 3600 tonnes of phosphorous and 10 000 tonnes of potassium

are found in urine excreted by the citizens within a year (calculated from Vinnerås, et al., 2006). These figures can be compared to the demand of mineral fertilizers in agriculture in Sweden, which is more than 184 000 tonnes of nitrogen, 14 000 tonnes of phosphorous and around 29 000 tonnes of potassium (Statistics Sweden, 2019). This way, recycling human urine can replace approximately 20% of N, 25% of P and 35% of K used in Sweden today, and can thus have a considerable impact on agricultural practices and resource use. Therefore, by closing the nutrient cycle and recycling the nutrients back to agriculture, positive environmental, health and social aspects can be expected (Tildåker, et al., 2007; Langergraber & Muellegger, 2005; Senecal & Vinnerås, 2017).

2.2.1 Properties and composition of urine

Human urine is composed of more than 95% water by weight; the remainder consists of inorganic salts and organic compounds (Harder, et al., 2019). Phosphorous is mainly found as inorganic phosphates (PO_4-P), whereas potassium is mostly found as free ions (K^+) (Dutta & Vinnerås, 2016; Karak & Bhattacharyya, 2011). The pH of freshly excreted urine ranges from 4.8 to 8.2 (Dutta & Vinnerås, 2016).

In fresh urine, the most common form of nitrogen is urea (CH_4N_2O), which makes up 85% of the total nitrogen, while the remaining portion can be found as total ammonia (5-7%), creatine (6%), and shorter peptides and free amino acids (Harder, et al., 2019; Dutta & Vinnerås, 2016). Moments after urination, the non-volatile urea breaks down into bicarbonate (HCO_3^-), carbonate (CO_3^{2-}), non-volatile ammonium (NH_4^+) and volatile ammonia (NH_3) (Harder, et al., 2019). This urea degradation process is called urea hydrolysis, after which around 90% of the total nitrogen in urine can be present as ammonia or ammonium (Harder, et al., 2019).

Human excreta can also contain heavy metals, such as copper, zinc, chromium, nickel, lead and cadmium (Dutta & Vinnerås, 2016). However, their concentration in urine is generally quite low compared to that in the faeces and in other wastes, such as animal manure (Dutta & Vinnerås, 2016; Harder, et al., 2019). In fact, some

flush water can actually add heavy metals and other pollutants which originate from water supply systems (e.g., copper or lead pipes) to urine (Harder, et al., 2019).

Besides heavy metals, urine can contain organic pollutants, such as pharmaceutically active compounds and (synthetic) hormones such as estrogens (Lamichhane & Babcock, 2013; Harder, et al., 2019). Additionally, pathogens can also be found in urine. However, fresh urine (especially from healthy people) contains fewer pathogens, and pathogens found in toilet bowls are mostly due to faecal cross-contamination of urine during and after excretion (Harder, et al., 2019).

2.2.2 Urine as a crop fertilizer

Improper application of chemical fertilizers has caused significant adverse effects on soil health, which has led in part to the increased popularity of organic agriculture (Karak & Bhattacharyya, 2011). Yet, the progress of organic farming has been slow due to rapid decline in available organic raw materials (animal waste, crop residues, and green manure), and due to burning of waste and usage of straw and grass as animal feed (Karak & Bhattacharyya, 2011). Besides the influence of sanitation and wastewater and wastewater concerns, the decline of organic raw materials and the negative effects of chemical fertilizers have also led to an increasing demand for transition to human urine-based fertilizers (Karak & Bhattacharyya, 2011).

Nutrients found in urine are in a form that is highly available to plants, and their uptake by plants is as good as that from synthetic fertilizers (Dutta & Vinnerås, 2016). Urine has been successfully tested and proven as a good liquid fertilizer for a variety of food crops (Pradhan, et al., 2007). However, applying urine as a liquid fertilizer comes with a set of financial and logistical burdens due to the storage, transportation and application of large volumes of collected urine (Johansson, et al., 2001). Since more than 550 kg $year^{-1}$ per person of urine is produced (Vinnerås, et al., 2006), a large storage tank or frequent emptying to a central storage is required due to low application rate (only twice per growing season) (Senecal, et

al., 2018). Therefore, to improve the sustainability of re-using human urine as a fertilizer, the objective is to minimize the volume of urine and to create systems that effectively recover and concentrate the nutrients during urine treatment (Senecal, et al., 2018).

2.3 Technologies for on-site urine treatment

There are several technologies that have been researched for nutrient recycling from urine, which can be sorted into two broad categories.

The first category of methods focuses on selective extraction of the nutrients present in urine. However, it also leaves the system users with wastewater streams (e.g. reject water) that need to be managed or that require additional treatment before they are allowed to be discharged into the environment (Simha, et al., 2020). The methods which belong in this category are stripping, freezing-thawing, ion exchange, electrolysis, adsorption and struvite precipitation (Simha, et al., 2020; Senecal & Vinnerås, 2017; Senecal, et al., 2018).

Struvite precipitation is one of the most popular and common methods (Harder, et al., 2019). The objective of the method is phosphorous extraction; however, there is also partial nitrogen capture in the form of struvite crystals (further explained in *Materials and Methods).* One of the drawbacks of struvite precipitation is that there is persistent pathogen build-up in the precipitate (Harder, et al., 2019). Another setback, as Etter et al. (2011) demonstrated in their pilot system implementation in Nepal, is that struvite can only harness 13% of the potential monetary value of urine as a crop fertilizer.

The second category of technologies mainly focuses on water removal from urine, which concentrates nutrients and thus produces urine-based fertilizers in the form of liquids, slurries or dry powders (Harder, et al., 2019). The technologies belonging in this category are reverse osmosis, forward osmosis, membrane distillation, acidification or alkalization followed by dehydration, and partial nitrification followed by distillation (Simha, et al., 2020; Senecal & Vinnerås, 2017; Senecal, et al.,

2018). Apart from nitrification-distillation and alkaline dehydration, most of these technologies do not efficiently recover all of the nutrients excreted in urine, especially nitrogen.

Biological nitrification followed by distillation is a process in which urine nutrients are firstly nitrified by membrane aerate biofilm reactor and then concentrated by distillation reactors, resulting in all nutrients being recovered (except 3% of ammonia) in an alkali concentrate called “aurin” (Udert & Wachter, 2012). However, even though it has complete recovery of the nutrients, this process has large energy requirements – more than 4 times that of conventional wastewater treatment (Udert & Wachter, 2012).

The dehydration method is the one that is the most attractive way of concentrating urine, since water constitutes more than 95% of the volume of human urine, whereas nutrients only constitute 5% (Putnam, 1971). Simple convective air drying where water is removed by heating urine and forcing a current of air over its surface is not effective, since evaporation is inhibited by a thin oily film that forms over the liquid surface (Tettenborn, et al., 2007). Therefore, to successfully dehydrate urine, stabilization techniques are employed, which include acidification, alkalization, salinization, or freezing-thawing (Lind, et al., 2001). Alkaline dehydration, developed at the *Swedish University of Agricultural Sciences,* not only effectively recovers most of the N-P-K (labelling used in conventional fertilizers given as percentage of weight) but also simultaneously decreases the volume of urine, making it an effective method that produces highly concentrated dry fertilizer (Simha, et al., 2020).

2.3.1 Alkaline urine dehydration

The biggest challenge for concentrating nutrients (mainly N) and for pipe transport and storage of urine is bacterial hydrolysis of urea (Eq. 1) (Senecal & Vinnerås, 2017; Simha & Ganesapillai, 2017). The hydrolysis of urea to ammonia occurs due to the catalytic action of the urea-specific enzyme urease (urea amidohydrolase),

which is produced by bacteria ubiquitously present in urine-collecting systems (Simha, et al., 2020).

$$H_2N - CO - NH_2 + H_2O \xrightarrow{Urease} H_2N - COOH + NH_3 \xrightarrow{H_2O} H_2CO_3 + 2NH_3 \quad (1)$$

There are several unfavourable consequences of urea hydrolysis. Firstly, it completely converts urea into carbon dioxide and ammonia, which subsequently volatilizes, and almost all of the nitrogen is then lost (Nordin, 2010). Secondly, it leads to pH elevation, which decreases the potential of post-storage nitrogen reuse (Simha & Ganesapillai, 2017). Thirdly, the elevated pH triggers precipitation of struvite and calcite, leading to creation of blockages in the pipelines and, subsequently, a need for frequent maintenance (Udert, et al., 2003). Lastly, during storage, it results in physio-chemical, as well as microbial, stratification of urine (Höglund, et al., 2000).

Besides enzymatic hydrolysis, urea can also break down by thermal degradation and chemical hydrolysis (Simha, et al., 2020). However, at low temperatures, the rate of enzymatic urea hydrolysis is much greater than that of its chemical hydrolysis (Senecal & Vinnerås, 2017).

In alkaline urine dehydration, source-separated urine is first alkalized and then concentrated by dehydration, where its volume is reduced (Simha, et al., 2020). The volatility of ammonia, however, provides a challenge to this method, since urine cannot simply be dehydrated (Senecal & Vinnerås, 2017). Therefore, in order to treat urine, inhibition of urease activity (stabilization) is essential to recover N (as urea) (Simha, et al., 2020).

By preventing the hydrolysis of urea, the dehydration of urine results in minimal loss of nitrogen while retaining most other macro- and micronutrients (Senecal, et al., 2018). The retention of such a high concentration of nutrients leads to production of a complete fertilizer, where the balance of N-P-K reflects the plant nutrient requirements for the crops (Simha, et al., 2020).

In order to inhibit the urease activity and enzymatic ureolysis, alkalization or acidification, and elevated temperature can be used (Senecal, et al., 2018; Simha, et al., 2020). By limiting urease enzyme with elevated pH and/or elevated temperature, uncatalyzed urea hydrolysis can be observed (Eq. 2) (Senecal & Vinnerås, 2017). However, at low temperatures (<30 °C), this type of hydrolysis is 10^{10} times slower than enzyme-catalysed hydrolysis, thus giving an opportunity for the dehydration of urine and, subsequently, production of the dry fertilizer (Senecal & Vinnerås, 2017).

$$H_2N - CO - NH_2 \xrightarrow{Uncatalysed} HN{=}C{=}O + NH_3 \xrightarrow{H_2O} H_2CO_3 + 2NH_3 \quad (2)$$

To inhibit urease activity with alkalization, the pH of the drying medium should be above 10, since no hydrolysis occurs in urine at such a high pH (Kabdasli, et al., 2006). Examples of drying media which can be used to prevent hydrolysis are wood ash and slaked lime (Dutta & Vinnerås, 2016). These types of alkaline media, over which urine is spread, are able to bypass the formation of oil films and thus enable dehydration of urine by convective heat and mass transfer, while still effectively concentrating and retaining urine nutrients (Simha, et al., 2018). Additionally, to achieve higher dehydration rates, the temperature and the volume of added urine are increased, while the amount of drying medium and the air flow are decreased (Simha, et al., 2018).

2.3.2 Setup of the alkaline drying system

Combining the knowledge of the technology of urine-diverting toilets and that of alkaline urine dehydration, a pilot-scale system was installed at the Swedish University of Agricultural Sciences (*Figure 4*). The goal is to provide container-based sanitation technologies that collect, contain, treat and reduce the volume of urine within the container (Senecal & Vinnerås, 2017).

Figure 4. Alkaline urine dehydration setup at the *Swedish University of Agricultural Sciences,* with urine diverting toilet (A), dehydration container (B) and ventilation pipe (C)

The urine is deposited continuously to a urine-diverting toilet (*Figure 4, A*), which is then dehydrated under constant ventilation in the drying container (*Figure 4, B*), reducing its mass by 90% (Senecal & Vinnerås, 2017). When the monitored pH of the dehydration medium decreases to <10 (due to capture of CO_2 from the air), it is immediately replaced with a fresh batch of drying medium (Simha, et al., 2018). The advantage of having the dehydration unit inside the bathroom is that no extra piping is required, except for connecting the drying container to ventilation (*Figure 4, C*). Since the system does not require a tank, extra plumbing or an additional sewer connection, most existing toilets (both dry and flush) can be adapted to these technologies with minimal capital costs.

In the case of these technologies, no liquid disposal is required from the toilet. The end product is a dry organic fertilizer that is rich in macronutrients, N (>10%), P (>1.5%), K (>5%), S (>0.5%), Ca (>5%), Mg (>0.3%) and C (>8%), and in micronutrients (Fe, Mn, Zn, Cl and Cu) (Simha, et al., 2020). This produced fertilizer can then be collected as a solid and transported to a central fertilization unit or directly applied to gardens and agricultural land (Simha, et al., 2017).

3 MATERIALS AND METHODS

The method of alkaline urine dehydration developed at the Swedish University of Agricultural Sciences is still in the development stage, with a Technology Readiness Level between 5-6 (NASA, 2012). Therefore, there is still a need for research and optimization of the system. The goal of this thesis is to contribute to development of this technology and help steer the research in the right direction.

3.1 Capture of free ammonium nitrogen

When human urine is treated by alkaline dehydration, all phosphorous and potassium and most of the nitrogen can be recovered (Simha, et al., 2018). Yet one of the challenges in alkaline urine dehydration is the loss of free ammonium nitrogen during urination and transport. When the dehydration system is set up next to the urine-diverting toilet (*Figure 4*), the alkalization of urine is done immediately after the fresh urine is excreted. The alkalization can be done with the addition of potassium hydroxide (KOH) solution or by using an alkaline drying medium such as calcium hydroxide or ash (Dutta & Vinnerås, 2016). However, since the fresh urine already contains free ammonia, it is estimated that up to 5% of nitrogen in a form of free ammonium nitrogen is lost during the alkaline dehydration treatment (Harder, et al., 2019; Simha, et al., 2018).

In the case of application of the drying system on a larger scale, for example, in a multi-store building with centralised dehydration systems where fresh urine is alkalized and treated off site (e.g. a basement or outside building), a significantly larger amount of urea is hydrolyzed during pipe transport (Simha, et al., 2018). Such a system can lead to a nitrogen loss of more than 30% (personal communication with Simha, 2020).

On average, in 550 kg of urine excreted by an adult per year, approximately 4.0 kg of nitrogen is present (Vinnerås, et al., 2006). This means that only approximately 0.2 kg of ammonium nitrogen can be lost if treated by alkaline dehydration.

However, when there is a piping system involved, the loss of ammonium nitrogen can rise to 1.2 kg, which is six times more than that in the localized dehydration system. Applying this calculation on a larger scale, the loss of the nitrogen in Stockholm alone can reach more than 1959 tonnes (World Population Review, 2020). Therefore, development of capture methods offers the opportunity for significant resource recovery.

The method this thesis explored was capturing free ammonium nitrogen by utilizing the advantages of struvite formation. In an earlier chapter (*Technologies for on-site urine treatment*), it was mentioned that struvite is one way to capture nitrogen in the form of ammonium nitrogen when treating hydrolyzed urine. Hence, this experiment aims to combine the methods of alkaline urine dehydration (for capture of N in the form of urea) and of struvite precipitation (capture of free ammonium nitrogen) and thus to recover the maximum amount of nutrients present in the urine.

3.2 Struvite

Struvite is a crystalline substance that can be in a form of potassium magnesium phosphate ($KMgPO_4$) or magnesium ammonium phosphate ($MgNH_4PO_4$) (Simha & Ganesapillai, 2017). Since the focus is capture of ammonium nitrogen, the precipitation of $MgNH_4PO_4$ is chosen (*Figure 8*). The formation of this type of struvite is shown in the chemical reaction equation below (Eq. 3) (Edahwati, et al., 2018).

$$Mg^{2+} + NH_4^{2+} + PO_4^{3-} + 6H_2O \rightarrow MgNH_4PO_4 * 6H_2O \quad (3)$$

From the chemical reaction, it is easy to understand that in order for struvite crystals to be formed, magnesium, ammonia and phosphate have to combine in water in a mole to mole to mole ratio of 1:1:1. However, the molar ratio of compounds in urine is 260:6:1 (NH_4^+:P:Mg) (Wilsenach, et al., 2007). Therefore, in order for ammonium nitrogen to be captured, a surplus of available Mg is needed (Yetilmezsoy & Sapci-Zengin, 2009).

The external addition of Mg can be in the form of MgO or $Mg(OH)_2$, either of which increases the pH, lowers the solubility of $(PO_4)^{3-}$ and induces supersaturation and spontaneous precipitation. By controlling the dosage of magnesium compounds and the pH of the urine, significant P and some N can be recovered (Simha & Ganesapillai, 2017).

Figure 5. Struvite crystals of magnesium ammonium phosphate (Mills, 2007)

Besides the magnesium compounds, a third substance this thesis investigated was calcium hydroxide ($Ca(OH)_2$) or lime. Lime is a low-cost alkalizing agent which is regularly applied to acidic agricultural soil (Simha, et al., 2020). It has been demonstrated to effectively increase the pH above 12 and to inhibit urease activity (Randall, et al., 2016).

Last, the inhibition of urease requires the pH to be above 10 (Dutta & Vinnerås, 2016). According to Wilsenach (2007), a pH of 9 or higher is crucial for complete struvite precipitation from urine. Since elevated pH is required in both of these scenarios and there are no conflicts, the combination of struvite precipitation and alkaline dehydration is theoretically possible.

Therefore, the experiment is about measuring the amount of captured ammonium nitrogen using magnesium compounds and comparing its efficiency to the known behaviour of a control sample of calcium hydroxide, while simultaneously applying the alkaline urine dehydration method.

3.3 Setup of the experiment and methods used

The experiment was performed at *Swedish University of Agricultural Sciences* in Uppsala, Sweden. The university's laboratory of *Energy and Technology* was used during a period of two months for execution of the experiment and for physiochemical analysis.

3.3.1 Urine collection and mixing.

Fresh human urine was collected every day from 15 volunteers (8 females and 7 males, aged 24-65 years old). The donations were gathered in polypropylene 0.5-L containers, which were strategically placed in the departmental bathrooms. Each container had a screw-on top and an inner lid to prevent ammonium nitrogen from escaping. The bottles were collected throughout the day when there were enough donations to perform the experiment (2-6 donors). The contents of the collected bottles were poured into a metal bucket (cleaned with ethanol) and stirred for proper mixing. From the mix, 20 ml of a sample was taken and placed into a Falcon tube (for future analysis of total-N and NH_4^+-N), which was then stored in a freezer.

The fresh urine was usually collected after 12:00 h and 16:00 h. The donations collected after 17:00 h were stored in the fridge and used the next day (urine older than 24 hours was not used). Prior to use, the refrigerated samples were first left to reach room temperature (21 °C ± 2 °C).

3.3.2 Alkalization of the urine

With a help of a funnel, 500 ml of the mixed urine was poured into a large beaker. The urine was stirred continuously throughout the whole alkalization process with

a help of a magnetic stirrer (VS-C7, VWR), stopping only to measure the pH (PHM210, Radiometer Copenhagen). Using a pipette, 1 M KOH solution was added to the fresh urine. After few minutes of stirring, the pH of the solution was measured. The addition of the 1 M KOH continued until the pH reached a value between 10 and 10.5. At the end, a 20-ml sample of the alkalized urine was pipetted into a Falcon tube and stored in a freezer.

3.3.3 Setup of the oven

For the experiment, a benchtop incubator (Electrolux, Sweden*)* with an inbuilt cavity of 470 x 330 x 580 mm was used. The temperature was adjustable, with a range from 30 to 60 °C. Before the experiment, additional adjustments were made to the drying system for good ventilation and airflow (*Figure 6*).

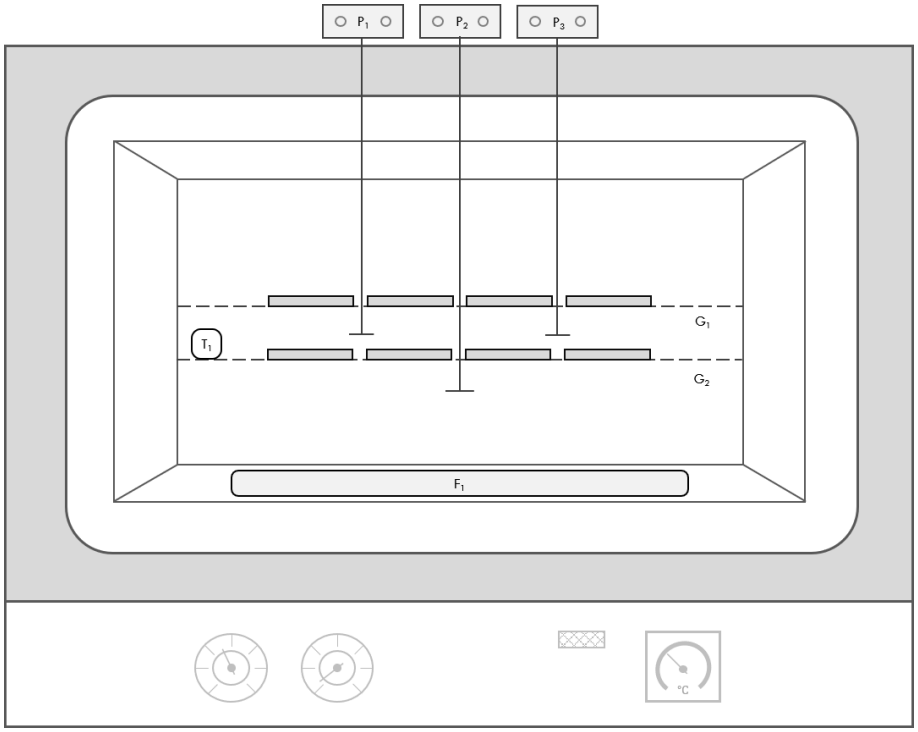

Figure 6. Schematic of the dehydration setup – air pumps (P1-P3), computer fan (F1), digital temperature monitor (T1) and grates (G1 and G2) with petri dishes containing the drying media (modified with permission from Simha, 2020)

A cooling fan (∅ 12'', 2300 rpm, Biltema) was installed at the bottom of the oven to direct the air upwards. Three electronic air pumps were inserted into three small holes at the top of the oven with the goal of introducing and directing the air

downwards, thus creating a circular drying environment (convective drying). Last, a temperature sensor (TGU-4500, Tinytag) was placed inside the oven to monitor temperature and relative humidity.

3.3.4 Setup of the petri dishes and drying media

Nine petri dishes were labelled and weighed (*Kern KB 200-2NM, Germany; 0.01g precision*). To each petri dish, 10 g of either MgO, $Mg(OH)_2$, or $Ca(OH)_2$ powdered compound was added (in triplicates). Then, 30 ml of distilled water was poured to cover the surface area and oven dried for approximately 24 hours. This was done to let the compounds settle and to prevent their loss during future addition of urine (10 ml of urine does not cover the whole surface area).

3.3.5 Urine additions and drying

For the additions, 10 ml of alkalized urine was pipetted onto each petri dish. The alkalized urine was continuously stirred during additions to trap any naturally formed precipitates and to keep the urine as homogenous as possible. Then, after the addition, the lids were closed (for ammonia to be captured, since without lids it may volatilize) for about 30 minutes for the urine to settle down and react with the compound. After half an hour, the lids were removed, and the petri dishes were placed inside the pre-heated oven for a duration of 2-3 hours. The temperature of the oven was chosen to be 40 °C to minimize the chemical hydrolysis of the urea and to avoid the need for optimization of the drying rate to higher temperatures. During drying, the door of the oven was left slightly ajar to vent out the humid air. The fresh urine was alkalized, added and dried 36 times. The experiment lasted 30 days in total.

3.4 Sampling and physiochemical analysis

Once the experiment was over (all 36 runs), the stored urine samples and the drying media were analyzed. The analysis was done for total-N, NH4-N, non-metals (P, S), metals (K, Ca, Na, Mn, Cu, Zn, Mg and Fe), total solids (TS), % ash, $pH_{1:5}$ and electrical conductivity ($EC_{1:5}$).

3.4.1 Stored urine samples

All 'fresh' urine samples, which were stored after each drying cycle (36 Falcon tubes), were taken out of the freezer and set aside to reach room temperature. The samples were then divided into 3 groups, in which each group had 12 Falcon tubes (runs 1-12, 12-24 and 24-36), and mixed. From each group mixture, duplicates were taken and used for further analysis.

The analysis of **total-N and NH_4-N** was done using Spectroquant® test kits following the instructions of the manufacturer (Merck KGaA, Darmstadt, Germany). For NH4-N analysis, urine was diluted 10-fold and analyzed using the Spectroquant® ammonium test kit with a detection range of 5-150 mg/L. To measure total-N, urine was first diluted 1000-fold and digested using Spectroquant® Crack-Set 20. After the digestion, the nitrate concentration was determined using a Spectroquant® nitrate test kit with a detection range of 1-25 mg/L. The standard deviation of the test kits is 5% (personal communication with Vinnerås, 2020).

The analysis of **metals and non-metals** was done by inductively coupled plasma–optical emission spectrometry (Optima Avio 200 optical emission spectrometer, PerkinElmer, USA) (for explanation of the method see Hou, et al., 2006).

3.4.2 Drying media

After the 12^{th}, 24^{th} and 36^{th} drying runs, 3 petri dishes were removed (1 per compound) and used in destructive physiochemical analysis. These petri dishes

were dried at 40 °C for additional amount of time until visibly dry. After dehydration and weighing of the petri dishes, the drying medium was transferred to a mortar and ground with a pestle until it became a powder.

Each drying medium was analyzed for **total N** using the DUMAS (10%) combustion method (OleiniTec, n.d.) on an elemental analyzer (LECO TruMac ® CN, USA), where standard deviation is 10 % (Simha, 2020). The analysis of **metals and non-metals** was also done by inductively coupled plasma–optical emission spectrometry.

Total solids were measured using an oven drying method at 105 °C (for explanation of the method see Ranjan & Rao, 2007), while **ash** was weighed after incineration at 550 °C for 6 h using a furnace (LH30/12, Nabertherm GmBH, Germany). **Electric conductivity** (Cond 340i, WTW, Germany) and **pH** were measured after the suspensions of drying media, and distilled water was mixed with medium at a ratio of 1:5 (medium:water) using a benchtop shaker and left to precipitate for an hour.

3.5 Calculations

Besides experimental recovery rates, the theoretical amount of nitrogen recovered by dehydrating urine was calculated. This was done using an equation (Eq. 4) proposed by Simha et al. (2020):

$$urea.rec_i = (urea.add_i + urea.rec_{i-1}) \times \left(1 - \frac{t}{2 \times h_{\frac{1}{2}}}\right), \quad (4)$$

Where, $\boldsymbol{urea.rec_i}$ [mg] is the theoretical amount of nitrogen recovered in the time period $\boldsymbol{i}$ [h], $\boldsymbol{urea.add_i}$ [mg] is the amount of nitrogen added, $\boldsymbol{urea.rec_{i-1}}$ [mg] is the amount of nitrogen recovered in the previous time period, $\boldsymbol{t}$ [h] is the time difference between the two periods and $\boldsymbol{h_{\frac{1}{2}}}$ [h] represents the half-life of urea (Eq. 5).

The estimation equation (Eq. 5) of the half-life of urea was also taken from Simha et al. (2020) and was calculated based on the parameters suggested by Warner (1942).

$$h_{\frac{1}{2}} = 1.352 \times 10^{6} \times e^{-(0.1257T)} \quad (5)$$

According to the equation, the half-life of urea in the pH range of 2-12 depends on the temperature *T* [°C]. Since the temperature of the drying system was 40 °C, it was calculated that the half-life of urea is 8858.2 hours (369 days).

The percent recovery $\boldsymbol{R}\,[\%]$ after ***n*** days of alkaline dehydration with struvite precipitations was calculated by using another equation (Eq. 6; Simha et al., 2020):

$$R[\%] = \left(\frac{urea.rec_n}{\sum_i^n urea.add_i}\right) \times 100 \quad (6)$$

The results from Eq. 6 were then compared to the obtained experimental values for nitrogen recovery.

4 RESULTS

After the physiochemical analysis and calculations were done, the results for the nutrient recovery (% recovery of different macronutrients), experimental and theoretical recovery of nitrogen (N), available free ammonium nitrogen and changes in physiochemical properties were obtained and are described below.

4.1 Mass balance and nutrient recovery

To estimate the efficiency of the dehydration treatment, a mass balance was carried out for the elements N, P, K, Ca, Mg, Na, S, Mn, Cu, Zn and Fe (*Appendix 1*). The focus was primarily on the recovery of N and other macronutrients (P, K, Ca, Mg, Na and S), since the rest of the nutrients (Zn, Mn, Cu and Fe) were present only in trace amounts.

The recovery rate (%) was calculated after drying runs 12, 24 and 36 for all three drying media. This amounted to the addition of 120, 240 and 360 ml of urine to 10 g of each drying medium. The results are shown and described in the *Table 1, Table 2* and *Table 3.*

Table 1. Recovery rate for elements during alkaline urine dehydration using MgO as a drying medium

Drying medium	Element	Recovery %		
		Drying Run 12	*Drying Run 24*	*Drying Run 36*
MgO	**N**	108%	111%	175%
	P	101%	98%	131%
	Ca	613%	352%	388%
	K	96%	102%	150%
	Na	102%	107%	159%
	S	104%	105%	143%
	Mg	59%	54%	67%

From the *Table 1*, it is clear that there was nearly complete recovery for most of the nutrients. Many recovery rates were calculated to be greater than 100%, especially Ca with recovery rate of more than 352% in all drying runs. In drying run

36, the recovery % of N, P, K, Na and S increased after previously being around to 100%. Magnesium recovery was between 54-67% for all of the drying runs.

Table 2. Recovery rate for elements during alkaline urine dehydration using $Mg(OH)_2$ as a drying medium

Drying medium	Element	Recovery %		
		Drying Run 12	*Drying Run 24*	*Drying Run 36*
$Mg(OH)_2$	**N**	110%	109%	127%
	P	87%	87%	105%
	Ca	99%	91%	115%
	K	91%	98%	110%
	Na	97%	99%	107%
	S	91%	96%	104%
	Mg	41%	45%	55%

In the *Table 2*, the first six elements have values which are between 87-110%, except for the values of N and Ca in drying run 36. The recovery rate of magnesium was around 41-55% for all drying runs.

Table 3. Recovery rate for elements during alkaline urine dehydration using $Ca(OH)_2$ as a drying medium

Drying medium	Element	Recovery %		
		Drying Run 12	*Drying Run 24*	*Drying Run 36*
$Ca(OH)_2$	**N**	107%	126%	130%
	P	82%	85%	100%
	Ca	87%	89%	104%
	K	97%	93%	109%
	Na	107%	98%	114%
	S	101%	103%	120%
	Mg	607%	324%	318%

The recovery values shown in *Table 3* for the $Ca(OH)_2$ medium are similar in some instances to those for MgO and $Mg(OH)_2$ (*Table 1, Table 2*). The recovery rates of nitrogen, potassium, sodium and sulfur are between 90% and 110%, except in drying runs 24 and 36 for N, and in run 36 for S and Na, where they were greater than 114%. The recovery rates for phosphorous and calcium for drying runs 12 and 24 were 80-90%, increasing to 100-104% in the last drying run. Compared to

results for MgO and $Mg(OH)_2$ media, however, magnesium recovery does not stay around 100% but jumps to a few hundred percent (>318%) for all the runs.

4.2 Experimental and theoretical recovery of nitrogen

Available nitrogen in the fresh urine for capture was calculated from the experimental total nitrogen and free ammonium nitrogen analysis (*Appendix 2*). The value calculated was 2.25 %, which is the amount of the free ammonium nitrogen lost during alkaline urine dehydration without struvite precipitation.

Table 4 compares experimental recovery values to calculated theoretical values for the three drying media (MgO, $Mg(OH)_2$, and $Ca(OH)_2$). It shows that for all three compounds, the recovery rate of nitrogen is very close to 100% across all 36 drying runs. The values range from having 107% to 175% of recovery. The percentage increased with each drying run, and there was no clear difference in recovery rates between the drying media.

Table 4. Experimental and theoretical recovery values of nitrogen during alkaline urine dehydration

Drying medium	Recovery rate (%)			
	Drying Run 12	***Drying Run 24***	***Drying Run 36***	**Theoretical**
MgO	108%	111%	175%	**99.90%**
$Mg(OH)_2$	110%	109%	127%	**99.79%**
$Ca(OH)_2$	107%	126%	130%	**99.98%**

The theoretically calculated values for recovery of N (calculated from *Equations 4-6)* were found to be ~100% for all media. Experimental recovery values were in a similar range to theoretically calculated percentages for the 12th (all compounds) and 24th (magnesium compounds) drying runs. Whereas, the experimental values for lime in the 24th and for all drying media in the 36th drying run, significantly differed from those predicted theoretically, with an increase in recovery (26-75%) much greater than the analytical method deviation (10%) would account for.

4.3 Changes in physiochemical properties

Measured pH and electrical conductivity (EC) are presented in the form of graphs. This way, the change observed throughout all 36 drying runs can be conveniently compared between the media.

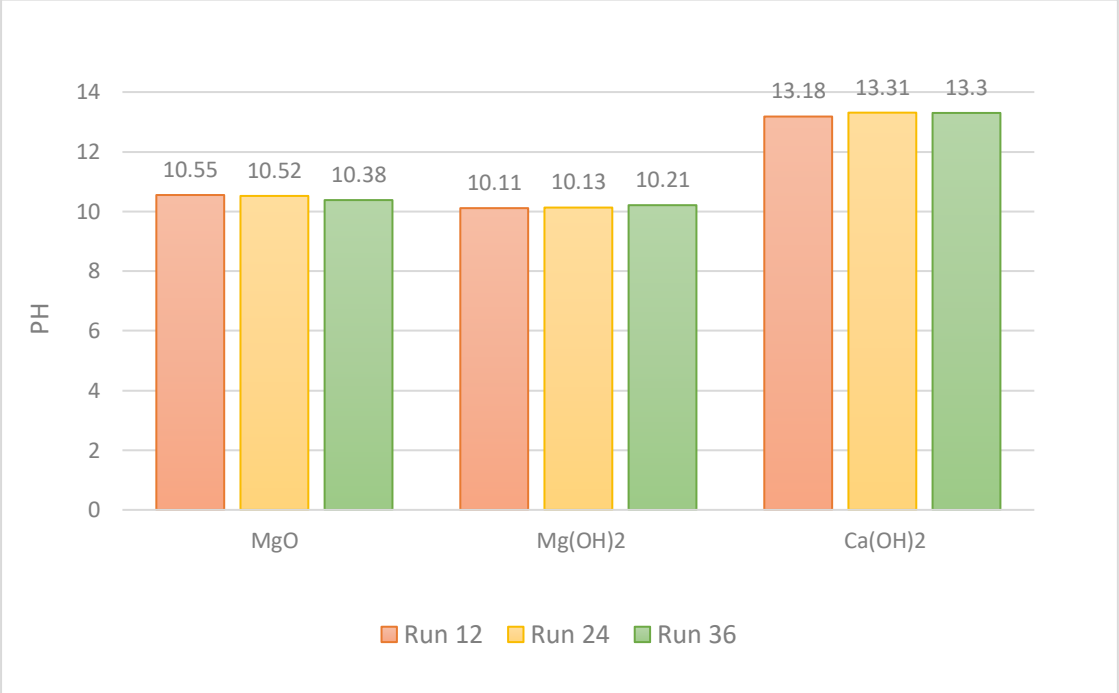

Figure 7. The pH values of different drying media after 12th, 24th and 36th drying runs

As shown in the figure above (*Figure 7*), there was only a minor change in pH throughout all 36 drying runs. The pH of all three media stayed above 10, with that of $Mg(OH)_2$ being the closest to the limit value. The pH values for MgO and $Mg(OH)_2$ were in a similar range (10.11-10.55), whereas the pH of $Ca(OH)_2$ was greater than 13, as expected.

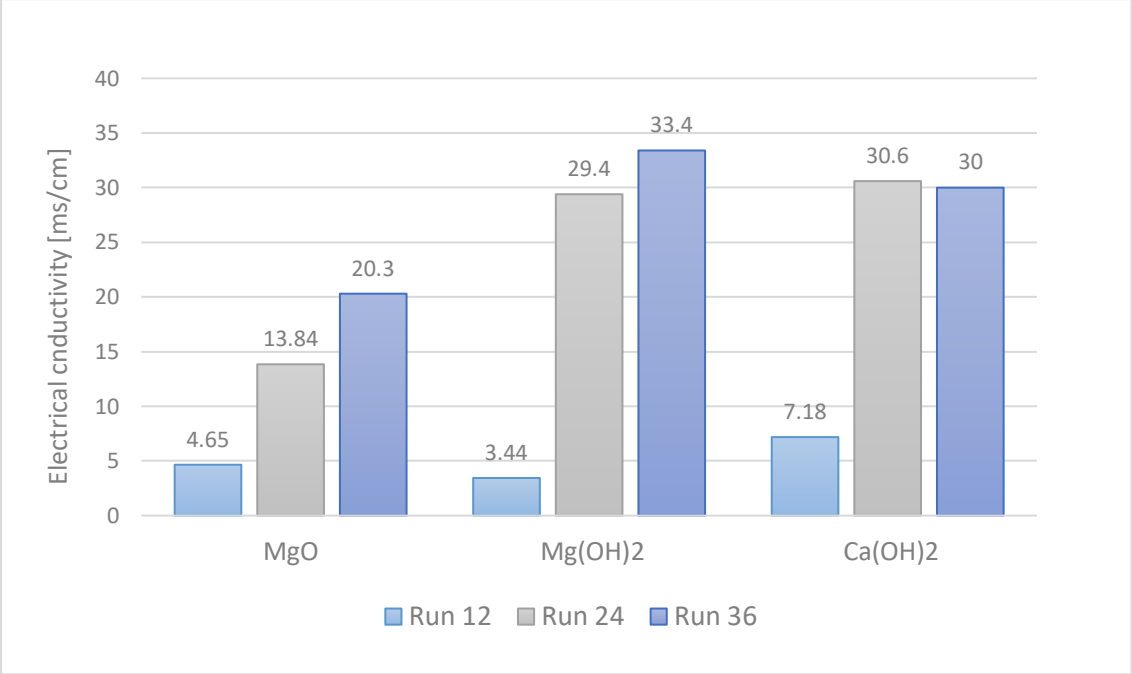

Figure 8. The values of electrical conductivity of different drying media after 12th, 24th and 36th drying runs.

As shown in the figure on electrical conductivity (*Figure 8*), the EC values increased with each drying run for magnesium compounds. For MgO, the increase was somewhat linear, whereas the EC of $Mg(OH)_2$ increased sharply after the 12th run. The EC values for lime only increased from 12th to 24th drying run (similarly to those for $Mg(OH)_2$), after which they slightly decreased by 0.6 mS/cm.

4.4 Fertilizer value

In the *Table 5*, results for the fertilizer value (N-P-K as % of dry matter) of the compounds after the 12th, 24th and 36th drying runs are shown *(calculations in Appendix 3*). The N-P-K values of the fertilizer produced from drying urine in MgO, $Mg(OH)_2$ and $Ca(OH)_2$ media were 11.3-0.5-6.1, 10.5-0.5-5.7 and 9.6-0.5-5.1, respectively, after 36 additions of urine.

Table 5. Fertilizer value for MgO, $Mg(OH)_2$ and $Ca(OH)_2$

Drying medium	Element	Fertilizer value		
		Drying Run 12	*Drying Run 24*	*Drying Run 36*
MgO	**N**	3.70%	6.10%	11.30%
	P	0.20%	0.30%	0.50%
	K	2.10%	3.50%	6.10%
Mg(OH)2	**N**	5.00%	7.70%	10.50%
	P	0.20%	0.40%	0.50%
	K	2.60%	4.40%	5.70%
Ca(OH)2	**N**	4.30%	7.60%	9.60%
	P	0.20%	0.30%	0.50%
	K	2.50%	3.50%	5.10%

The fertilizer value increased with increasing urine additions (12, 24 and 36 additions). The average N content of the fertilizer was 10.5%, whereas the average P and K contents were 0.5% and 5.6%, respectively.

5 DISCUSSION

In this chapter, the results are discussed in relation to the main research question and goals of the thesis – can free ammonium nitrogen lost during alkaline urine dehydration be captured by struvite precipitation, and which of magnesium based media trap it in the most efficient way.

5.1 Mass balance and nutrient recovery

The recovery rates for all three drying media are close to 100% for most of the nutrients. However, some of the values (e.g., 175% recovery) are much greater than would be explained by the standard deviation of the method. It is possible that some of these results are statistical outliers; this could be addressed by the use of multiple rather than single samples.

However, these high recovery rates do not apply to all of the nutrients. At 40 °C, there should not be volatilization of any of the elements besides sulfur and nitrogen (Simha, 2020), yet magnesium recovery rates for both magnesium compounds were around 50%. This occurrence is rather unusual, and further chemical analysis is needed to explain it.

Moreover, very high recovery rates were observed for Ca recovery in MgO medium and for Mg recovery in $Ca(OH)_2$ medium. This can be explained by impurity of the drying media, where these elements are already present in trace amounts after being manufactured (Vinnerås, 2020).

As for the efficiency of the drying media, it can be seen that the two hydroxide-based media have similar efficiencies. However, compared to them, MgO has the highest recovery rates for all the nutrients, where almost all their values are drastically above method deviation. Unfortunately, this does not indicate that this medium is indeed the most efficient. Its behaviour and the reason for these results

cannot be explained without additional chemical analysis that is outside the scope of this bachelor's thesis.

5.2 Capture of the free ammonium nitrogen

When trying to compare the effectiveness of compounds in capturing nitrogen, no significant difference between the compounds can be observed. Unfortunately, this is because the amount of available ammonium nitrogen in fresh urine was too little (only 2%), and it was hard to reliably identify differences with the methods used in this study (as the standard deviation of the methods was more than 5%).

My hypothesis was that the three compounds should differ in their recovery of nitrogen. Calcium hydroxide should recover the least amount of nitrogen, since it keeps the pH of urine above 12, which reduces the half-life of urea and increases its chemical hydrolysis. The two Mg compounds have different solubilities, which means they should provide different amounts of Mg^{2+} in solution when urine is added, should form different amounts of struvite and thus should capture different amounts of free ammonium nitrogen.

When comparing the recovery rates between the drying runs, a slight increase in the amounts can be observed. It is, however, hardly reasonable to compare the percentages larger than 100%. A possible explanation for such unusually high recovery rates is the use of different types of analysis methods for determining the amount of nitrogen in the samples and in the media (Simha, 2020). For the fresh urine samples, the Spectroquant® test kits were used; they had a standard deviation of 5%. The method used for the analysis of the total nitrogen for the drying media was the DUMAS combustion method, which has a deviation of 10%. This poses an issue because the internal deviation of the methods is unclear, which makes it impossible to consistently compare the analyzed results.

However, the measured nitrogen recovery is close to that predicted by theoretical calculations. Therefore, more accurate analytical methods and triplicate experiments are expected to yield similar results.

5.3 Changes in physiochemical properties

In *Figure 7 and Figure 8*, the changes in physiochemical properties for all three drying media (throughout all 36 drying runs) can be observed.

All three drying media managed to maintain a pH greater than 10 (*Figure 7)*. These are good results, since they show that the enzymatic hydrolysis of the urease was successfully prevented throughout the urine drying process. It can be concluded that all tested media are suitable for alkalization of urine and therefore should have been able to capture that 2% of ammonium nitrogen in the form of struvite precipitate.

There was a visible increase of electrical conductivity values with each drying run for all media (*Figure 8)*. It can be explained by the fact that the concentrations of urine salts in the petri dish increased with each urine addition, which then led to a higher electrical conductivity of the end product.

However, the increase in the electrical conductivity should be linear, which was not the case for the hydroxide-based compounds. One of the reasons could be the method used, since the analysis of the electrical conductivity for the first 12 runs was done the day after the sample was allowed to precipitate.

Moreover, electrical conductivity values for the 24^{th} and 36^{th} drying runs in lime differ only by 0.6 mS/cm. One possible explanation for this occurrence could be oversaturation of the solution, resulting in the observed plateau. Unfortunately, the actual reason for such behaviours is unknown and replicate (at least triplicate) experiments should help resolve this anomaly.

5.4 Fertilizer value and suggestions for future research

The fertilizer values of the end product (as % N-P-K) obtained from my experiments are comparable to those of a conventional synthetic fertilizer. The fertilizer value of dehydrated urine was found to be between 9.6-0.5-5.1 and 11.3-0.5-6.1, which is much higher than those of human urine (0.7-0.06-0.2 according to Vinnerås, et al., 2006) and liquid dairy manure (0.4-0.1-0.25 according to Brown, 2013).

It can also be compared to the values for blended fertilizers used in Sweden, which were 21-4-7 for cereal crops and 11-5-18 for vegetables, fruits and berries (Yara, u.d.). When comparing the N-P-K values of the MgO medium to those of the blended fertilizer for vegetables, it can be concluded that the nitrogen content of the drying medium actually meets the requirements of the blended fertilizer for the vegetables. However, to produce a more balanced fertilizer, it has to be supplemented with additional P and K or blended with other substrates, such as ash. Based on this comparison, it can be concluded that the alkaline dehydration does recover and concentrate enough nutrients to produce an effective high-value dry fertilizer and substitute (at least partially) for synthetic and blended fertilizers.

In order to see further advancement in the direction of full nutrient recovery with ammonium capture and increase in fertilizer efficiency, several adjustments to the research method described here should be made.

My first suggestion is to increase the amount of freely available ammonium nitrogen in the fresh urine by external addition of soluble ammonia before the dehydration process. By applying this method, the effectiveness of capture of known amounts of ammonia (higher than the method deviation) would be more accurately assessed.

Another suggestion is to utilize the same nutrient analysis method for both fresh urine samples (before dehydration) and drying medium samples (after dehydration). This would give results which could be more accurately compared, thus leading to more reliable data on recovery rates.

Moreover, the dehydration medium composition should be analyzed as well. This way, known amounts of the trace elements present in the medium can be determined, and a more complete mass balance can be done.

Last, there should be an increase in the number of the tested samples per drying run. Instead of one petri dish for each destructive analysis, I suggest using at least triplicates. This way, more statistically reliable results can be calculated, and a more accurate relationship between the drying method and the nitrogen capture (and other nutrient recoveries) can be determined.

6 CONCLUSION

Recycling of human urine can produce many benefits and positive impacts on the world by providing safe sanitation and reducing the load on the wastewater treatment plants, as well as by providing sustainable fertilizer for agriculture. When it comes to fertilizer production, there are several technologies that are used to recycle nutrients found in urine. When compared to other methods of urine treatment, one of the most effective methods is alkaline urine dehydration, which was developed at Swedish University of Agricultural Sciences. This method uses alkalization techniques paired with dehydration to produce effective dry fertilizer with a high nutrient value that can at least partially replace synthetic fertilizers.

One of the setbacks of the method is that there is still some nitrogen lost during the drying process in the form of free ammonium nitrogen. The experiment described in this thesis aimed to capture this lost nitrogen by combining alkaline dehydration and struvite precipitation. The results of the experiment showed that there is almost complete nutrient recovery, the end product of which can be used in a form of a very highly concentrated dry organic fertilizer. However, the success of capture of the free ammonium nitrogen is unclear due to the low amount of available ammonium nitrogen for capture and the standard deviation of the methods. Even though the results of this experiment were inconclusive, it provides important preliminary data about this dehydration method. Therefore, it provides a necessary base for future research towards the objective of complete nutrient capture and creation of more sustainable fertilizers from safe and affordable sanitation systems.

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MASS BALANCE AND RECOVERY RATE CALCULATIONS

The mass balance can be described as an equilibrium between the input values before the treatment and the output values after the treatment. In the case of the experiment described in this thesis, the balance can be shown as:

$$\text{Medum} + \text{Urine} + \text{KOH solution} \xrightarrow{\text{Dehydration}} \text{End product}$$

In theory, the mass of the inputs pre-dehydration should be equal to the mass of the outputs post-dehydration. This, however, is often not the case in many experimental setups due to sampling errors, deviation of analytical measurement methods, losses of the compounds during the experiment, etc.

The calculations for the amount of each element in the end product were done by following equation:

$$\boldsymbol{m_{end\,(i)} = c_{end\,(i)} \times ww_i,}$$

Where,	$\boldsymbol{m_{end}}$	amount of the element in the end sample	[g]
	$\boldsymbol{c}$	concentration of the element in the end sample	[mg/kg]
	$\boldsymbol{ww_i}$	wet weight of a sample	[g]
	$\boldsymbol{i}$	drying round (12, 24 or 36)	[-]

After which, recovery rates were calculated as:

$$\boldsymbol{R_x = \frac{m_{end\,(i)}}{m_{1(i)} + m_{2(i)} + m_{3\,(i)}} \times 100\ \%}$$

Where	$\boldsymbol{R}$	recovery rate for the element	[g]
	$\boldsymbol{m_{end(i)}}$	amount of an element in the end-product	[g]
	$\boldsymbol{m_{1(i)}}$	amount of an element in the media	[g]
	$\boldsymbol{m_{2(i)}}$	amount of an element in the fresh urine	[g]
	$\boldsymbol{m_{3(i)}}$	amount of an element in KOH solution	[g]
	$\boldsymbol{i}$	drying round (12, 24 or 36)	[-]

The mass balances and recovery rates for all nine tested samples are shown on the following pages.

Mass balance for MgO - Drying run 12

	Medium [mg]	Urine [mg]	KOH [mg]	SUM [mg]	DRYING		*Wet weight [g] 36.604* End product c [mg/kg]	End product m [mg]	Recovery
N		642.0000		642.0000		N [%]	1.90	694.74	**108%**
P		38.2257		38.2257		P	1057.26	38.70	**101%**
Ca		11.6774		11.6774		Ca	1954.97	71.56	**613%**
K		203.8036	201.37	405.1728		K	10578.55	387.22	**96%**
Na		161.6538		161.6538		Na	4496.34	164.58	**102%**
Mn		0.0003		0.0003		Mn	1.76	0.06	19382%
Cu		0.0066		0.0066		Cu	5.62	0.21	3100%
Zn		0.0256		0.0256		Zn	<0.002	<0.002	N/A
S		38.5508		38.5508		S	1092.76	40.00	**104%**
Mg	6035.604977	6.1765		6041.7814		Mg	97654.08	3574.52	**59%**
Fe		0.0263		0.0263		Fe	18.26	0.67	2543%

Mass balance for MgO - Drying run 24

	Medium [mg]	Urine [mg]	KOH [mg]	SUM [mg]	DRYING		*Wet weight [g] 35.436* End product c [mg/kg]	End product m [mg]	Recovery
N		1242.0000		1242.0000		N [%]	3.89	1377.44	**111%**
P		75.7794		75.7794		P	2102.73	74.51	**98%**
Ca		22.6474		22.6474		Ca	2248.69	79.69	**352%**
K		380.9786	404.30	785.2779		K	22511.35	797.72	**102%**
Na		346.3712		346.3712		Na	10462.49	370.75	**107%**
Mn		-0.0016		-0.0016		Mn	1.36	0.05	-2938%
Cu		0.0121		0.0121		Cu	2.06	0.07	604%
Zn		0.0376		0.0376		Zn	0.519	0.02	49%
S		76.9682		76.9682		S	2283.05	80.90	**105%**
Mg	6034.64	12.5737		6047.2138		Mg	92687.71	3284.50	**54%**
Fe		0.0304		0.0304		Fe	17.87	0.63	2085%

Mass balance for MgO - Drying run 36

	Medium [mg]	Urine [mg]	KOH [mg]	SUM [mg]	DRYING		*Wet weight [g] 40.006* End product c [mg/kg]	End product m [mg]	Recovery
N		1836.0000		1836.0000		N [%]	8.05	3218.98	**175%**
P		113.8760		113.8760		P	3716.81	148.70	**131%**
Ca		33.7070		33.7070		Ca	3265.97	130.66	**388%**
K		556.8884	600.20	1157.0934		K	43511.23	1740.72	**150%**
Na		506.3257		506.3257		Na	20083.46	803.46	**159%**
Mn		-0.0036		-0.0036		Mn	1.65	0.07	-1820%
Cu		0.0179		0.0179		Cu	0.13	0.01	29%
Zn		0.0528		0.0528		Zn	<0.002	<0.002	N/A
S		117.2851		117.2851		S	4192.12	167.71	**143%**
Mg	6031.685205	18.9761		6050.6614		Mg	101571.45	4063.49	**67%**
Fe		0.0341		0.0341		Fe	23.43	0.94	2750%

Mass balance for Mg(OH)2 - Drying run 12									
							Wet weight [g] 14.776		
	Medium [mg]	Urine [mg]	KOH [mg]				End product		
				SUM [mg]			c [mg/kg]	m [mg]	Recovery
N		642.0000		642.0000	DRYING	N [%]	4.77	704.39	**110%**
P		38.2257		38.2257		P	2250.26	33.25	**87%**
Ca		11.6774		11.6774		Ca	783.85	11.58	**99%**
K		203.8036	201.37	405.1728		K	25049.06	370.11	**91%**
Na		161.6538		161.6538		Na	10654.44	157.42	**97%**
Mn		0.0003		0.0003		Mn	2.23	0.03	9930%
Cu		0.0066		0.0066		Cu	2.19	0.03	488%
Zn		0.0256		0.0256		Zn	<0.002	<0.002	N/A
S		38.5508		38.5508		S	2384.13	35.23	**91%**
Mg	4174.119819	6.1765		4180.2963		Mg	117033.91	1729.23	**41%**
Fe		0.0263		0.0263		Fe	8.98	0.13	505%

Mass balance for Mg(OH)2 - Drying run 24									
							Wet weight [g] 18.318		
	Medium [mg]	Urine [mg]	KOH [mg]				End product		
				SUM [mg]			c [mg/kg]	m [mg]	Recovery
N		1242.0000		1242.0000	DRYING	N [%]	7.37	1350.00	**109%**
P		75.7794		75.7794		P	3608.21	66.10	**87%**
Ca		22.6474		22.6474		Ca	1128.16	20.67	**91%**
K		380.9786	404.30	785.2779		K	41867.84	766.95	**98%**
Na		346.3712		346.3712		Na	18721.24	342.94	**99%**
Mn		-0.0016		-0.0016		Mn	1.88	0.03	-2093%
Cu		0.0121		0.0121		Cu	1.37	0.03	208%
Zn		0.0376		0.0376		Zn	1.913	0.04	93%
S		76.9682		76.9682		S	4052.43	74.23	**96%**
Mg	4168.70	12.5737		4181.2756		Mg	103351.94	1893.23	**45%**
Fe		0.0304		0.0304		Fe	7.87	0.14	475%

Mass balance for Mg(OH)2 - Drying run 36									
							Wet weight [g] 23.329		
	Medium [mg]	Urine [mg]	KOH [mg]				End product		
				SUM [mg]			c [mg/kg]	m [mg]	Recovery
N		1836.0000		1836.0000	DRYING	N [%]	9.99	2331.23	**127%**
P		113.8760		113.8760		P	5111.21	119.24	**105%**
Ca		33.7070		33.7070		Ca	1654.67	38.60	**115%**
K		556.8884	600.20	1157.0934		K	54422.64	1269.62	**110%**
Na		506.3257		506.3257		Na	23225.03	541.81	**107%**
Mn		-0.0036		-0.0036		Mn	30.59	0.71	-19694%
Cu		0.0179		0.0179		Cu	2.22	0.05	289%
Zn		0.0528		0.0528		Zn	0.520	0.01	23%
S		117.2851		117.2851		S	5239.78	122.24	**104%**
Mg	4171.369191	18.9761		4190.3453		Mg	98382.28	2295.15	**55%**
Fe		0.0341		0.0341		Fe	7687.18	179.33	526112%

Mass balance for Ca(OH)2 - Drying run 12									
							Wet weight [g] 16.788		
	Medium [mg]	Urine [mg]	KOH [mg]				End product		
				SUM [mg]			c [mg/kg]	m [mg]	Recovery
N		642.0000		642.0000	DRYING	N [%]	4.08	684.31	**107%**
P		38.2257		38.2257		P	1866.17	31.33	**82%**
Ca	5413.842188	11.6774		5425.5196		Ca	281196.86	4720.73	**87%**
K		203.8036	201.37	405.1728		K	23353.82	392.06	**97%**
Na		161.6538		161.6538		Na	10258.71	172.22	**107%**
Mn		0.0003		0.0003		Mn	66.03	1.11	333545%
Cu		0.0066		0.0066		Cu	<0.002	<0.002	N/A
Zn		0.0256		0.0256		Zn	0.368	0.01	24%
S		38.5508		38.5508		S	2329.51	39.11	**101%**
Mg		6.1765		6.1765		Mg	2234.28	37.51	**607%**
Fe		0.0263		0.0263		Fe	153.62	2.58	9812%

Mass balance for Ca(OH)2 - Drying run 24									
							Wet weight [g] 22.934		
	Medium [mg]	Urine [mg]	KOH [mg]				End product		
				SUM [mg]			c [mg/kg]	m [mg]	Recovery
N		1242.0000		1242.0000	DRYING	N [%]	6.85	1569.99	**126%**
P		75.7794		75.7794		P	2819.71	64.67	**85%**
Ca	5414.60	22.6474		5437.2469		Ca	210555.41	4828.94	**89%**
K		380.9786	404.30	785.2779		K	31928.69	732.26	**93%**
Na		346.3712		346.3712		Na	14741.67	338.09	**98%**
Mn		-0.0016		-0.0016		Mn	47.36	1.09	-66073%
Cu		0.0121		0.0121		Cu	<0.002	<0.002	N/A
Zn		0.0376		0.0376		Zn	3.580	0.08	218%
S		76.9682		76.9682		S	3447.39	79.06	**103%**
Mg		12.5737		12.5737		Mg	1778.18	40.78	**324%**
Fe		0.0304		0.0304		Fe	111.80	2.56	8440%

Mass balance for Ca(OH)2 - Drying run 36									
							Wet weight [g] 26.699		
	Medium [mg]	Urine [mg]	KOH [mg]				End product		
				SUM [mg]			c [mg/kg]	m [mg]	Recovery
N		1836.0000		1836.0000	DRYING	N [%]	8.91	2377.72	**130%**
P		113.8760		113.8760		P	4250.42	113.48	**100%**
Ca	5411.786676	33.7070		5445.4936		Ca	211130.14	5636.98	**104%**
K		556.8884	600.20	1157.0934		K	47266.87	1261.98	**109%**
Na		506.3257		506.3257		Na	21635.60	577.65	**114%**
Mn		-0.0036		-0.0036		Mn	48.51	1.30	-35736%
Cu		0.0179		0.0179		Cu	<0.002	<0.002	N/A
Zn		0.0528		0.0528		Zn	0.932	0.02	47%
S		117.2851		117.2851		S	5288.55	141.20	**120%**
Mg		18.9761		18.9761		Mg	2256.87	60.26	**318%**
Fe		0.0341		0.0341		Fe	122.21	3.26	9572%

AMMONIUM NITROGEN CALCULATION

The data in the table below shows the values obtained for total nitrogen and ammonium- nitrogen for all 36 drying runs by using Spectroquant® test kits.

	Total - N [g/L]	NH4-N [g/L]
Urine used in Runs 1-12	5.30	<0.002
	5.40	0.108
Urine used in Runs 13-24	5.10	0.116
	4.90	0.112
Urine used in Runs 25- 36	5.80	0.123
	4.10	<0.002
Average	**5.10**	**0.115**

From the table, the percentage of the available ammonium nitrogen is calculated as a ratio between the average concentrations of total nitrogen and ammonium nitrogen:

$$\%\ ammonium\ nitrogen = \frac{c\ (NH_4^+ - N) \times 100}{c\ (Total - N)} = \frac{0.115\ [g/l] \times 100}{5.10\ \ [g/l]} = 2.25$$

Where $c\ (NH_4^+ - N)$ concentration of ammonium-nitrogen [g/l]
$c\ (Total - N)$ concentration of total nitrogen [g/l]

From which, the urea-N content $\boldsymbol{U}$ of urine can estimated as:

$$\boldsymbol{U} = 100\ \% - 2.25\ \% = \mathbf{97.75}\ \%$$

FERTILIZER VALUE CALCULATIONS

To get the fertilizer value for all 36 drying rounds, I did the following calculations, starting with the percentage of total solids:

$$\boldsymbol{TS_i = \frac{m_{x,i}}{m_{y,i}} \times 100\ \%,}$$

Where	$\boldsymbol{TS}$	total solids of a sample	[%]
	$\boldsymbol{m_x}$	sample weight before oven drying	[g]
	$\boldsymbol{m_y}$	sample weight after oven drying	[g]
	$\boldsymbol{i}$	drying round (12, 24 or 36)	[-]

Then, calculations for dry weight (dw_i) of a sample were done:

$$\boldsymbol{dw_i = ww_i \times TS_i,}$$

Where	$\boldsymbol{dw_i}$	dry weight of a sample	[g]
	$\boldsymbol{ww_i}$	wet weight of a sample	[g]
	$\boldsymbol{TS}$	total solids of a sample	[-]
	$\boldsymbol{i}$	drying round (12, 24 or 36)	[-]

After which, the amount of urea was calculated using the following formula:

$$\boldsymbol{m_{urea} = \frac{U \times m_{end-N}}{a},}$$

Where	$\boldsymbol{m_{urea}}$	mass of urea	[g]
	$\boldsymbol{U}$	percentage of urea in urine	[-]
	$\boldsymbol{m_{end-N}}$	amount of nitrogen in the end sample	[g]
	$\boldsymbol{a}$	molar percent of elemental N in urea	[-]

The value of U can be found in Appendix 2. Calculated values for m_{end-N} are shown in Appendix 1. The molar percent of elemental N in urea is 0.46.

From this, the adjusted dry weight was determined:

$$\boldsymbol{dw_a = m_{urea} + dw_i}$$

Where			
	$\boldsymbol{dw_a}$	adjusted dry weight	[g]
	$\boldsymbol{m_{urea}}$	mass of urea	[-]
	$\boldsymbol{dw_i}$	dry weight of a sample	[g]
	$\boldsymbol{i}$	drying round (12, 24 or 36)	[-]

Lastly, from the adjusted dry weight, fertilizer values were determined as a ratio between the amount of the element in the end sample (*see Appendix 1)* and the adjusted dry weight:

$$\boldsymbol{X_i = \frac{m_{end}}{dw_a} \times 100\ \%}$$

Where			
	$\boldsymbol{X}$	fertilizer value of the sample	[%]
	$\boldsymbol{i}$	drying round (12, 24 or 36)	[-]
	$\boldsymbol{m_{end}}$	N, P or K amount in the end sample	[g]
	$\boldsymbol{dw_a}$	adjusted dry weight	[g]

The fertilizer values for each drying medium can be seen in table below.

Drying medium	Run no.	% TS	*dw* [g]	Urea [g]	dw_a [g]	Fertilizer value		
						N	P	K
MgO	**12**	47%	17.37	1.48	18.85	**3.7%**	**0.2%**	**2.1%**
	24	56%	19.77	2.93	22.70	**6.1%**	**0.3%**	**0.4%**
	36	54%	21.77	6.84	28.61	**11.3%**	**0.5%**	**6.1%**
$Mg(OH)_2$	**12**	85%	12.63	1.50	14.13	**5.0%**	**0.2%**	**2.6%**
	24	80%	14.59	2.87	17.46	**7.7%**	**0.4%**	**4.4%**
	36	74%	17.16	4.95	22.11	**10.5%**	**0.5%**	**5.7%**
$Ca(OH)_2$	**12**	85%	14.30	1.45	15.75	**4.3%**	**0.2%**	**2.5%**
	24	76%	17.36	3.34	20.70	**7.6%**	**0.3%**	**3.5%**
	36	74%	19.72	5.05	24.78	**9.6%**	**0.5%**	**5.1%**