Optimisation of a low-cost urine treatment system for resource recovery

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Executive summary

Today, over 800 million people are estimated to be chronically undernourished and 2.5 billion live without basic sanitation facilities (FAO, IFAD and WFP, 2014; UN Water, 2014). Current sanitation challenges require new alternatives to conventional sewerage systems. Among them, the reuse of source-separated urine could help mitigate poverty and malnutrition by providing an in-country supply of fertiliser.

Historically urine has been applied as a fertiliser to a variety of crops in numerous countries including Japan, Yemen and Sweden (Schönning, 2001). Scientific research as well as development projects have studied and demonstrated the efficiency of urine reuse in agriculture (Richert *et al.*, 2010). Lately, research has focused on nutrient recovery from urine as struvite (Wilsenach *et al.*, 2007). However, studies on the recovery of urea from urine are scarce although urea constitutes the main source of nitrogen in urine. Therefore this study aims to provide an understanding of the potential for nutrient recovery from urine as urea and recommendations of practices based on experimental work.

Sponsored by WaterAid UK, this project aimed to test a new low-cost solution for urine management in developing countries based on the production of urea using solar powered evaporation for use as a fertiliser. This study investigated the performance of urine evaporation in producing urea, leading to recommendations on the design and broader application of the system and on its viability as a business model. As concluding remarks, the project assessed the relevance of the proposed system in the production of urine derived-urea for agricultural purposes in developing countries.

Preliminary analyses of urine chemical composition were carried out in order to compare the results with the literature review. Five samples of one-litre urine were evaporated using an experimental set-up under different conditions. In particular, the influence of pasteurisation and the design characteristics of the prototype (surface area and height of the light) on the chemical composition of the final product and its stability were considered. The results suggest that pasteurisation of urine impacts the initial chemical characteristics and crystallisation process. Ventilation and temperature were also identified as a parameter of major impact on the speed of crystallisation of urine. With a view to a possible implementation, the mass of recovered urea crystals, around 13 g of dry product per litre of urine, appears as a limitation to this technique.

Although the recovery of urea by solar pasteurisation and evaporation requires little maintenance and low-cost materials, and is particularly adapted for developing countries, our work showed that urine evaporation is a slow process producing limited amounts of urea. In addition, the end product appeared very unstable as it easily absorbs atmospheric moisture. Our findings suggest that the implementation of the current system is not viable. Further research is specifically required to optimise the process in terms of yield and quality/stability of the final product.

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Table of contents

List of figures

List of tables

List of abbreviations

AAS	Atomic Absorption Spectroscopy
COD	Chemical Oxygen Demand
DAP	Di-Ammonium Phosphate
IBDU	Isobutylaldehyde-Diurea
IBU	Isobutyricaldehyde
N	Nitrogen
Total P	Total phosphorus
XRD	X-Ray Diffraction

1 Introduction

The work presented in this report is the result of a 2-month group project as part of Cranfield University MSc "Community Water and Sanitation". Sponsored and commissioned by WaterAid UK, this work followed an innovative plan for urine reuse in Tanzania.

1.1 Background of project

The Society of Public Health Engineers (SoPHE) runs an annual "Young Engineers Award" competition. The 2013 competition was aiming to find an innovative solution to the problem of urine management and reuse from urine-separating toilets in developing countries. The two winners, consultant engineers Paul Foulds and Ivana Rusnakova, proposed a low-cost system for the production of urea crystals from urine, for potential reuse as a fertiliser.

The proposed system combined solar powered urine pasteurisation and evaporation units (Figure 1). After separation of urine and storage, the urine is pasteurised by flowing through a pipe heated using solar energy placed on a black metallic plate, (a). When the pasteurisation temperature is reached (i.e. 72 °C), a valve opens (b) to allow the urine to flow into the evaporation trough (c). Water then evaporates by solar energy and high temperatures generated by a greenhouse effect below the glass panel, until evaporation and recovery of the dry end-product occur. Liquid should condense on the inclined glass and collect in the water recovery trough.

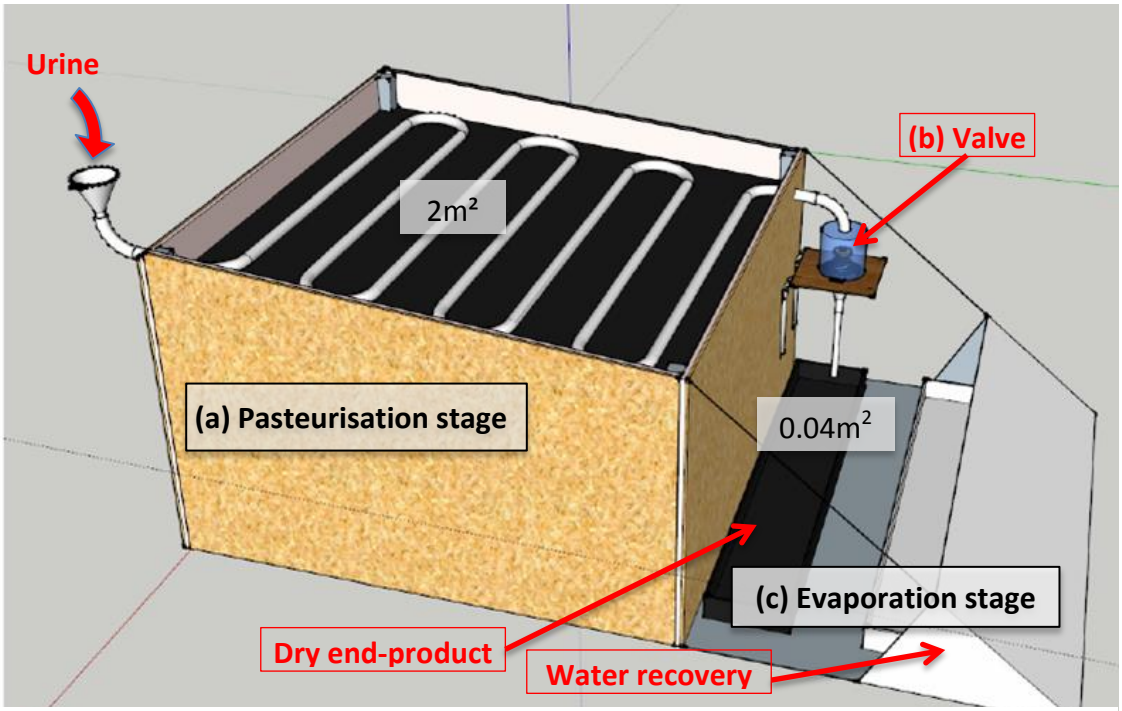


Figure 1: 2013 'Young Engineers Award' winning prototype of urine treatment (courtesy of Paul Foulds) - Dimensions are given for a system designed for 2 persons

1.2 Relevance of the system for Tanzania

Like many Sub-Saharan African countries, Tanzania is facing important challenges in the agricultural sector which represents around 25% of the country's GDP, providing work for 75% of the population (MAFAP, 2013). In particular, one of the agricultural sector's current goals is to increase productivity to achieve sustainable economic growth and development, as well as to mitigate poverty and malnutrition. Isaac (2005) reports that Tanzania's low-productivity is, among other factors, due to low soil fertility worsened by a lack of nutrient replenishment.

As mineral fertilisers have been proved to be the most influential factor to improve yield (Isaac, 2005), confronting the problems of their supply and access is a major challenge for agricultural productivity. In particular, urea is a popular fertiliser in Tanzania, due to a government subsidy set up in 2003 for urea and di-ammonium phosphate (DAP) fertiliser options. In 2010 those two fertilisers accounted for half of all fertilisers used in Tanzania (Ariga and Heffernan, 2012).

A visit to Tanzania in April 2014 explored the potential implementation of the system by meeting the main stakeholders as a first assessment of the technical feasibility, economic viability and effective demand from users and industry. The WaterAid team visited the Rufiji District and Temeke Municipality, and had discussions with the relevant authorities, engineering company EEPCO, and potential users. The findings showed that there was significant interest in the proposed process, especially from a business perspective. It also highlighted schools as a potential location to implement the project.

During this field visit, WaterAid also learnt that the current fertilisers used in the country were compost and commercial fertilisers such as phosphorus-based compounds and urea. While phosphorus-based fertilisers are produced by a local factory in Arusha, urea is mainly imported from China. Given a high requirement of fertiliser, transport is a challenge to reach a sustainable agricultural productivity and can add up to 40% to the final price of fertilisers (Ariga and Heffernan, 2012). Although ecological sanitation or urine diverting toilets are not current practice in Tanzania, there has been interest in on-site production of urea from urine reuse at a medium-scale.

1.3 Objectives of the project

The project aimed to assess the viability of such a practice for in-country production of urea from urine reuse and more specifically its potential for implementation in Tanzania.

Consequently, several parameters and factors were taken into account to provide guidelines for the design brief. The objectives were:

- To design a low-cost system that could easily be reproduced
- To take into consideration local conditions including parameters such as weather and temperature.

  Temperature values were chosen as the average of the measurements at three climatic stations Dar-Es-Salam, Dar-Es-Salam Airport and Ubunga, 30 km far from Temeke Municipality.

  Data were taken from the FAO software ClimWat (Water Development and Management Unit, 2013) and gave a minimum yearly temperature of 18.5°C in July and August and a maximum of 31.9°C in December.

  Full table is available in Appendix 1.

- To evaluate a business model for the system based on the local culture and social acceptance as well as current farming practices.

2 Understanding of nutrient recovery from urine and urea crystallisation process

2.1 Principal characteristics of urine

Each individual produces 1 to 1.5 L of urine per day (Corwin, 2011) with an average of 500 L urine per year (Richert *et al.*, 2010). However, children typically produce half this amount of urine.

Human urine is a liquid composed of 95% water. Other components include urea, electrolytes (such as sodium, potassium, ammonium, calcium, chloride, sulfate, phosphate and bicarbonate), creatinine and other organic compounds.

Although the nutrient content depends on the diet (Richert *et al.*, 2010), nitrogen concentrations in urine typically range from 7 to 9 g/L (**Erreur ! Source du renvoi introuvable.**). With an average concentration of 16.3 g/L, urea accounts for 75 to 90 % of the total nitrogen, while only 7 % of it is ammonia (Karak and Bhattacharyya, 2011, Dutta, 2012).

Table 1: Physico-chemical characteristics of urine

Category	Component	Range (g/L)			Reference
		Average	Min	Max	
General compounds	Water (H_20)				
	Total solutes	41.7	36.7	46.7	(Putman, 1971)
Electrolytes	Calcium (Ca^{2+})	0.2	0	0.4	
	Chloride (Cl^-)	5.1	1.9	8.4	
	Magnesium (Mg^{2+})	0.1	0	0.2	
	Phosphorus total (P)	0.8	0.5	1.1	
	Potassium (K^+)	1.7	0.8	2.6	
	Sodium (Na^+)	2.8	1.2	4.4	
	Sulfur, total (S)	1.3	0.2	2.3	
Other nitrogenous substances	Nitrogen total (N)	8	7	9	(Kirchmann and Pettersson, 1995)
	Ammonia (NH_3)	0.5	0.2	0.7	(Putman, 1971)
	Urea ($CO(NH_2)_2$)	16.3	9.3	23.3	
Other parameters	COD	7.53			
	Conductivity (mS/cm)	11.5	14.8	25.4	(Karak and Bhattacharyya, 2011)
	Colour	Transparent to yellow			
	pH	6.4	4.8	8.2	(Dutta, 2012; Putman, 1971)

The pH of fresh urine varies from 4 to 8 (Table 1), and can increase up to 9-9.3 when urease, a natural enzyme present in urine, hydrolyses urea resulting in the formation of ammonium (Dutta, 2012).

Concentrations of heavy metals in urine (eg. lead, cadmium, mercury) vary greatly from one study to another. Copper concentration reported by Jönsson *et al.* (2005) ranges from 0.047 to 60 mg/pp/d and zinc concentration is of 0.3 mg/pp/d. In this study, lead and cadmium concentrations were lower than 10^{-3} mg/pp/d. Overall, concentrations of heavy metals would be 10% lower than that of farmyard manure and commercial fertilisers (Karak and Bhattacharyya, 2011; Winker, 2009).

In addition, urine is not totally pathogen-free but a simple storage or its direct addition in the soil can sterilise it (Karak and Bhattacharyya, 2011).

Therefore the use of urea in agriculture is not considered as an issue regarding heavy metals or pathogens (Pahore *et al.*, 2010).

2.2 Nutrient recovery from urine

Organic fertilisers from animal manure are popular in agriculture. Interest has recently grown for the use of excreta as an accessible source of plant nutrients such as nitrogen, phosphorus and potassium (WHO, 2006). In particular, nutrient recovery from excreta has several advantages:

- It encourages the conservation of water by promoting alternatives to conventional wastewater systems.
- It contains fewer chemicals than commercial fertilisers.
- It can improve the economy by providing a direct supply of fertiliser at household level or an in-country local market if adopted at a larger scale.
- It is applicable in developing countries as well as more industrialised ones.

Separating urine and faeces at source for nutrient reuse presents some advantages. First urine contains the majority of nutrients excreted by humans with 2.5-4.3 kgN, 0.7-1.0 kgP and 0.9-1.0 kgK in urine compared to 0.5-0.7 kgN, 0.3-0.5 kgP and 0.1-0.2 kgK in faeces (Kirchmann and Pettersson, 1995). In addition it presents the advantage of producing a dry solid fraction of faeces, which makes the faeces safer to handle and easier to use.

2.2.1 Existing systems for nutrient recovery from urine

Different methods are available to recover nutrients from source-separated urine, mainly as nitrogen and phosphorus. In their study on treatment processes for source-separated urine, Maurer *et al.* (2006) listed various options such as the recovery of struvite or isobutylaldehyde-diurea (IBDU) (see Appendix 2). Overall they identified seven main options to the treatment of urine:

- Hygienisation (storage)

- Volume reduction (evaporation, freeze-thaw, reverse osmosis)
- Stabilisation (acidification, nitrification)
- Phosphorus recovery (struvite formation)
- Nitrogen recovery (ion-exchange, ammonia stripping, IBDU precipitation)
- Nutrient removal (anammox)
- Handling of micropollutants (electrodialysis, nanofiltration, ozonation)

Behrendt *et al.* (2002) summarised the main methods of urine reuse (Figure 2): if not reused directly or stored and reused, urine can be treated to produce struvite, IBDU, ammonia or a dry evaporated product.

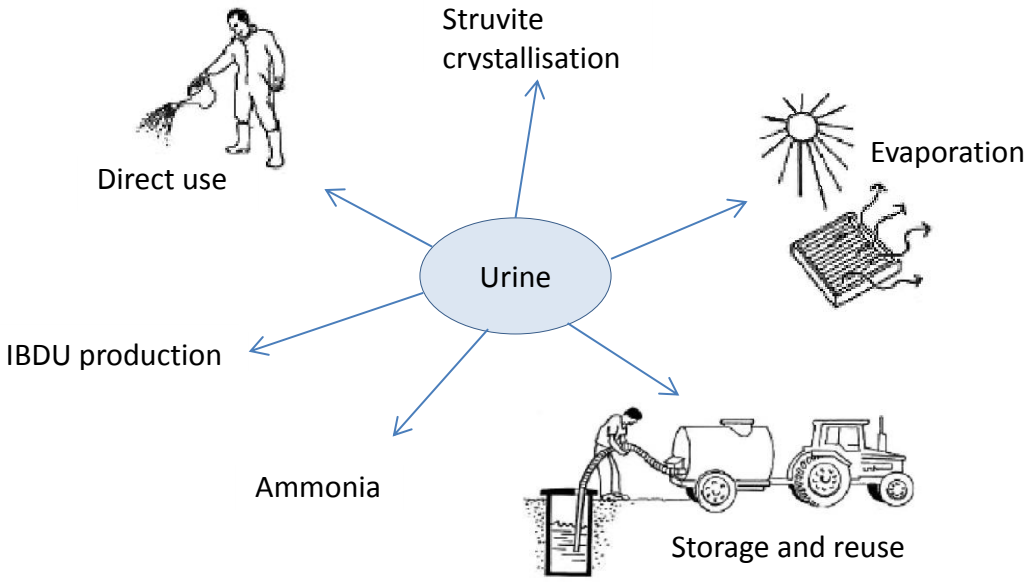


Figure 2: Main methods of urine reuse
(Adapted from Behrendt *et al.*, (2002))

An interesting example described by Behrendt *et al.* (2002) is the production of IBDU as a fertiliser, based on the recovery of urea from urine. The urea in fresh urine forms a complex with isobutyricaldehyde (IBU) to precipitate IBDU that is popular as a slow release commercial fertiliser as it is more long term owing to slow degradation rates and lower nitrogen losses into the environment. However, high temperatures and a pH of 3 are required in addition to a high amount of IBU chemical (Behrendt *et al.*, 2001), all of which making this kind of recovery technique impractical for a low income country.

The most relevant study to the current project was carried out by the University of Can Tho, Vietnam. For this study, Antonini *et al.* (2012) ran a pilot test with a solar thermal prototype. A weight of 360 g of dry product was recovered from 50 L of urine. This fertiliser contained only 2% of phosphorus and nitrogen, with sodium chloride being the predominant recovered constituent with a value of 90% of its

crystal structure. Other experiments involved the addition of acid to the urine whilst in storage, prior to evaporation, resulting in a significant increase in the nitrogen and phosphorus content. However, the study concludes that acidification is not advisable for developing countries due to its additional costs and the risks associated with the handling of acid.

2.2.2 Urea crystallisation

Urea was first identified in the 19th century and numerous techniques for its synthesis have been developed since then (Berliner, 1936; Chattaway, 1909). Two methods are predominantly used in industry, the hydrolysis of calcium cyanamide and the interaction of ammonia and carbon dioxide (Berliner, 1936). Overall, 90% of the industrial urea produced worldwide is used as fertiliser (The University of York, 2013).

2.2.2.1 Characteristics of urea

Urea is an organic compound with the chemical formula $CO(NH_2)_2$, crystallising as needle-like crystals (Table 2). It is naturally produced by mammals as a way of excreting nitrogen from the body, via the kidneys; the amount of renal nitrogen excreted being equal to that of the nitrogen intake (Weiner *et al.*, 2014).

Table 2: Physical properties of urea (Berliner, 1936)

Chemical formula	$CO(NH_2)_2$
Molecular Weight (g/mol)	60.047
Aspect	white odourless crystalline solid when pure  **Figure 3: Urea crystal** Source: Prism Glow (2011)
Melting Point (°C)	132.7
Crystalline Form	Tetragonal-scalendohedral
Crystal Habit	Needle or rhombic prisms

2.2.2.2 Mechanism of urea crystallisation

The main process to obtain dry urea crystals from human urine involves the evaporation of water (Maurer *et al.*,2006).

Using a physical water removal system is found to be the only way to achieve a final product with all recovered urine nutrients. Indeed, freeze thaw method or reverse osmosis can only remove maximum 80% of the water and recover 80% of the nutrients (Udert and Wächter, 2012). Furthermore they are advanced technologies, requiring resources which are unlikely to be available in developing countries.

2.2.2.3 Yield of urea crystallisation by solar powered evaporation

In fresh undiluted human urine, the total nitrogen concentration ranges from 7-9 g/L, of which 85% is urea (Kirchmann and Pettersson, 1995). The theoretical yield of urea ranges from 6.0 to 7.6 g/L of urine.

Although the presence of urea was not assessed in Antonini *et al.*'s (2012) study, their experimental work on a pilot-scale evaporation prototype gave a solid fraction weighting 360 g for 50 L of undiluted urine after 26 days of exposure to sun, thus giving a yield of 7.2 g/L of dry product.

2.2.2.4 Parameters influencing urea crystallisation

Crystallisation of urea by evaporation of urine occurs naturally – and is the main advantage of this technique. However, several physical parameters can affect this process and impact on the quality and quantity of the end-product.

- **Hydrolysis of urea into ammonium by urease enzyme**

The main losses of nitrogen in urine comes from its transformation into gaseous ammonia, resulting in unpleasant odours (Udert and Wächter, 2012). Urea is naturally hydrolysed into ammonia by the following chemical reaction:

$$(NH_2)_2CO + H_20 \rightarrow CO_2 + 2NH_3 \qquad (1)$$

As a result of the release of ammonia (NH_3) through the hydrolysis of urea, the pH of urine typically increases from around 6 to 9 due to microbial activity and causes calcium and magnesium to precipitate as carbonates and phosphates (Maurer *et al.*, 2006). Inorganic precipitates can lead to the creation of a sludge and therefore be an issue regarding the storage of urine or the clogging of the pipes (Udert *et al.*, 2003a).

The presence of the urease enzyme (found naturally in fungi, numerous bacteria, invertebrates and soils) (Sujoy, 2013), enhances the reaction of hydrolysis (Krajewska *et al.*, 2012; Qin and Cabral, 2002). Therefore urease is one of the main parameters that could affect the potential formation of urea from urine. Urease activity is optimal at a pH of 7-7.5 and a temperature of 45-65°C but is inhibited by heavy metal ions (Krajewska *et al.*, 2012; Larsen *et al.*, 2009; Maurer *et al.*, 2006).

- **Influence of the pH**

Low pH is reported to be an important factor in inhibiting urease (Arnold and Gresens, 2009; Maurer, Pronk and Larsen, 2006; Udert *et al.*, 2003b). This inhibiting effect starts at pH values below 5 (Udert and Wächter, 2012). Therefore, acidification is often advised to avoid nitrogen losses through hydrolysis (Arnold and Gresens, 2009).

- **Humidity and temperature**

As expected, humidity and temperature strongly influence the evaporation process: high temperature and low humidity rates allow a faster drying of the water by solar powered evaporation.

Both parameters have a strong influence on the stability of the end-product. A phenomenon of deliquescence through which dry urea crystals will capture atmospheric moisture, hence breaking down and returning to a liquid state, occurs at 18°C and a humidity rate of 80%. Although it is possible to restore the liquid back to crystal form by drying it -either by blowing dry air over the liquid or placing it in an oven for a few minutes at 70°C (Mills *et al.*, 2010), the stability of urea crystals is an issue regarding their storage and transportation, especially in countries such has Tanzania where the relative humidity can range from 70 to 85%.

2.3 Interests in producing urea from urine

2.3.1 Urine reuse as a fertiliser

Perceptions to excreta and its reuse vary hugely between different cultures but attitudes to reuse of urine usually differ from that of faeces (Drangert, 1998). Evidence suggests that urine is not met with revulsion, shown by its acceptance as a fertiliser in several countries. However, the fact that users must compromise on comfort to collect urine is preventing a widespread acceptance.

As illustrated in Section 2.1, urine contributes the most nutrients and micro pollutants to wastewater so urine diversion is proposed as a way to overcome wastewater management challenges (Larsen *et al.*, 2001). By utilising the source separated urine, the volume of wastewater is reduced and wastewater arriving at a wastewater treatment plant has a lower nutrient load, saving on energy and treatment costs (Pynnönen and Tuhkanen, 2012).

Urine has been proved to be an effective fertiliser in various studies and particularly on crops usually limited by the supply of nitrogen, such as maize, rice, wheat and millet (EcoSan Club, 2010; Schönning, 2001; WHO, 2006). For example, Figure 4 compares barley yields from plots fertilised with human urine to those using artificial fertiliser: as illustrated, the yield of barley fertilised with human urine is equal to or higher than with use of mineral fertiliser.

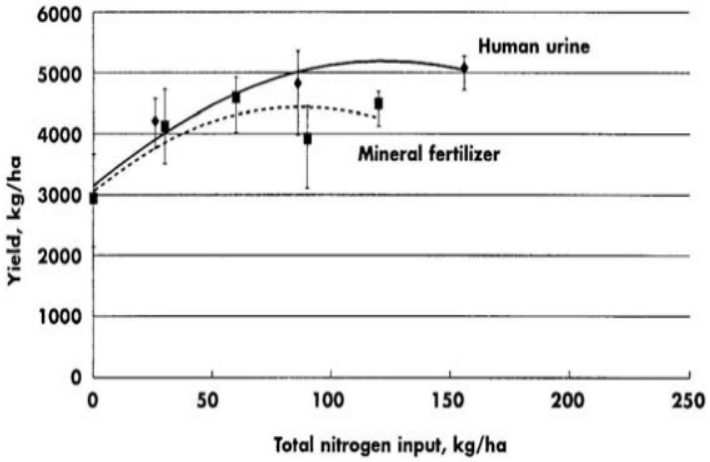


Figure 4: Yield from barley plots
Source: Johansson *et al.*, 2001 cited in Schönning 2001

2.3.2 Economics

Most wastewater treatment options in developing countries fail due to a lack of financial resources to pay for maintenance and running costs. As fertilisers have high economic value, urine reuse is seen as a sustainable business model (Pronk and Koné, 2009).

Urea is a popular fertiliser in Tanzania, due to a government subsidy set up in 2003 for urea and DAP (diammonium phosphate) fertiliser options. Like in many Sub-Saharan Africa countries, low soil fertility in Tanzania is a key limiting factor for agriculture (Isaac, 2005). The current demand for fertiliser in Tanzania is 4.4 kg/ha, significantly below the 141 kg/ha world average in 2012.

During a field visit to Tanzania, WaterAid UK received feedback about current prices of urea, imported from China, ranging from £13-£33/ 50 kg bag. During this visit, it was also found the Ministry of Agriculture offer subsidies for fertilisers but these are "small and targeted".

A National Agriculture Input Voucher Scheme was introduced in 2008/2009 which has been shown to have a positive contribution in poverty reduction (Aloyce *et al.*, 2014). Reports state that the scheme was suspended in May 2014 (Domasa, 2014). However, it should be noted that a urea fertiliser plant is due to open in Tanzania following a prefeasibility study, illustrating the interest for this type of fertiliser (Oirere, 2014).

Further investigation on the ground in the proposed area in Tanzania is required. Information on when the urea plant will open and what impact that might have on fertiliser prices is needed to fully understand whether urine derived urea would be able to compete with industrial urea.

2.4 Source-separation of urine for urea production: process design considerations

2.4.1 Storage of urine

Storage is a necessary step in the process or urine reuse, either directly after production or before central collection and treatment. Thus, several parameters or risks must be taken into account.

2.4.1.1 Health risks management

For direct reuse on crops without extensive treatment, Maurer *et al.* (2006) recommend storing urine for at least 6 months at minimum 20°C in order to deactivate pathogens.

Regarding hygiene issues, Höglund *et al.* (1998) advise monitoring several parameters such as pH, temperature and storage time. High pH and temperature or a longer storage time will make the urine safe. However, extended storage is the cheapest and easiest form of urine treatment: bacteria, viruses, intestinal helminths and parasitic protozoa will die once a pH of 9 is reached through hydrolysis of urea (Werner and Bracken, 2009).

2.4.1.2 Nitrogen losses

The main challenge of storage is nitrogen loss, which can reach 50% due to its transformation into gaseous ammonia (Udert and Wächter, 2012). If urine was stored at 38°C in absence of urease, it would have a half-life of 3.6 years (Udert *et al.*, 2003a).

As detailed in Section 2.2.2.4, the main effect of hydrolysis on the characteristics of urine during storage is an increase of pH and temperature. As a consequence, an increase of pH can induce the formation of sludge as a by-product in the storage container. In a waterless sanitation system this sludge will be composed of up to 30% of the phosphorus contained in the urine. Therefore it can be used as a fertiliser for plants with a high phosphorus demand (Sphuler, 2015).

As stated by Hellström *et al.*, (1999) ammonia is formed during the storage of urine and remains in equilibrium with its gaseous form. The amount of partial pressure above the surface of the liquid will have a direct effect on the reaction of change of state from liquid to gas. In order to minimise nitrogen losses, the gas exchange rate must thus be reduced by preventing ventilation (Hellström *et al.*, 1999). This will reduce the quantity of ammonia being allowed to volatilise (Sphuler, 2015).

2.4.2 Pasteurisation

The primary aim of this treatment stage is to eliminate the pathogens present in urine such as viruses, bacteria and protozoa. Secondly, as temperature strongly influences enzymatic activity, pasteurisation could be a solution to inhibit urease activity, if operating at the right temperature.

Pasteurisation can be performed either by maintaining the liquid at 63°C for 30 minutes (classic pasteurisation) or by heating the liquid to 72°C for at least 15 seconds (flash pasteurisation) (Gunn, 2014). The pasteurisation process must take place in an anaerobic environment in order to be effective. However a control is required to ensure that the pasteurisation has been effective and the urine is safe at the end of this stage. Other options to reduce the microbial matter are available, such as acidification, microfiltration or ultrafiltration (Maurer, Pronk and Larsen, 2006). However pasteurisation is one of the best ratio cost/efficiency options. Indeed, some existing pasteurising systems, in the beer industry for example, can treat incoming flow from 10 hL/h to 500 hL/h (Gunn, 2014).

In a paper summarising the main properties of urease, Qin and Cabral (2002) report an optimum temperature around 60-70°C for urease activity. Regarding its inhibition, despite the availability of numerous papers, it is difficult to find a clear inhibitive temperature for urease. Sahrawat (1984) reports deactivation of urease activity in soil starting from 100°C.

2.4.3 Evaporation

In order to produce an effective fertiliser, the nutrients in urine need to be concentrated through an appropriate volume reduction technique. Concentrating urine is also the best response to the problems of transportation and storage associated with the use of urine as a liquid fertiliser (Dutta, 2012).

Evaporation is a simple method with low operation and maintenance costs, particularly adapted for developing countries. It is usually suited to these countries with hot climates but faces two challenges: the loss of ammonia and the amount of energy required (intensity of solar light, temperature) (Maurer *et al.*, 2006).

As Putman (1971) explains, the boiling point of urine rises as a function of its liquid fraction, due to the increasing content of solids in urine. Udert and Wächter (2012) establish the boiling point of synthetic urine of 130°C, allowing the evaporation of 99.8% of the water. In terms of energy, Maurer *et al.* (2003) reported a need for 54 MJ/kgN for urea production.

Little in-field work has been done to characterise the evaporation properties of urine. The most relevant study of solar evaporation of urine was performed in Vietnam in 2012 (Antonini *et al.*, 2012). The authors report an evaporation rate of almost 1 L/m^2/day in outdoor conditions. The complete evaporation of the liquid fraction of 50 L of undiluted urine was reached within 26 days for a surface area of 2 m^2. The losses of the liquid fraction were of 17% probably due to poor insulation of the pilot system.

Other experiments have been done under different conditions and with improved evaporation systems. Pahore *et al.* (2010) tested a volume reduction system based on water evaporation from a vertical gauze sheet. The system was applied to the dry climate in southern Pakistan, having an air temperature of 30–40°C and air humidity of 20–40%. It achieved an 80% volume reduction of 10 L of urine per day, which gives an evaporation rate of 8 L/day.

2.5 Potential risks associated with recovery of urea from urine

2.5.1 Risks of pathogen contamination

Although urine is considered as safe by many studies on reuse of urine (Karak and Bhattacharyya, 2011; Pahore *et al.*, 2010), there is a pathogenic risk of using urine from unhealthy humans (Santos *et al.*, 2004).

Urine taken from healthy humans has been shown to be prone to faecal contamination. For instance, in their study on faecal sterols in source separated urine collection tanks, Schonning *et al.* (2002) showed that 37% of samples taken in urine collection tanks were found to be contaminated by faeces. There are two main ways of eliminating this risk, the first by storing the urine for at least six months, which will mean it is safe enough to use on any crop (Höglund, *et al.*, 2002; Maurer *et al.*, 2006). The second is by pasteurisation, which has been trialled extensively through SODIS, and is assumed to kill all pathogens (Antonini *et al.*, 2012).

After storage or pasteurisation has eliminated pathogens there is a chance of recontamination, leading to the advice that urine should only ever be spread on the soil rather than edible parts of the plants (Richert *et al.*, 2010). Urine application has been shown to increase the electrical conductivity of the soil, which, depending on the levels applied can have an adverse effect on the crop (Neina and Nii Noi Dowuona, 2013). Other risks to consider are prevalence of heavy metals, although these are low compared with other organic fertilisers (Kirchmann and Pettersson, 1995).

2.5.2 Ammonia

Failure to deactivate urease in the pasteurisation stage of prototype would result in large quantities of ammonia being produced, putting the operator at risk. Indeed, ammonia can cause severe skin irritation or burns due to high solubility in water: Makaya *et al.* (2014) demonstrated a significant risk of skin problems in handlers of urine based fertilisers in Burkina Faso. Furthermore, ammonia can also cause serious eye problems such as conjunctival ulceration and glaucoma (Agency for Toxic Substances and Disease Registry, 2004).

2.5.3 Pharmaceuticals

Concern has been expressed that pharmaceutical residues in urine will enter the human food chain and have a negative effect on plant and human health. So far, the results show that pharmaceutical levels in average urine do not affect plant growth, supported by the fact that residues are much lower than those in animal manure, which has widespread use in agriculture. Widespread use of urine as a fertiliser suggests that urine is currently estimated to be safe due to a very low content of pharmaceuticals (Pynnönen and Tuhkanen, 2012; Winker *et al.*, 2010). However, more research is required and Winker (2009) concluded that despite no toxic effects

of pharmaceuticals being proved, the use of urine from people on medication should be avoided when producing urine-derived fertiliser.

In addition, Pynnönen and Tuhkanen (2012) highlight that the presence of pharmaceuticals is likely to be more prevalent in poorer countries with inadequate sanitation and large quantities of antiprotozoal pharmaceuticals and drugs for the treatment of HIV are likely to be present. Such substances have been detected in the aquatic environment and minimal levels are known to impact human health (Pynnönen and Tuhkanen, 2012). However, these heightened risks in low-income countries must be balanced against possible benefits to human health by improved soil fertilisation and subsequent harvest (Schönning, 2001).

2.6 Recommendations based on the literature

From the scientific literature findings, urine appears to be a suitable fertiliser for crops as it has a high content of nitrogen, mostly in the form of urea.

Urine is generally considered to be safe with regards to health although it requires some monitoring such as a sufficient storage time. Regardless, urine appears to have lower concentrations of pharmaceuticals and heavy metals than commercial fertiliser or farmyard manure.

Significant research has been done on direct use of urine on crops, which seems a realistic option. Unlike excreta reuse, for example for composting, social acceptance of urine reuse seems good, especially in Tanzania.

As little work has been done on urea recovery, it is difficult to say how efficient urine-derived urea can be compared to synthetic urea. Further work is then required to scale-up the field of study under real agricultural conditions. However, Antonini *et al.* (2012) were optimistic as they obtained a yield as high as the one of a commercial fertiliser when using urea on maize crops at lab-scale.

However, the main issue of urea recovery from urine seems to be its hydrolysis into ammonia, which could lead to significant losses. The process is strongly influenced by pH and temperature.

In order to avoid hydrolysis of urea, findings from literature review suggest that pasteurisation of urine should be the first step of the process before storage. Indeed, by using high enough temperature for a given period of time, pasteurisation could deactivate urease and therefore consequently reduce urea and nitrogen losses during storage and evaporation.

3 Experimental work

The experimental part of this project aimed to evaluate at lab-scale the performance of the evaporation stage in producing urea crystals. The effect of the evaporation surface area and temperature on the chemical composition of the final product was investigated.

3.1 Material and method

3.1.1 Urine collection

Urine was collected anonymously from six healthy volunteers who gave informed consent. It should be noted that the handling of fresh human urine is regulated under the Human Tissue Act (2004) which makes provision with respect to activities involving human tissue. Therefore, in the study, the collection and handling of fresh urine samples complied with the Act and was validated by the Cranfield University Health Research Ethics Committee to ensure that the study conformed to general ethical principles and standards.

3.1.2 Urine Characterisation

Prior to each set of experiments the urine was analysed for COD, nitrates, ammonium, nitrates, total nitrogen, phosphate and sulfate using Merck Spectroquant cell tests (Fisher Scientific, UK) as detailed in Table 3.

Table 3: Summary of cell tests used

Compound		Range (mg/L)	Dilution required
Ammonium	NH_4^+	4-80	1:10
Chemical oxygen demand	COD	25-1500	1:10
Nitrate	NO_3-	1-50	
Total nitrogen	Tot-N	10-150	1:10
Total phosphorous	Tot-P	0.5-25	1:10
Phosphate	PO_4-	0.5-25	
Sulfate	SO_4^{2-}	5-250	1:10

In addition, the samples were analysed for (aluminium, sodium, potassium, magnesium, calcium) using Atomic absorption spectrometry (AAS) (Perkin Elmer AAnalyst 800). The samples were analysed by the AAS in triplicate and the mean reported in mg/L.

3.1.2 Evaporation prototype

The small-scale evaporation system developed and tested in this study included a 150 Watt lamp as a heat source (Exo Terra Glow light, PT2054, Exo Terra, UK) and a glass pane (45.5 cm x 61 cm) supported by clamp stands, as illustrated in Figure 5.

The glass allowed the temperature of the urine to increase via the greenhouse effect. The experiment was left to run until the urine had evaporated and appeared dried. The tray was weighed at the start and end of the experiment and where possible, every 1.5 hours within working hours. The temperature of the urine was recorded at the same interval. A tray was also placed under the edge of the glass to eventually catch liquid condensing on the glass.

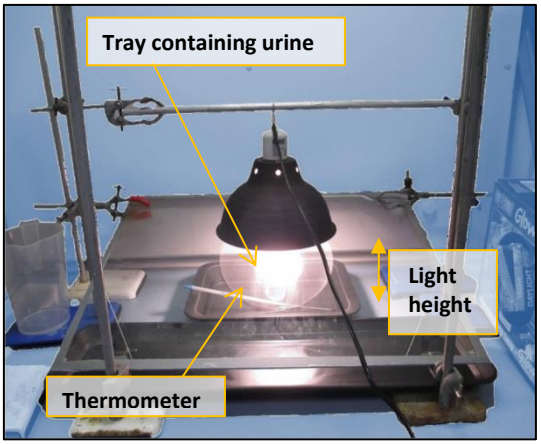


Figure 5: Evaporation stage set-up

Five experiments using urine collected on five distinct occasions were performed and will be referred to as experiments 1 to 5. They had variable parameters, as described in Table 4.

Table 4: Summary of experiments

N°	**Pasteurised?** (Y/N)	**Type of tray** shallow (S), deep (D)	**Light height** (cm)	**Ventilation** ON/OFF
1	N	D	16	OFF
2	Y	D	16	OFF then ON
3	Y	S	16	ON
4	N	S	16	OFF
5	Y	D	30	ON

The experiment was conducted in a fume hood (Zurich local exhaust ventilation system) which was turned ON or OFF. The distance of the light from the glass panel was adjusted in experiment 5 to simulate reduced solar intensity and temperature. The surface area was adjusted by choice of two evaporation plates. A shallow tray (S) measuring 29cm x 26cm x 2.5cm (surface area: 75.4 cm^2) or a deep tray (D) measuring 28cm x 22cm x 4.5cm (surface area: 61.6 cm^2).

For experiments 2, 3 and 5 the urine was pasteurised within 3 hours of collection. In 3 and 5 the urine was heated to 72°C for a minimum of 15 seconds in a round bottomed flask in a 500 watt Electromantle® (Electrothermal, Bibby scientific, UK). In experiment 2 the pasteurisation was achieved by heating the urine in a baking tray on a hot plate (Bibby Stuart SB162, Bibby scientific, UK). For experiments 1 and 4, the urine was not pasteurised.

3.1.3 Dry product analysis

The dry end-product from each experiment was analysed using an optical microscope and X-ray Diffraction (XRD) (Siemens D5005) to assess the presence of urea crystals. The samples will be referred to as 1-5 corresponding to the experiment that produced the dry product (Table 4).

The optical microscope uses visible light and a system of lenses to magnify images of the sample that are captured by a camera to generate micrographs. The Nikon Eclipse ME600 has a magnification range up to 1500 times. The samples were first viewed without further drying on glass slides. They were also analysed after being further dried with a vacuum oven (Gallenkamp vacuum oven, Fistreem Int., UK) (with vacuum setting OFF) at 60°C for 12 hours and stored in the oven at 40°C for a further 5 days.

XRD analyses were used to provide information on the molecular structure and help identify the crystalline phases. The XRD requires perfectly dry samples so the product was dried further in a Gallenkamp Vacuum oven (with vacuum setting OFF) at 60°C for 12 hours and samples 2, 3 and 4 were left in the oven at 40°C for a further 48 hours.

3.2 Results and Discussion

3.2.1 Urine characterisation

Urine characterisation allowed the chemical characteristics of the samples to be investigated and the differences between pasteurised and non-pasteurised urine demonstrated.

Experiments 1 and 4 used non- pasteurised fresh urine. The results, summarised in Table 5, were similar in both experiments. Average COD concentrations were around 5000 mg/L, while concentration in total phosphorus (Total P), including phosphorus and phosphate elements, ranged from 193.0 mg/L for to 240 mg/L. Phosphate levels ranged from 46.0 to 66.5 mg/L. As expected, the ammonium and nitrate levels were lower than the nitrogen levels, with values ranging from 530 to 1360.0 mg/L for experiment 1 and from 590 to 750 mg/L for experiment 2.

Table 5: Unpasteurised experiments results

Compound (mg/l)	Experiment 1			Experiment 4		
	Minimum	Maximum	Average	Minimum	Maximum	Average
COD	4590.0	5570.0	**4980.0**	4940.0	5220.0	**5080.0**
Total P	235.0	240.0	**235.0**	193.0	211.0	**202.0**
Phosphates	46.0	65.6	**54.9**	56.4	66.5	**61.4**
Nitrates	41.6	64.2	**52.9**	107.6	108.1	**107.8**
Sulfates	940.0	1460.0	**1163.3**	1300.0	1370.0	**1310.0**
Ammonium	285.4	293.1	**289.3**	286.7	304.7	**295.7**
Nitrogen	530.0	1360.0	**945.0**	590.0	750.0	**670.0**

Experiments 3, 4 and 5 were conducted using urine pasteurised at 72°C for at least 15 seconds. Surprisingly, the results, summarised in Table 6, show a difference between experiment 2 and experiment 3 and 5. For instance, the COD level of 4645 mg/L measured for experiment 2 is comparable to the results obtained with unpasteurised urine (Table 5), whereas for experiments 3 and 5 average COD levels are 2320 and 2805 mg/L respectively. The other major difference is related to the sulfate level, which is much lower (410 and 635 mg/L) for experiments 3 and 5 than for experiments 1, 2 and 4 (respectively 1163, 1310 and 1280 mg/L).

Table 6: Pasteurised experiments results

Compound (mg/l)	Experiment 2			Experiment 3			Experiment 5		
	Minimum	Maximum	Average	Minimum	Maximum	Average	Minimum	Maximum	Average
COD	4600.0	4690.0	**4645.0**	2260.0	2380.0	**2320.0**	2700.0	2910.0	**2805.0**
Total P	194.0	209.0	**201.5**	184.0	193.0	**188.5**	202.0	205.0	**203.5**
Phosphates	56.1	65.0	**60.5**	69.0	71.1	**70.0**	71.1	80.0	**75.5**
Nitrates	76.6	73.5	**75.1**	40.7	41.2	**41.0**	56.7	58.5	**57.6**
Sulfates	1000.0	1740.0	**1280.0**	360.0	460.0	**410.0**	630.0	640.0	**635.0**
Ammonium	340.7	342.0	**341.4**	142.7	149.1	**145.9**	351.0	358.7	**354.9**
Nitrogen	710.0	870.0	**790.0**	4170.0	4500.0	**4335.0**	2590.0	4360.0	**3475.0**

The difference between experiments 2,3 and 5 could be explained by the quality of the pasteurisation, which was not optimal in experiment 2 due to the type of pasteurisation equipment used (a hot plate rather than an Electromantle). This is well illustrated on Figure 6, with experiment 2 showing nitrogen levels expected for unpasteurised urine rather than pasteurised urine.

Finally, the nitrogen level is much higher for experiments 3 and 5 with 4335 and 3475 mg/L respectively, than for the others experiments with average values ranging from 670 to 790 mg/L as shown on Figure 6.

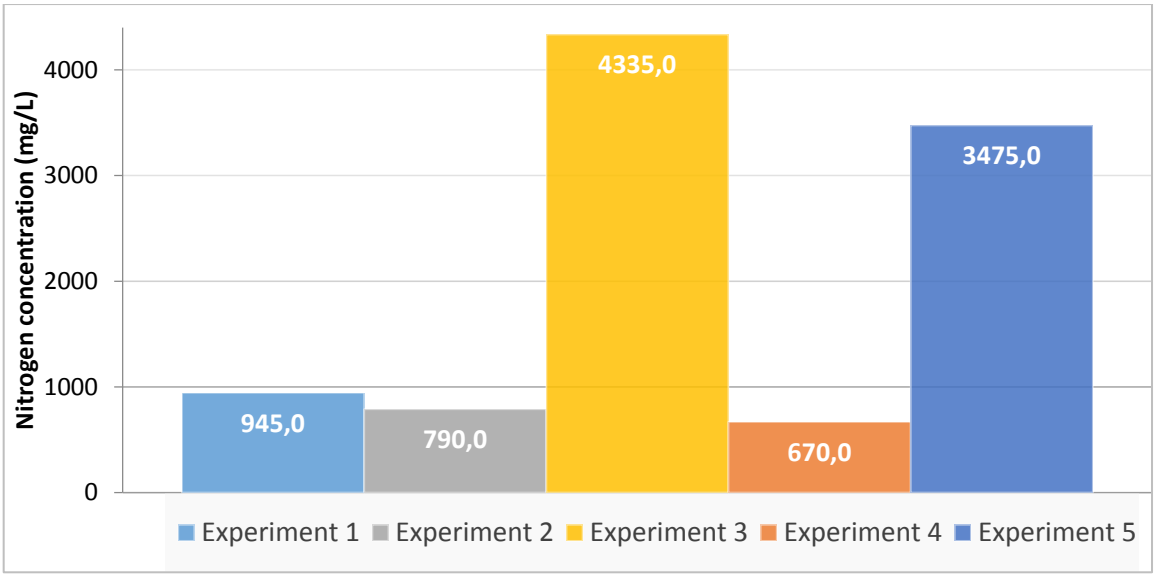


Figure 6: Experimental nitrogen concentration

When comparing these results to values reported in the literature, they are of the same order of magnitude (Table 7). The total phosphorus and nitrates levels are slightly below the values found in experiments here, around 400 mg/L for the phosphorous and 200 mg/L for the nitrates. The COD and sulfate levels reported in the literature are in the range of the values measured here for pasteurised and unpasteurised urine. However, in terms of nitrogen, the results reported in the literature seemed more comparable to the results obtained for pasteurised urine, indeed it is around 3800 mg/L in the literature and between 3475 mg/L and 4335 mg/L for the experimental measures.

Table 7: Literature data for urine components

Compound (mg/l)	Literature	Reference
COD	4000	(Jönsson *et al.*, 2005)
Total P	400	(Schönning, 2001)
Nitrates	200	(Mitchell *et al.*, 1916)
Sulfates	1000	(Institute of Medicine Food and Nutrition Board, 2004)
Nitrogen	3800	(Schönning, 2001)

The results in Table 6 andTable 7 also suggest that the pasteurisation process reduces the sulfate concentration in the urine, as well as the COD content. This low COD level may be explained by the effect of the pasteurisation on organic components within the urine sample as these organic matters would be eliminated by the heat of the pasteurisation. On the contrary, the nitrogen content is much higher in the pasteurised samples than in the unpasteurised ones. This could be explained by the fact that the pasteurisation limits hydrolysis in the urine, and therefore avoids significant nitrogen losses.

Overall, the low level of urine components measured in the urine samples tested in this study could be due to dilution effects. Indeed, as done anonymously, it was not possible to track whether the volunteer consumed an above average amount of water, which may have diluted the samples. This dilution would have reduced the levels of the different components while measured, distorting the results.

The urine samples have also been tested through AAS analysis in order to determine their metal content. Table 8 summarises the AAS results for the unpasteurised samples.

The results obtained for unpasteurised urine show much higher levels of potassium for experiment 1 (1186 and 1197 mg/L) than for experiment 4 (581 and 565 mg/L). The other difference between the two experiments is the aluminium level, above 1 mg/L for the first one, and below 1 mg/L for the fourth one. Sodium and calcium levels are quite similar.

Table 8 : Metals concentrations in unpasteurised urine

Sample (mg/L)	Aluminium		Sodium		Potassium		Magnesium		Calcium	
	Measure	Std. Dev.	**Measure**	Std. Dev.	**Measure**	Std. Dev.	**Measure**	Std. Dev.	**Measure**	Std. Dev.
Exp 1 Dissolved	**1.00**	0.1319	**714.8**	0.283	**1186.0**	0.037	**41.9**	0.0325	**20.8**	0.0160
Exp 1 Total	**1.43**	0.1080	**717.0**	0.159	**1197.3**	0.122	**42.9**	0.0360	**20.4**	0.0287
Exp 4 Dissolved	**0.18**	0.0933	**641.0**	0.137	**581.3**	0.343	**69.8**	0.0665	**23.8**	0.0655
Exp 4 Total	**0.05**	0.0381	**624.3**	0.099	**565.8**	0.077	**67.1**	0.0472	**21.7**	0.0574

Table 9 summarises the results obtained for pasteurised urine. There is no major difference between the three experiments for the pasteurised samples. The level of aluminium is equal or below 1 mg/L, the others concentrations are all of the same order of magnitude as for unpasteurised urine (Table 8).

Table 9 : Metals concentrations in pasteurised urine

Sample (mg/L)	Aluminium		Sodium		Potassium		Magnesium		Calcium	
	Measure	Std. Dev.	**Measure**	Std. Dev.	**Measure**	Std. Dev.	**Measure**	Std. Dev.	**Measure**	Std. Dev.
Exp 2 Dissolved	**1.04**	0.0358	**632.5**	0.054	**694.8**	0.230	**39.1**	0.0335	**11.5**	0.0340
Exp 2 Total	**0.87**	0.0661	**643.0**	0.240	**695.3**	0.694	**39.3**	0.0273	**11.2**	0.0512
Exp 3 Dissolved	**0.422**	0.0987	**390.3**	0.126	**494.8**	0.137	**16.7**	0.0078	**13.4**	0.0109
Exp 3 Total	**0.199**	0.1098	**385.5**	0.118	**503.8**	0.130	**16.3**	0.0078	**12.2**	0.0382
Exp 5 Dissolved	**0.01**	0.0613	**473.75**	0.121	**296.5**	0.075	**27.83**	0.0312	**21.0**	0.0975
Exp 5 Total	**0.024**	0.0487	**490.25**	0.140	**308.25**	0.050	**28.04**	0.0161	**20.21**	0.0316

The comparison between both sets of results suggests that metal concentrations are not influenced by the pasteurisation process. Figure 7 represents the sodium concentration obtained for all experiments, illustrating the homogeneity of the results, with similar ranges in concentrations.

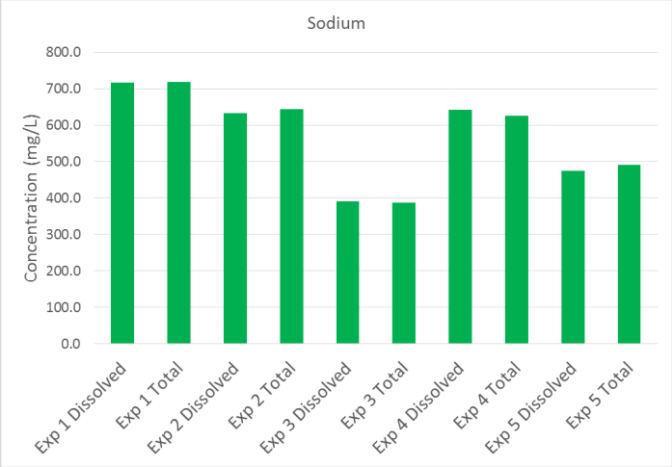


Figure 7: Experimental sodium concentration

A comparison of these results with the literature shows that except for sodium and aluminium, the concentrations measured here for metals are lower than those reported in the literature. For instance, the highest measured potassium concentration is 1197.3 mg/L while Schönning (2001) reported a value of 1500 mg/L. For calcium, It should be noted that concentrations measured here, ranging from 11.2 to 20.2 mg/L, were much lower than those reported in the literature with for example a calcium level of 135 mg/L reported by McCrudden (1911). In contrast, the lowest value measured for sodium across the five experiments was 385.5 mg/L whereas (Dugdale, 2013) reported a level of 130 mg/L.

Possible explanations may be differences in diet and liquid intake of the volunteers involved in the current study compared to those of the individuals providing urine samples in other studies. Indeed, the composition of urine is known to vary significantly depending on the diet (EcoSan Club, 2010). Therefore the results obtained here are case specific and should not be taken as representative of what would be obtained for urine collected in Tanzania.

3.2.2 Prototype

After the urine characterisation stage was completed, the urine was used to feed the evaporation prototype. The impacts of surface area and temperature, as well as pasteurisation, on the evaporation process were investigated. Table 10 summarises the characteristics that were recorded during the experiments using unpasteurised urine.

Table 10: Unpasteurised urine experiments characteristics

Experiment 1		Experiment 4	
% reduction	Time (h)	% reduction	Time (h)
98.4	41.0	98.9	71.5
Reduction (g)	Rate (g/h)	Reduction (g)	Rate (g/h)
979.8	23.9	1011.7	14.1
Surface (m2)	Temperature (°C)	Surface (m2)	Temperature (°C)
0.0616	22.5	0.0754	33.4
Rate (g.h.m2)	387.96	Rate (g.h.m2)	187.66

Experiment 1 was performed using a deep tray with a surface area of 61.6 cm^2 whereas experiment 4 was performed using a shallow tray with a surface area of 75.4 cm^2. As showed in Table 10, the reduction as mass percentage of the urine sample is similar in both sets of experiment, with 98.4% of mass reduction for experiment 1 and 98.9% for experiment 4, which represents for both of experiments around 1000 g. The major difference between these two experiments is the time duration and consequently the evaporation rate. Indeed, for experiment 1 the evaporation lasted 41 hours, resulting in an evaporation rate of 387.96 $g/h/m^2$ while for experiment 4 the duration was 71.5 hours resulting in an evaporation rate much lower with 187.66 $g/h/m^2$. There is also a difference in temperature between the two sets of experiments, with an average temperature of 22.5°C for experiment 1 as opposed to 33.4°C for experiment 4.

Similar experiments were performed using pasteurised urine and results obtained for this second set of experiments are summarised in Table 11 below.

Experiments 2 and 5 used the shallow tray with a surface area of 75.4 cm^2 while experiment 3 used the deeper one with a surface area of 61.6 cm^2. For the three experiments the percentage of mass reduction was above 98%, which represents a reduction of about 950 g for each experiment. Once again the duration time is the major difference between the experiments, indeed while experiments 2 and 3 lasted 28.4 and 23.4 hours respectively, experiment 5 lasted almost twice this time with 52.5 hours. The rates calculated for experiments 2 and 3 were 530.34 and 551.69 $g/h/m^2$ respectively and 294.93 $g/h/m^2$ for experiment 5. However it should be noted that the temperature was above 22°C for both experiments 2 and 3, and it was 16.5°C for experiment 5, making it the coldest experiment of both pasteurised and unpasteurised.

Table 11: Pasteurised urine experiments characteristics

Experiment 2		Experiment 3		Experiment 5	
% reduction	Time (h)	% reduction	Time (h)	% reduction	Time (h)
98.3	28.4	99.3	23.4	98.5	52.5
Reduction (g)	Rate (g/h)	Reduction (g)	Rate (g/h)	Reduction (g)	Rate (g/h)
927.3	32.7	974.8	41.6	953.8	18.2
Surface (m2)	Temperature (°C)	Surface (m2)	Temperature (°C)	Surface (m2)	Temperature (°C)
0.0616	22.5	0.0754	23.4	0.0616	16.5
Rate (g.h.m2)	530.34	Rate (g.h.m2)	551.69	Rate (g.h.m2)	294.93

The first observation that can be made is based on the surface area and depth of the tray used. The hypothesis was that a shallower tray and higher surface area would allow a maximum contact between urine and air, hence resulting in faster evaporation rates. However, the results described above, do not confirm this hypothesis. There does not seem to be a connection between the evaporation rate and the choice of the tray as illustrated on Figure 8 which shows evaporation rate as a function of surface area.

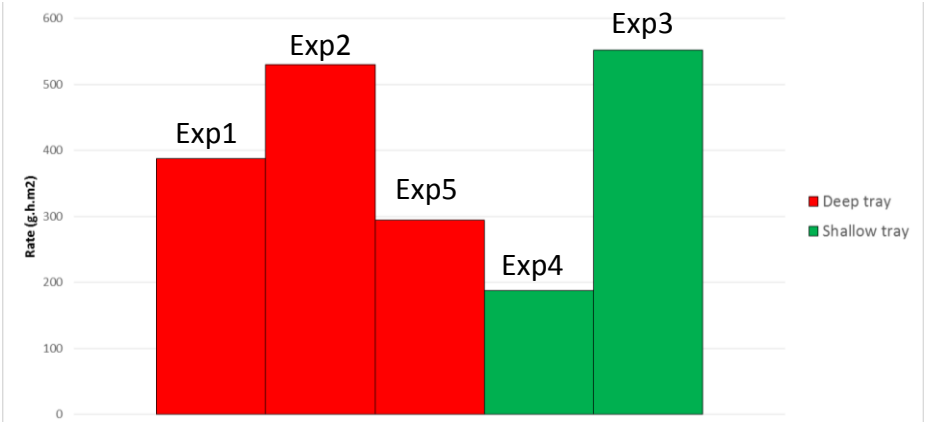


Figure 8: Evaporation rate and tray size

This could be explained by the nature of the prototype. In the shallow tray experiments with the largest surface area, the impact of the heat from the light is restricted to the central part of the tray, hence not evenly covering the whole surface and reducing the evaporation efficiency. This corroborated with visual observations of crystal formation during the process, which looked different in the area under the light beam than on the sides of the tray, suggesting lower evaporation rate on the edges.

Under similar temperature conditions, pasteurised urine seemed to evaporate more easily, resulting in faster crystal formation than unpasteurised urine (Table 10 and 11). This could be due to the elimination of some organic matter during pasteurisation that would slow down the evaporation. Another aspect to take into account is also the initial temperature, which is higher for the pasteurised urine since it goes straight from pasteurisation apparatus to the evaporation tray with a temperature of at least 75°C.

The evaporation of urine was plotted over time for all experiments (**Erreur ! Source du renvoi introuvable.**). It should be noted that for experiment 4 complete evaporation of the system could not be recorded appropriately due to access issues over a 2-day period. Therefore, evaporation rate of urine could be recorded over a day, while the dry product was recovered after 4 days. As can be seen on **Erreur ! Source du renvoi introuvable.**, pasteurised samples seem to be the quickest to reach the dry conditions.

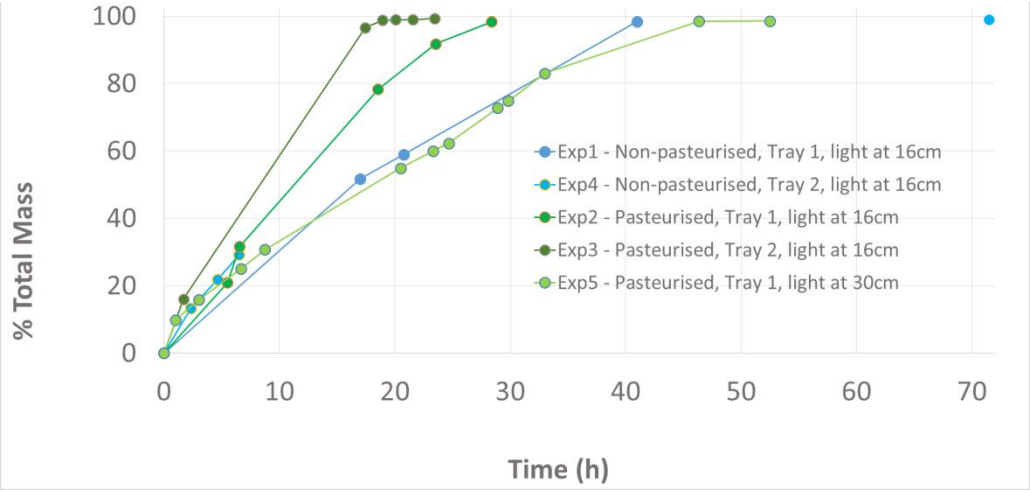


Figure 9: Evaporation of urine

Another parameter influencing the evaporation was the use of the ventilation in the fume cupboard. Without the ventilation the temperature was much higher which increases evaporation. Temperature is one key parameter, as shown in Table 9, the experiment with the lowest temperature, due to the higher position of the lamp, resulted in the lowest evaporation rate.

Although not possible in this study, the observation of the evolution of pH over time should be recorded as a subject of further research. It could help to assess the occurrence of hydrolysis which increases the pH and gives an indication of nitrogen losses.

3.2.3 Optical Microscope and XRD analysis of product from urine evaporation

The resulting samples will be referred to as 1-5, corresponding to the experiment which produced them.

For all experiments, after up to a week of drying at room temperature the samples looked very different. They varied from crystalline to amorphous and all had showed signs of becoming more viscous since recovery from the evaporation unit. The growth of crystals in samples 1, 2 and 3 was visible to the naked eye. Sample 5 was particularly amorphous as the sample had not dried completely in the lower temperature conditions of experiment 5.

The samples recovered from the evaporation unit were viewed under the optical microscope. However, these were too wet to give conclusive results but did show evidence of the presence of individual crystals. As shown in Figure 10 and Figure 11, the crystals appeared isolated in amorphous material.

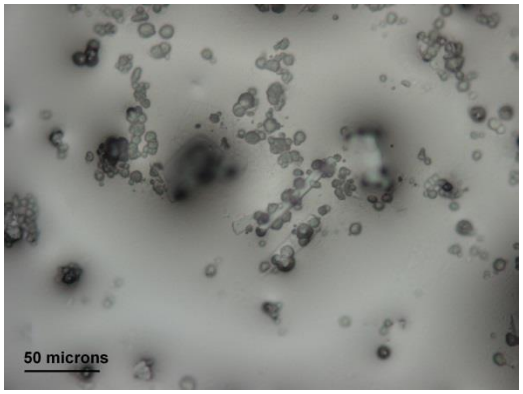


Figure 10: Micrograph of pre drying sample 1

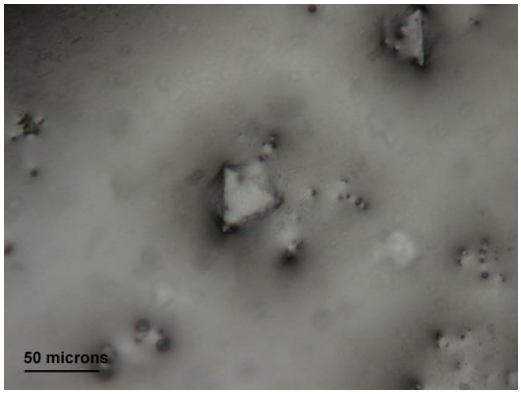


Figure 11: Micrograph of pre drying sample 2

For this reason, the samples were further dried to allow better optical microscope analysis. After being stored in the oven at 40°C for 5 days water was still present in the samples but crystal growth was enhanced in size and quality.

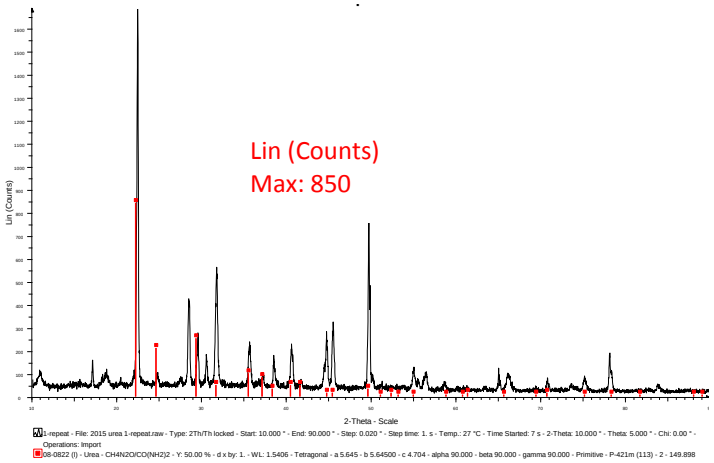
Figure 10 and Figure 11 show a comparison of the micrographs obtained from these dried experiments with the XRD patterns from the corresponding sample. The full collection of micrograph and XRD spectrums can be found in Appendix 3Appendix 4.

Rectangular shaped crystals that resemble struvite crystals can be seen in all but sample 5, and are most numerous and defined in sample 2. However, XRD analysis of these samples did not identify struvite as present in any of the samples. Triangular shaped crystals are also common in samples 1 and 4 (the pasteurised samples) and could not be observed in the other samples. The crystals in sample 5, seen in Figure 12 have a unique fan-like structure not seen in the other samples, although their definition is hindered by the water content in the sample.

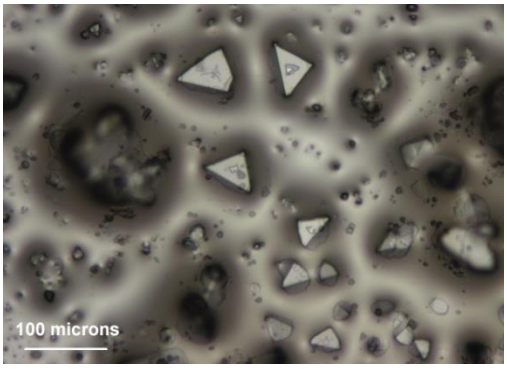
The XRD pattern generated from the samples corresponded with database models of the urea crystalline phase in terms of position and intensity of the peaks. This confirmed the presence of urea in all 5 samples. Sample 3 showed the highest peak intensity value (Figure 12) indicating high concentrations of urea in relation to other substances.

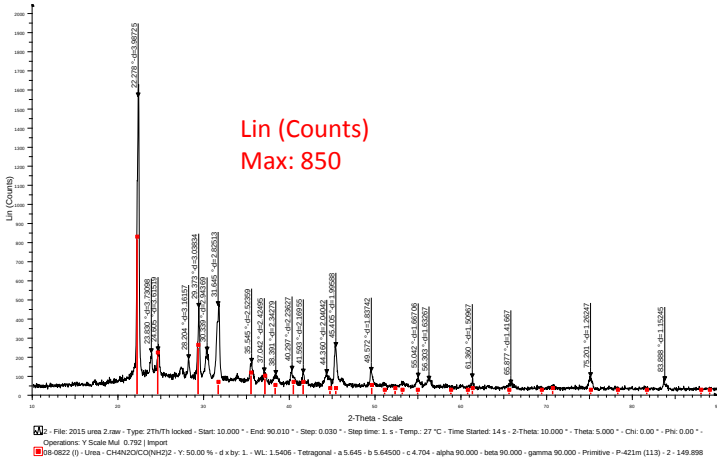
Sample 5 showed the lowest lin (counts) for urea (Figure 12), which is consistent with the observation that the sample had limited crystals visible to the naked eye. The noisier pattern indicates higher levels of amorphous material in the sample. Optical microscope observations also indicate that this sample had a different crystal structure compared to samples 1,2,3,4.

The two peaks at 19^{o} and 20^{o} theta angles in sample 5 could not be identified, despite a range of crystalline compounds being investigated. For all 5 samples numerous crystalline phases including combinations of struvite ($MgNH_4PO_4.gH_20$), potassium struvite ($KMgPO_4.6H_20$) and other potassium derivatives (KSO, KPO, KPOH, KClO), sodium and phosphate compounds and other combinations including the elements hydrogen, carbon, nitrogen and oxygen were looked for on the XRD.



Sample 1





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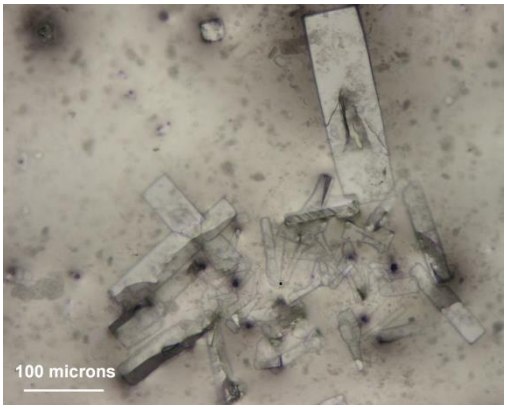

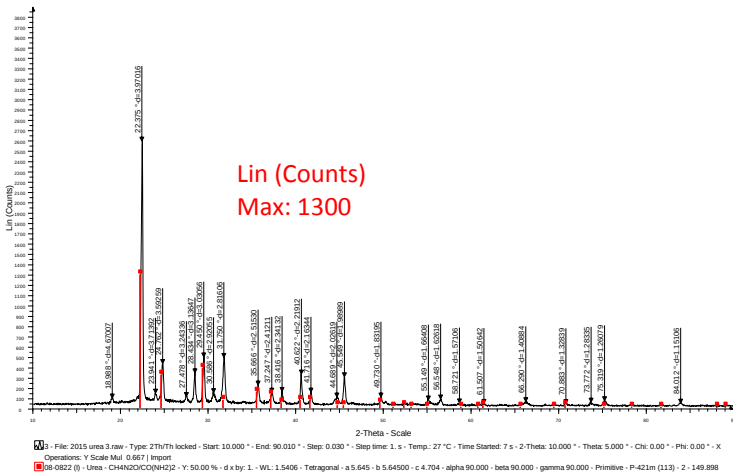

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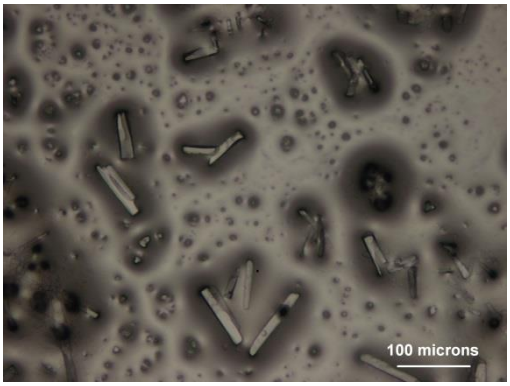
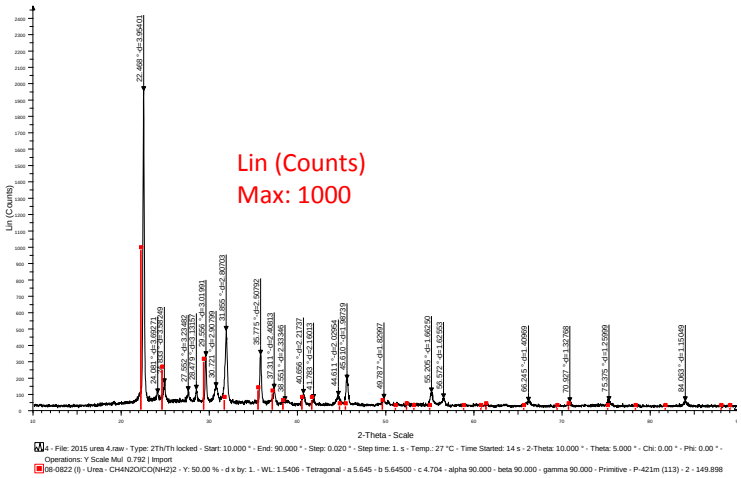
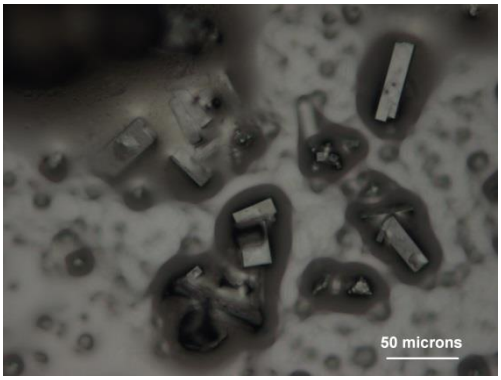
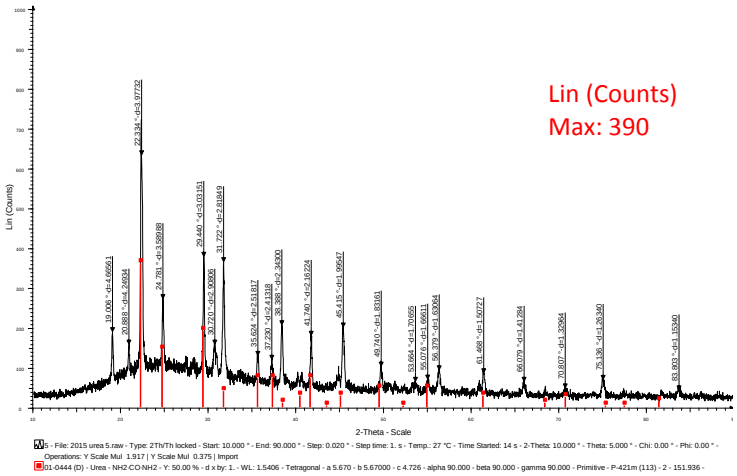




Sample 4





Sample 5

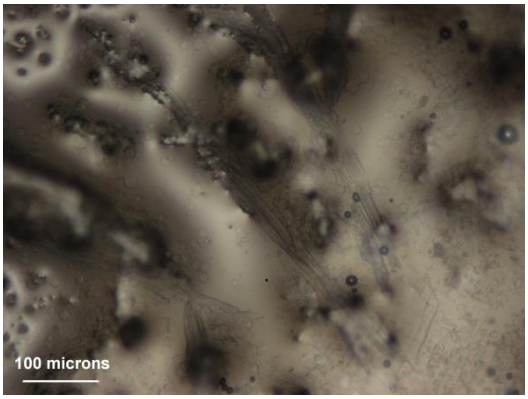


Figure 12: XRD spectrums and optical microscope images of samples 1-5

Other crystalline phases were also identified in the samples; such as halite in sample 5 and sodium potassium sulfate in sample 1 (Figure 13 and Figure 14, respectively).

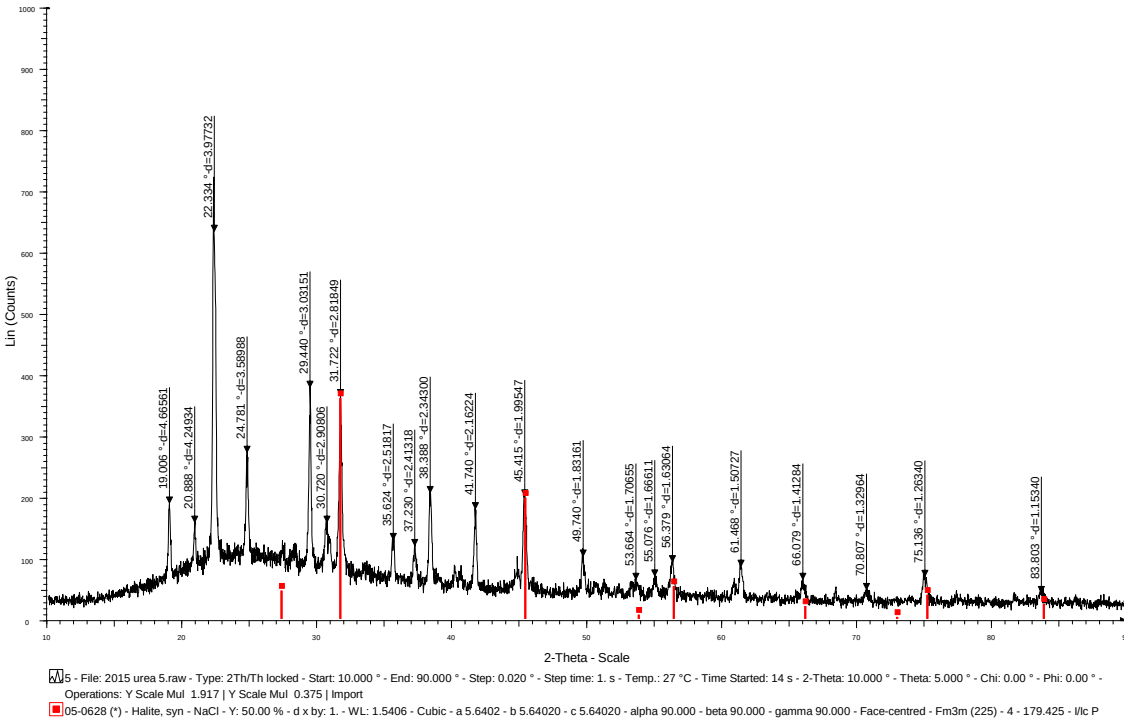


Figure 13: XRD halite spectrum- sample 5

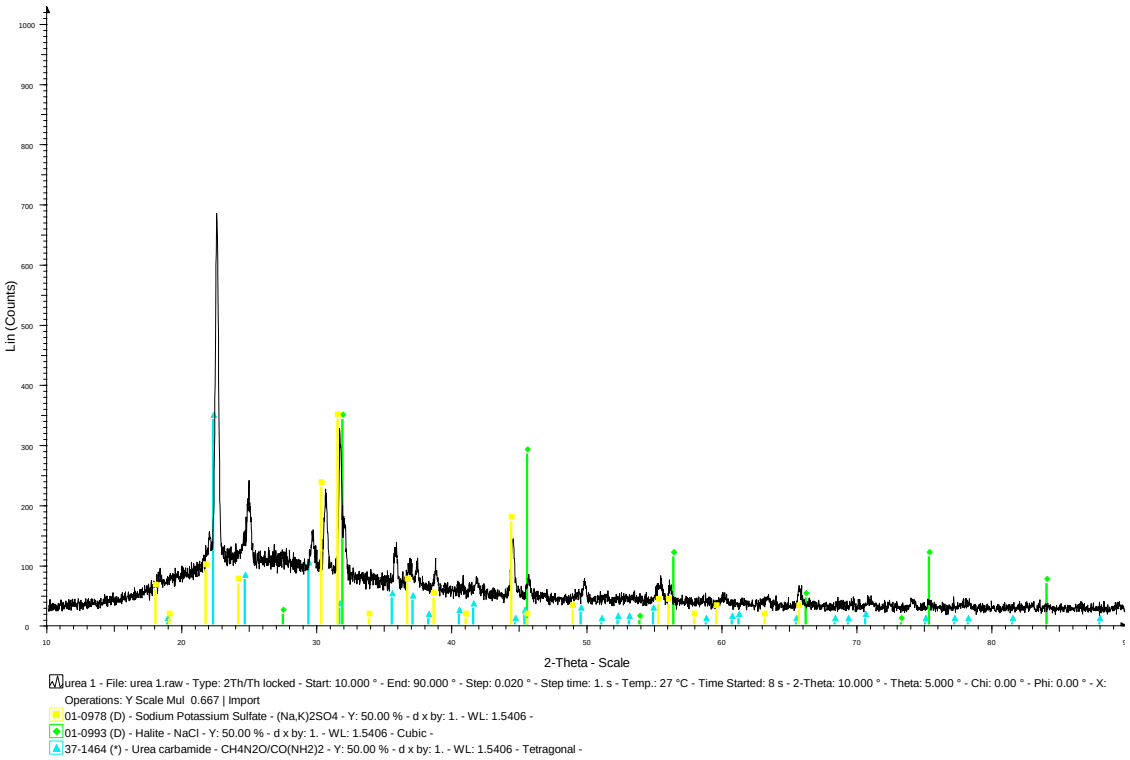


Figure 14: XRD spectrum- sample 1

3.3 Conclusion of the experimental work

Characterisation of the urine and testing the prototype has provided insight into the effectiveness of the current design.

Pasteurisation proved to increase the nitrogen levels in the urine, indicating that the pasteurisation stage is a necessary stage in the prototype. The metal concentrations in the urine are consistent with other literature that they are low and within safe levels (Karak and Bhattacharyya, 2011). Temperature and ventilation have been shown to have significant impacts on the evaporation rate. These are both important factors that would influence field trials if they were to occur as air flow and temperature would be much more variable than in the lab trial here. Repetition of the experiments would be necessary in order to confirm optimal conditions for urine evaporation.

The results of the analysis on the dry samples confirm that urine evaporation leads to a dry product that contains urea. This confirms the results from a previous study on urea crystallisation led by Kristell Le Corre Pidou in November 2014 for WaterAid UK. In this work SEM-EDS analysis was able to confirm the presence of urea crystals in evaporated urine samples.

According to the current results, it is clear that the samples behave very differently depending on how they are prepared and stored. It can be concluded that the evaporation process needs to be complete and the sample perfectly dry in order to obtain urea crystals. However, achieving a fully dry and stable product is very challenging, with oven drying not successfully removing the water content. Atmospheric moisture is not removed in the oven and thus still captured by the product. Furthermore, it has been shown that urea crystals are unlikely to precipitate alone but in the presence of other salts.

Sample 5, with pasteurised urine evaporated at a low temperature has lower amounts of urea compared to the other compounds and has different crystal growth to the other samples. However, the effect of pasteurisation and varying evaporation temperatures on crystal growth and urea content cannot be concluded from the analysis and results of this experiment.

In addition to pasteurisation to improve the nitrogen yield, additional technologies would be acidification (Antonini *et al.*, 2012) or adding ash (Dutta 2012). Research could also be conducted into packaging options (for instance the addition of sand (Bethune *et al.*, 2014) to keep urea crystals stable for storage and transportation.

4 Potential for Implementation

Throughout Tanzania, maize is the most prevalent food crop being grown, it accounts for around 45% of all cultivated land in Tanzania; the majority of maize grown can be found in the Southern Highlands, the lake and northern zones, such as Dar Es Salaam, Lindi, Singida, the coast and Kigoma (Stephen *et al.*, 2014).

In 2010, over 250,000 metric tonnes of fertiliser was imported into Tanzania, of this over 150,000 metric tonnes of it was Nitrogen fertiliser (Benson *et al.*, 2013). Between 2006 and 2010 urea was 61% of the total imported fertiliser (Benson, Kirama and Selejio, 2013). The high usage of urea as a fertiliser is partly due to the fact that the government, in 2003, began to subsidise urea and DAP fertiliser (Ariga and Heffernan, 2012). This also explains one of the reasons why maize is so popular to grow, as urea is a prevalent fertiliser and is shown to be particularly effective on maize (Schönning, 2001).

85% of the farms that are producing maize are small farm holders of <10 ha in size (Kaliba *et al.*, 1998). Despite this fact, a mere 9% of farmers in 2008 were using fertiliser on a regular basis. Indeed, the cost of fertiliser for these small farms is too high due to the additional costs incurred by having to import and transport it (Benson *et al.*, 2013). There is also a lack of information available to the farmers on how best to use the fertilisers (Kaliba *et al.*, 1998). The price of the crops are very fickle and make the additional investment in fertiliser more of a risk, in the past the government has even stopped farmers from selling their crops using a government directive (Benson *et al.*, 2013).

However, current use of urea crystals in farming and results of a urine reuse investigation in April 2014 seem promising. Urine is deemed to be much cleaner than faeces (Höglund *et al.*, 2002). There currently are signs that urine is being accepted as a fertiliser, indicated by the several countries that now use it (Larsen *et al.*, 2001).

According to WaterAid, Tanzania currently imports urea from China, costing £13-33/ 50kg bag, and is highly dependent on the cost of natural gas as it is a primary material. Therefore manufacture of urea for fertiliser from urine seems like a cost effective option.

The price at which the urine-derived urea crystals are sold would have to be competitive enough to compete with the industrially produced fertilisers. Recently there is a potential for nitrogen fertilisers to be produced in Tanzania due to the discovery of offshore reserves in Southern Tanzania (Benson et al, 2012). Pending a feasibility study it may also have to compete with a new urea fertiliser plant. There are currently no domestically produced nitrogen fertilisers in Tanzania, only in South Africa and North Africa (Ariga and Heffernan, 2012).

With no solution to avoid the deliquescence of the crystals, the selling of crystals would not be viable in a developing country. With deliquescence occurring in a humidity of around 80% and once the temperature reaches 18°C (Mills *et al.*, 2010).

Table 12 below shows that this is potentially a year-round issue in Tanzania with the possible exception of June, July and August (although this would require further research).

Table 12: Temperature and humidity in Tanzania
(Water Development and Management Unit and Climate Change and Bioenergy Unit, 2013)

Month	Min Temp	Max Temp	Humidity
	°C	°C	%
January	24.3	31.1	82.0
February	23.9	31.5	81.7
March	23.4	31.9	86.3
April	22.7	30.6	87.0
May	21.4	29.8	83.7
June	19.4	29.3	79.7
July	18.5	28.8	78.7
August	18.5	29.2	79.7
September	18.8	29.7	81.3
October	20.0	30.2	87.7
November	22.0	30.7	84.7
December	23.5	31.1	83.0
Average	21.4	30.3	82.9
Min	18.5	28.8	78.7
Max	24.3	31.9	87.7

The prototype could not currently be scaled up to a viable business in Tanzania. A way of pasteurising the urine at 80°C-100°C (Sahrawat, 1984) would need to be found, in order to succeed in deactivation of urease and thereby reducing nitrogen loss. A solution to the issue of the crystal deliquescence is also required to make their transportation and packaging possible. A way of optimising the system to increase the yield is also necessary.

A flow chart in Appendix 5 illustrates the process, methods, potential issues and solutions of the current system. An example of implementation of the system for fertilising 1 ha of fields was designed based on this flow chart. Figure 15 summarises the model. Full details of the calculations are available in Appendix 6 andAppendix 7.

Taking the average daily production of urine of 1.5 kg/pp/day (Corwin, 2011), urine collection from 226 individuals' daily urine would be required to produce around 4.4 kg of fertiliser, the average amount of fertiliser being applied to 1 ha (The World Bank, 2015). Hence this gives a volume required for storage of 338.5 L.

Schools should be targeted as potential locations for urine collection. However, as children produce roughly half the amount of urine as an adult, and given that the

average size of a Tanzanian government run class is 66 children (UNICEF, 2011), it would take 7 days to collect 338.5 L of urine.

A maximum surface area of 2.1 m² per pasteurisation plate was chosen, having a treatment capacity of 7.5 L (0). The total volume of urine would need to be pasteurised using 45 pasteurisation plates, either successively or with several in parallel. With the lowest temperature in Tanzania being 23.5°C, it would take at least 1 day to reach the suggested temperature of 72°C.

Following the pasteurisation, another storage tank is needed. It should be large enough to hold enough urine for a 10 ha farm, thereby being around 4,000 L.

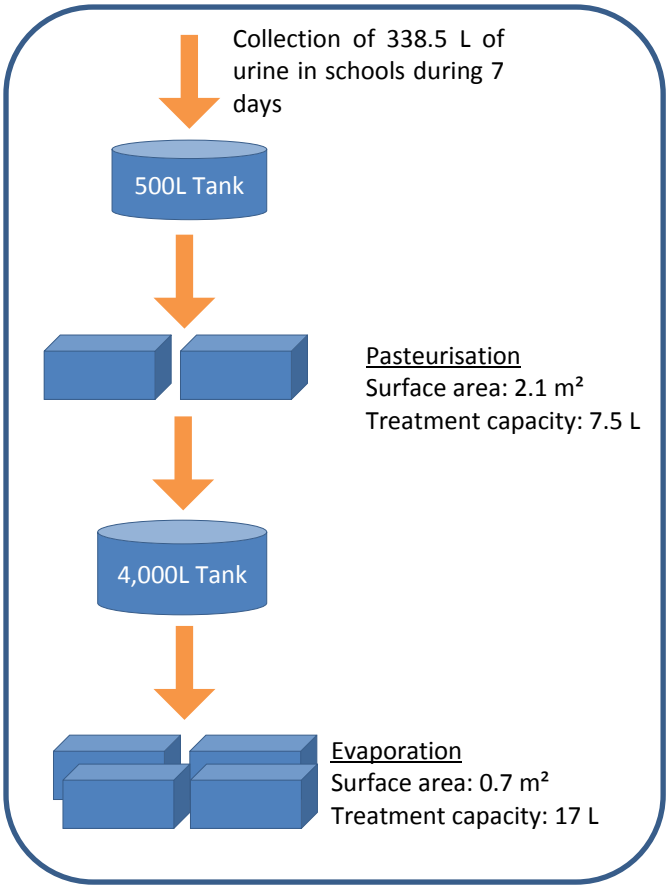


Figure 15: Model of implementation

The evaporation stage follows. It should be noted that the evaporation trough shouldn't be too large, as the entire system has to be covered by a glass panel. Therefore, a maximum surface area of evaporation was chosen, equal to 0.7 m² with a volume of 17 L of urine. This surface would allow several evaporation batches to run at the same time. However, as it would only treat 17 L of urine, this process would need to be repeated up to 20 times, with each evaporation time lasting for 2 weeks. Even if 4 evaporation batches were run in parallel, 10 weeks would be needed to evaporate the entire 338.5 L.

This model shows that the prototype is currently not viable as a business model. The two major issues requiring further research arising from the model are:

- The yield: the required volume of urine is very large to generate sufficient fertiliser.
- Time or space: it takes a long time to evaporate and pasteurise and in order to speed the process increasing the number of units is necessary (demonstrated in the model) which would require an unfeasible amount of space.

Conclusion

Following the design of a prototype to produce urea from urine using solar pasteurisation and evaporation, this study was commissioned by WaterAid UK. The efficiency of the evaporation stage of the prototype was tested on a lab set-up by varying the temperature and surface area of the evaporation trough. The yield, evaporation rate and quality of the crystals produced at the end of the process were also analysed. An evaluation of the technique for a potential implementation in Tanzania was also conducted through a comprehensive literature review.

Although this study showed that it is possible to evaporate water from urine, its total drying was more problematic. The end-product was wet and difficult to handle and store, in addition to small yields being obtained. Furthermore, although the presence of urea crystals was confirmed in the product after oven-drying, the amount could not be determined from the analysis performed. Generally, the quantities of compounds found in the urine were smaller than expected possibly due to urine dilution or diet of the individuals.

From the analysis of the composition of urine and of the dry-product, it is apparent that the pasteurisation is an important factor. The experiments showed that pasteurised urine contains significantly more nitrogen than unpasteurised urine and influenced the evaporation rate. A possible explanation is that the pasteurisation deactivated the enzyme urease, reducing the amount of nitrogen loss through hydrolysis.

Regarding the physical parameters, the influence of the size of the evaporation trays was not conclusive as the variations in the evaporation rate were probably due to an uneven distribution of the lamp over the urine.

Regarding the implementation of the prototype, it seems well suited to the Tanzania in terms of the materials, costs and acceptance of urine reuse. Moreover, there seems to be a potential in terms of demand for an implementation in Tanzania. However, without further optimisation the technology it seems to face various issues linked to the stability of the end-product, yield and nitrogen recovery.

Therefore, there is a need for further research to find solutions to increase the yield and quality of the final product. An interesting area of work would be the possibility of reaching the temperature needed for total urease deactivation during the pasteurisation stage using solar heat only. In addition to pasteurisation to improve the nitrogen yield, additional technologies would be acidification or adding ash. Research could also be conducted into packaging options (for instance the addition of sand) to keep urea crystals stable for storage and transportation.

Finally, this study showed that the prototype could not currently be scaled up to a viable business in Tanzania. However there is a clear market for an affordable fertiliser and potential for urine reuse in Tanzania which could be in the form of an optimised urea recovery technology.

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Appendices

Appendix 1. Climatic data in Temeke municipality area (Tanzania)

Values of the different parameters were adapted from ClimWat climatic data (Water Development and Management Unit, 2013). The presented values were calculated from the monthly average between available data in 3 stations: Dar Es Salaam, Dar Es Salam airport and Ubungo Dar. Each of them is located less than 30 km far from Temeke Municipality.

Therefore, they give an indicative value of monthly temperature and humidity in the area of Temeke Municipality.

Table 13: Monthly climatic data in Temeke Municipality

	Min Temp	Max Temp	Humidity	Wind	Sun	Rad	ETo
Month	°C	°C	%	km/day	hours	MJ/m²/day	mm/day
January	24,3	31,1	82,0	181,5	7,2	20,9	4,7
February	23,9	31,5	81,7	160,0	7,4	21,4	4,8
March	23,4	31,9	86,3	108,0	6,3	19,4	4,3
April	22,7	30,6	87,0	99,0	4,7	15,8	3,5
May	21,4	29,8	83,7	117,0	6,2	16,6	3,7
June	19,4	29,3	79,7	147,0	6,9	16,7	3,7
July	18,5	28,8	78,7	155,5	6,8	16,9	3,8
August	18,5	29,2	79,7	151,0	6,9	18,4	4,0
September	18,8	29,7	81,3	155,5	6,9	19,8	4,3
October	20,0	30,2	87,7	151,0	7,4	21,2	4,4
November	22,0	30,7	84,7	151,0	7,6	21,5	4,6
December	23,5	31,1	83,0	151,0	7,3	20,8	4,7
Average	21,4	30,3	82,9	144,0	6,8	19,1	4,2
Min	18,5	28,8	78,7	99,0	4,7	15,8	3,5
Max	24,3	31,9	87,7	181,5	7,6	21,5	4,8

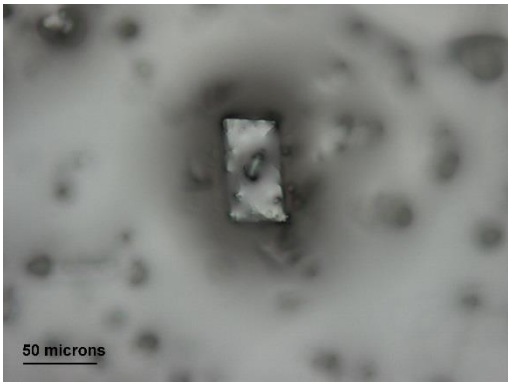
Appendix 2. Options for nutrient recovery

Table 14: Overview of treatment methods for nutrient recovery from source-separated urine (Maurer, 2006)

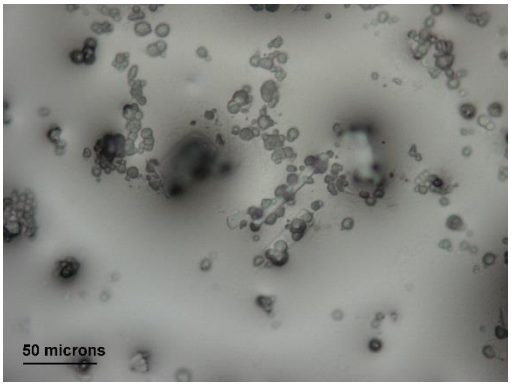
	Hygiene	Vol. reduction	Stabilisation	P-recovery	N-recovery	MP elimination	Nutrient–MP separation	Nutrient elimination	Solidification	Need of pre/post-treatment	Info literature
Hygienisation											
Storage	+	o	o	o	o	o	o	o	(+)	-	+
Volume reduction											
Evaporation	+	++	+	++	++	o	o	o	++	+	o
Freeze-thaw	?	+	o	++	++	o	o	o	o	o	+
Reverse osmosis	?	+	o	++	++	o	o	o	o	+	+
Stabilisation											
Acidification	+	o	++	o	o	?	o	o	o	o	+
Microfiltration	+	o	++	o	o	o	o	o	o	o	+
Nitrification	+	o	++	o	o	?	o	o	(+)	o	+
P-recovery											
Struvite	o	++	+	++	+	o	++	o	++	o	++
N-recovery											
Ion exchange	o	+	o	o	++	o	+	o	++	o	+
Struvite	o	++	+	++	++	o	++	o	++	o	++
NH_3 stripping	o	+	o	o	++	o	++	o	o	o	+
Isobutylaldehyde-diurea	o	+	o	o	++	o	+	o	+	o	+
Nutrient removal											
Anammox	+	o	++	o	o	?	+	++	(+)	+	++
Others	+	o	+	(+)	o	?	o	+	(+)	(+)	+
Micropollution removal											
Electrodialysis	++	+	+	+	+	o	+	o	o	o	+
Nanofiltration	++	o	+	o	o	o	++	o	o	+	+
Ozonation	+	o	+	o	o	++	o	o	o	o	+

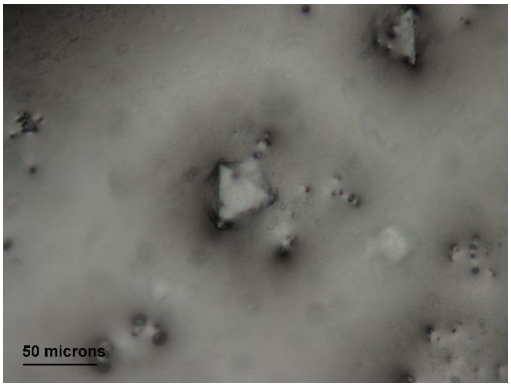
The columns represent the goals that can be achieved with a specific process; the rows list the technological process.Legend: o: no effect, +: positive effect, ++: strong effect, –: not applicable.

Appendix 3. Optical Microscope Analysis



Sample 1





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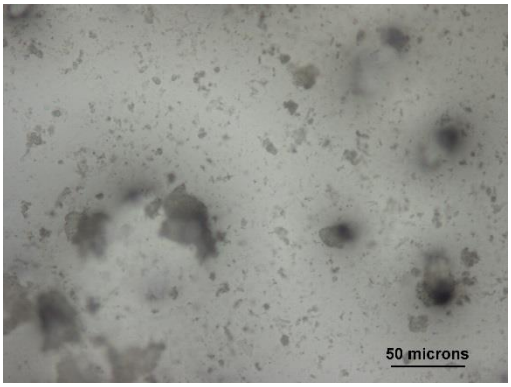

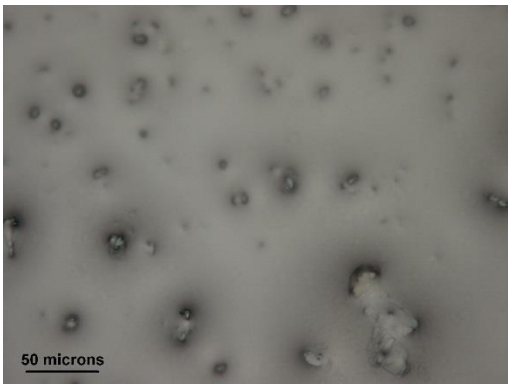

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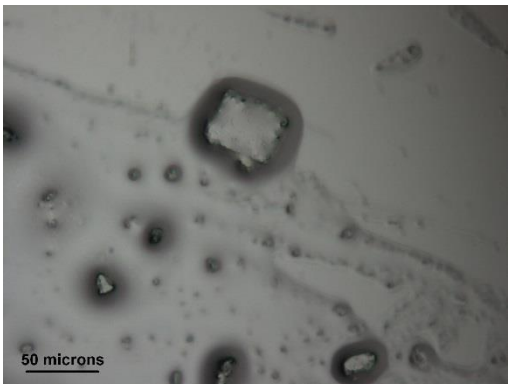

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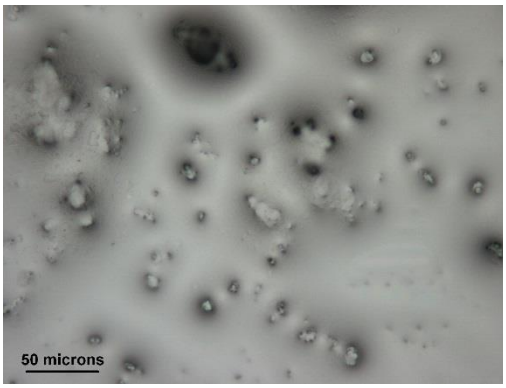
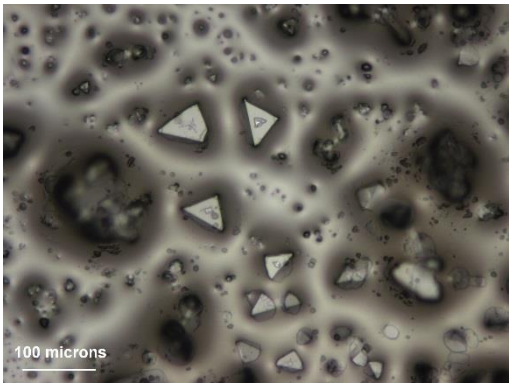
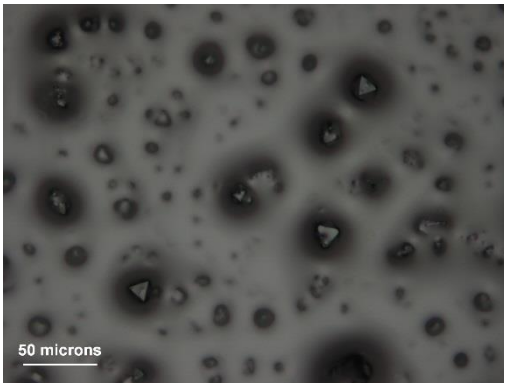
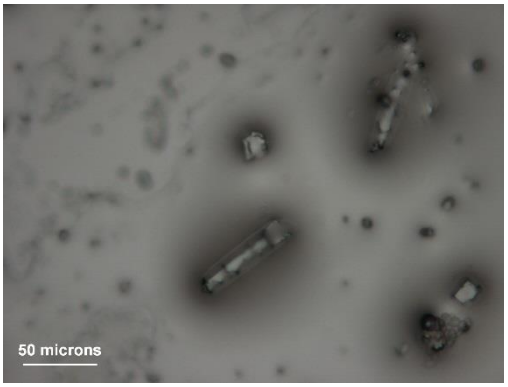


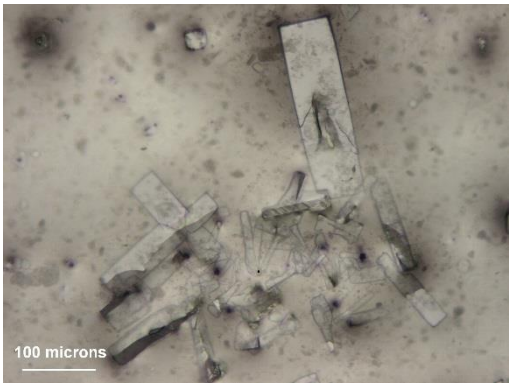
Figure 16: Micrographs of samples 1-5 pre-oven drying

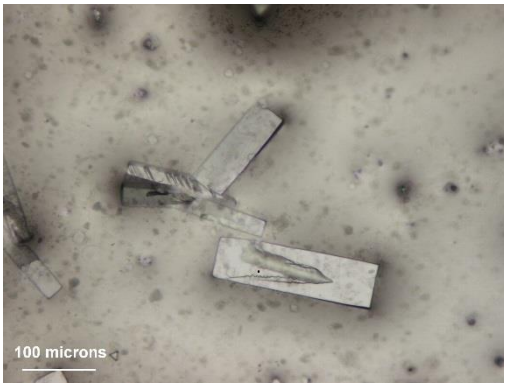




Sample 1







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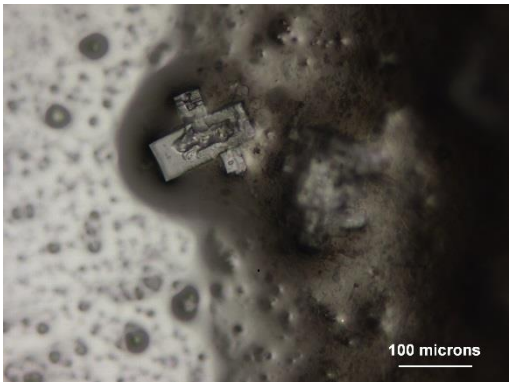

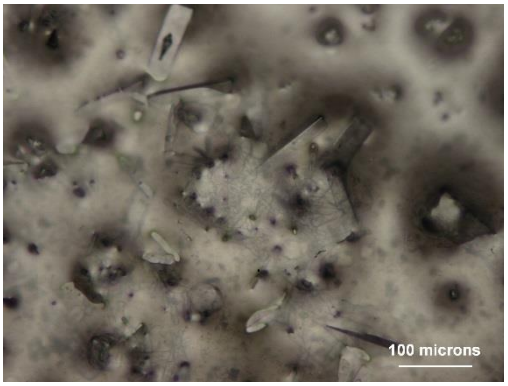

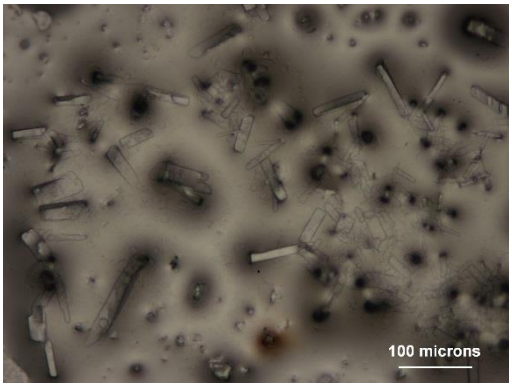

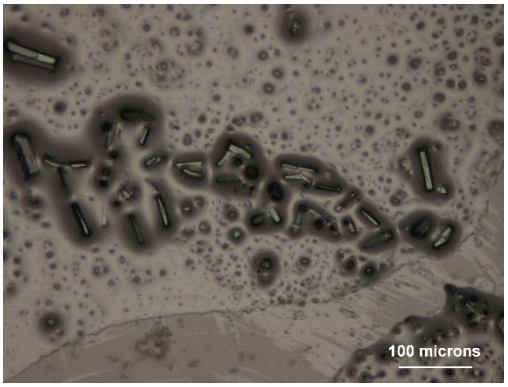

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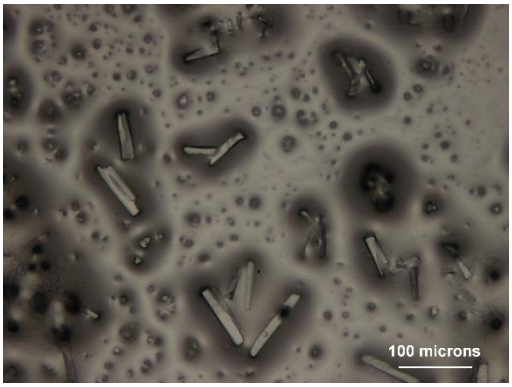

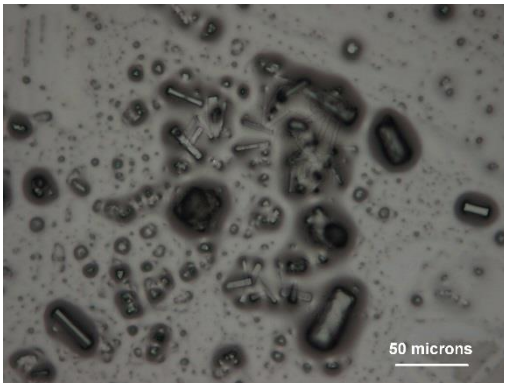

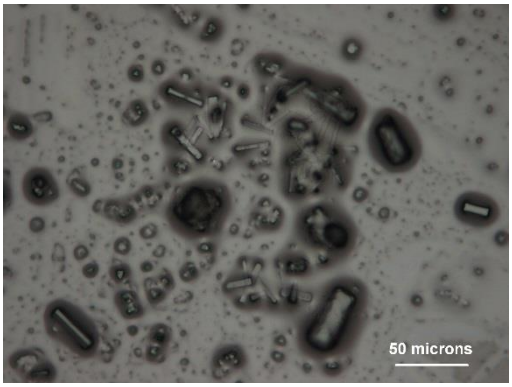

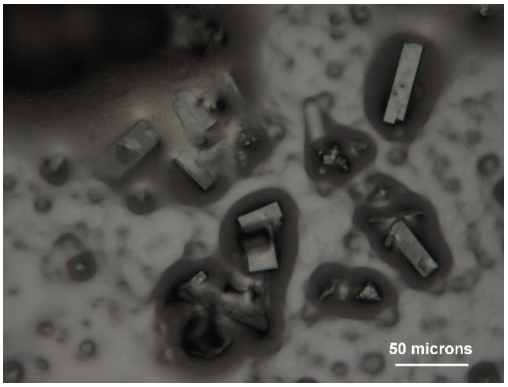

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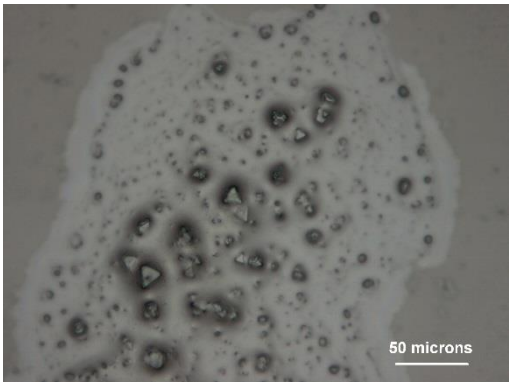

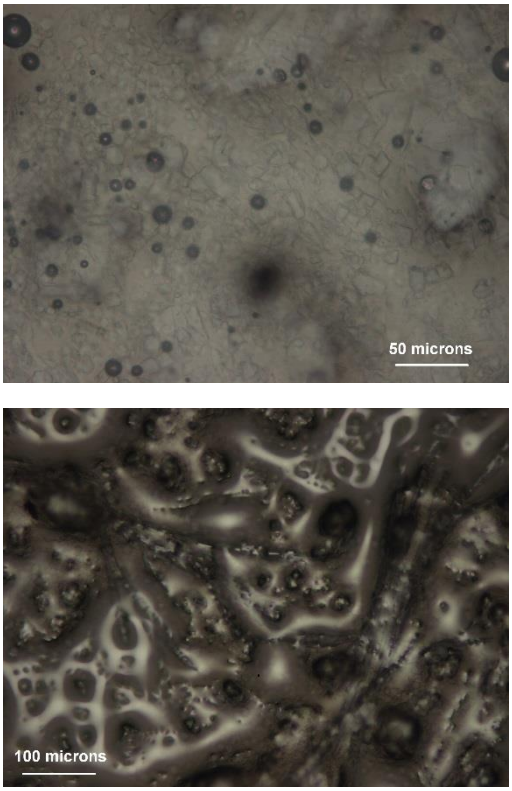

Figure 17: Micrographs of samples 1-5 post- oven drying

Appendix 4. XRD Analysis

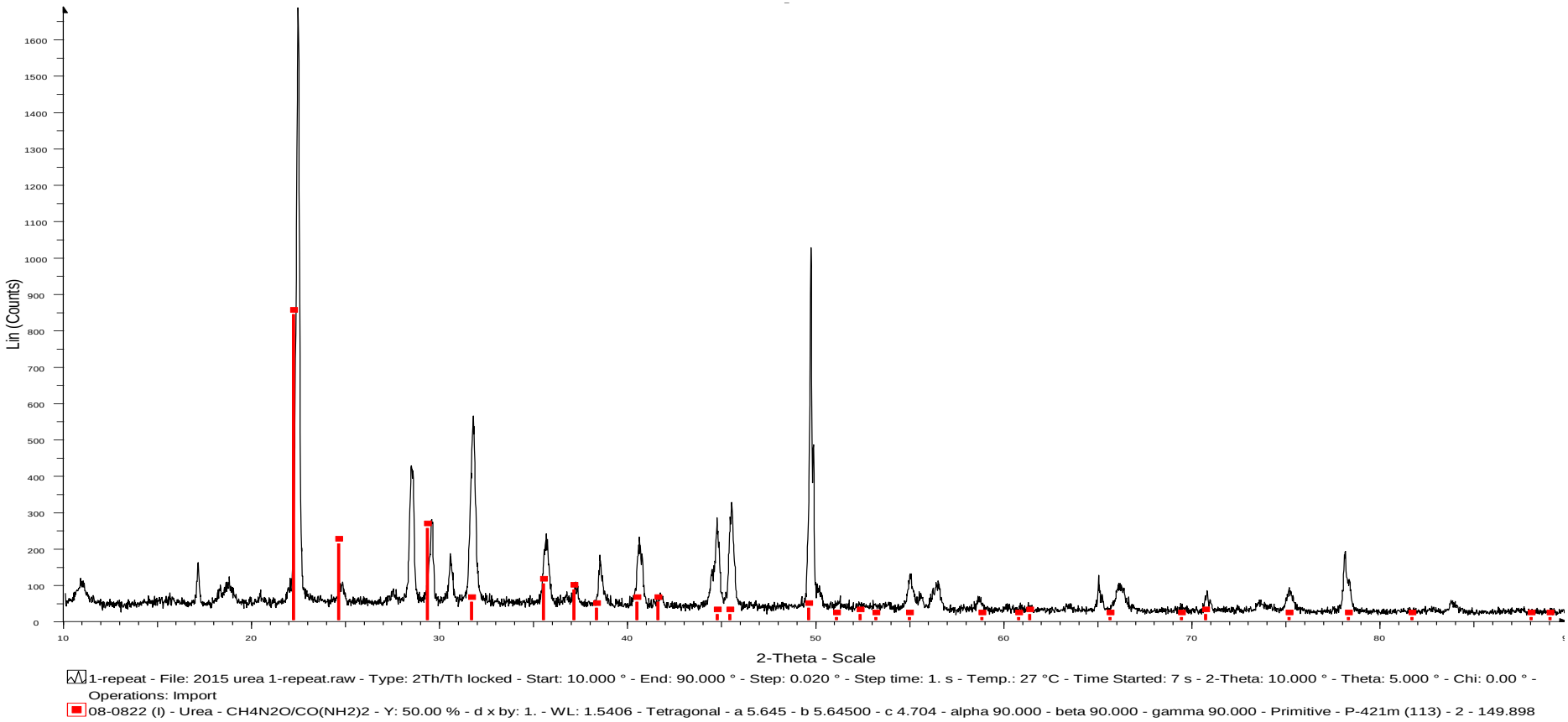

Figure 18: XRD spectrum urea- sample 1

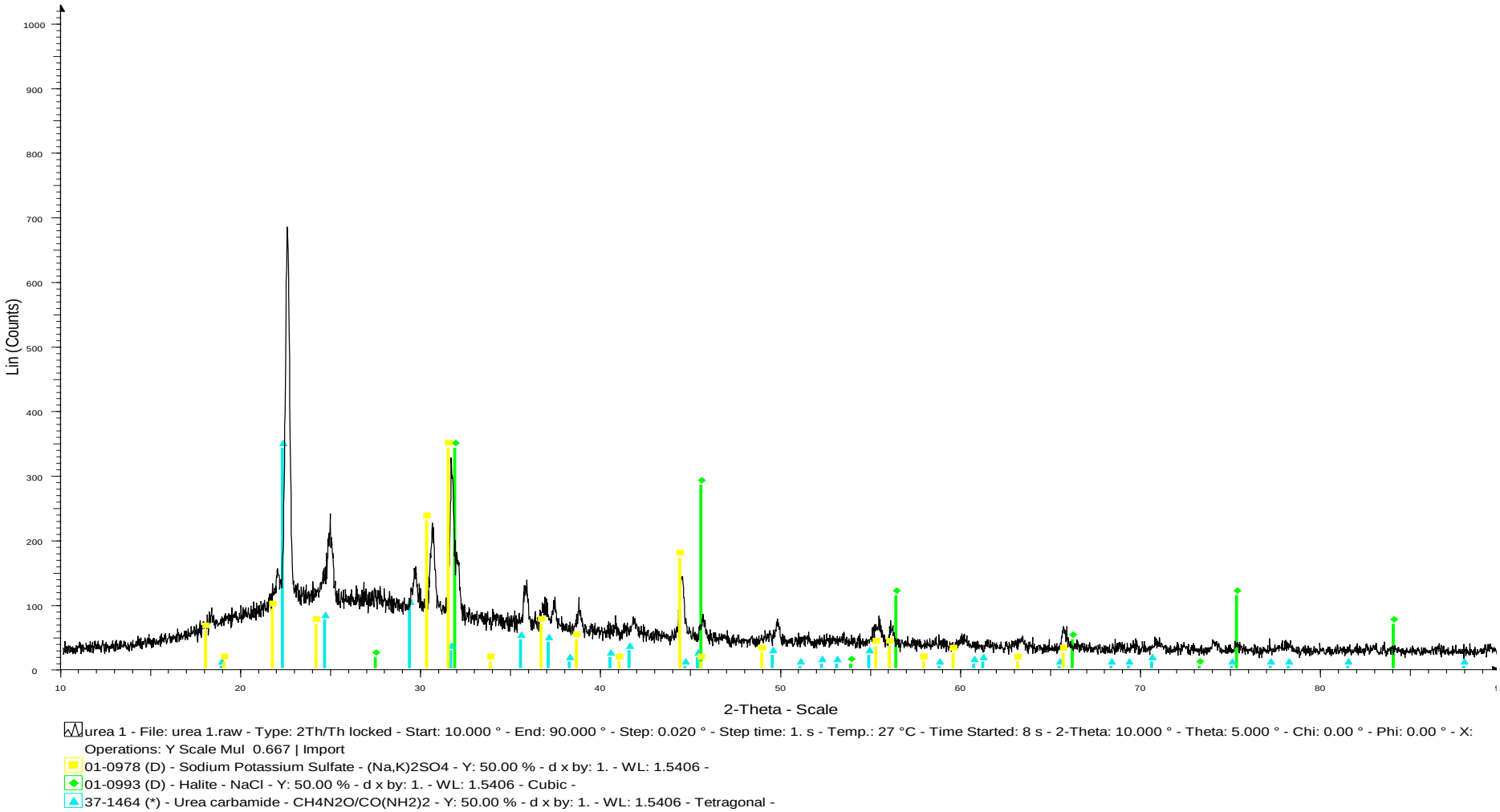

Figure 19: XRD spectrum sodium potassium sulfate (halite incorrect)- sample 1

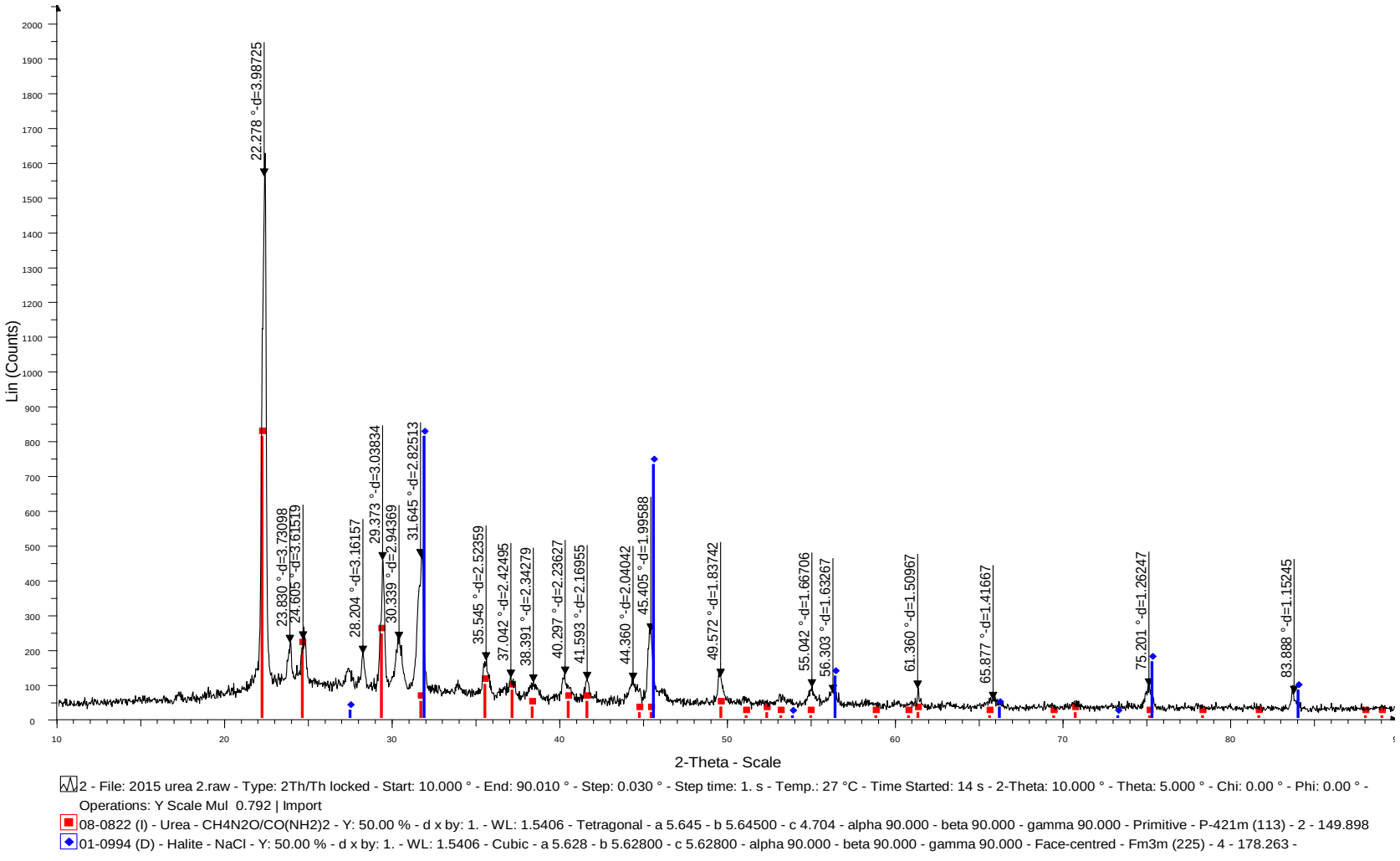

Figure 20: XRD spectrum urea and halite- sample 2

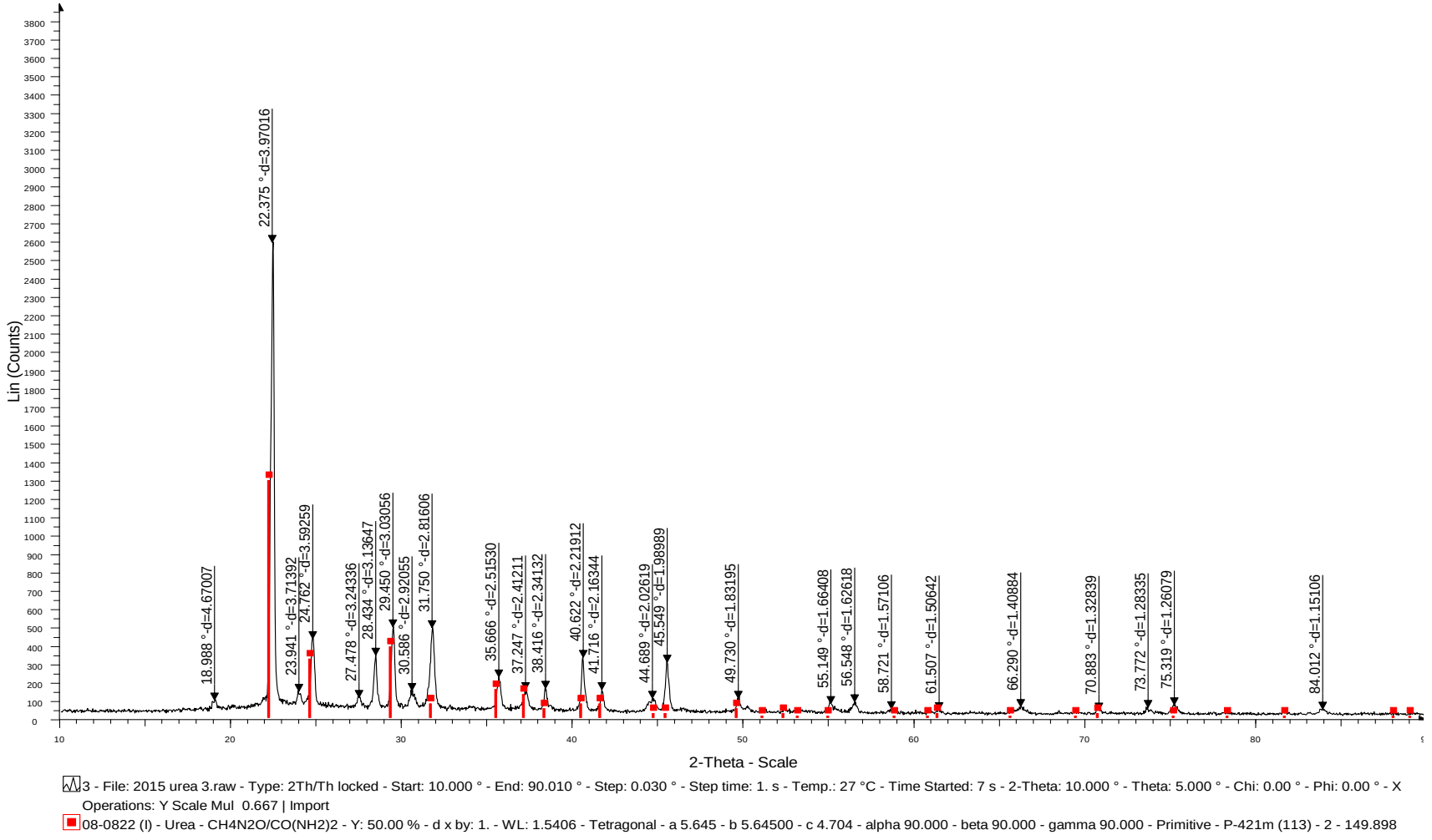

Figure 21: XRD spectrum urea- sample 3

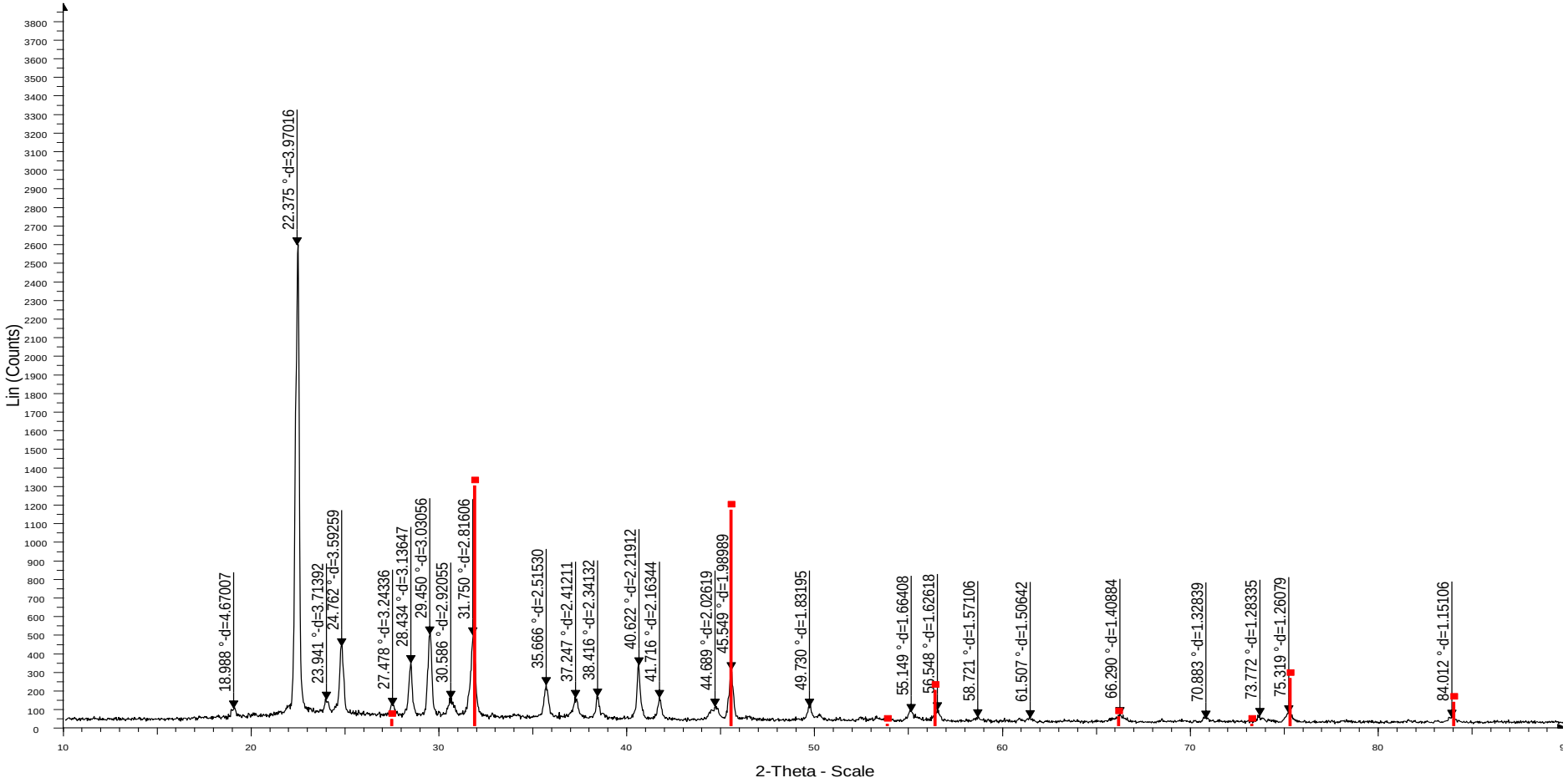

Figure 22: XRD spectrum halite- sample 3

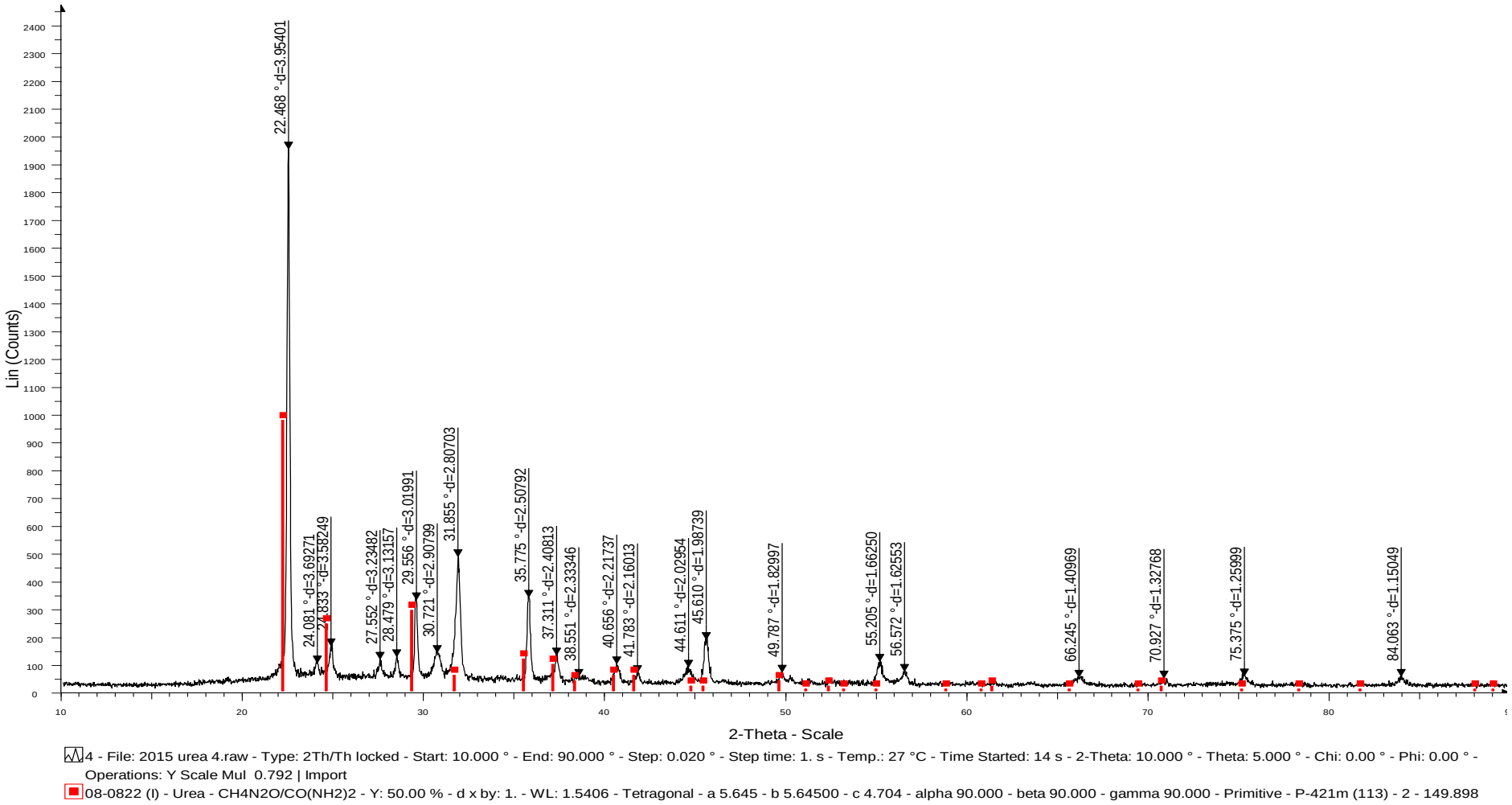

Figure 23: XRD spectrum urea- sample 4

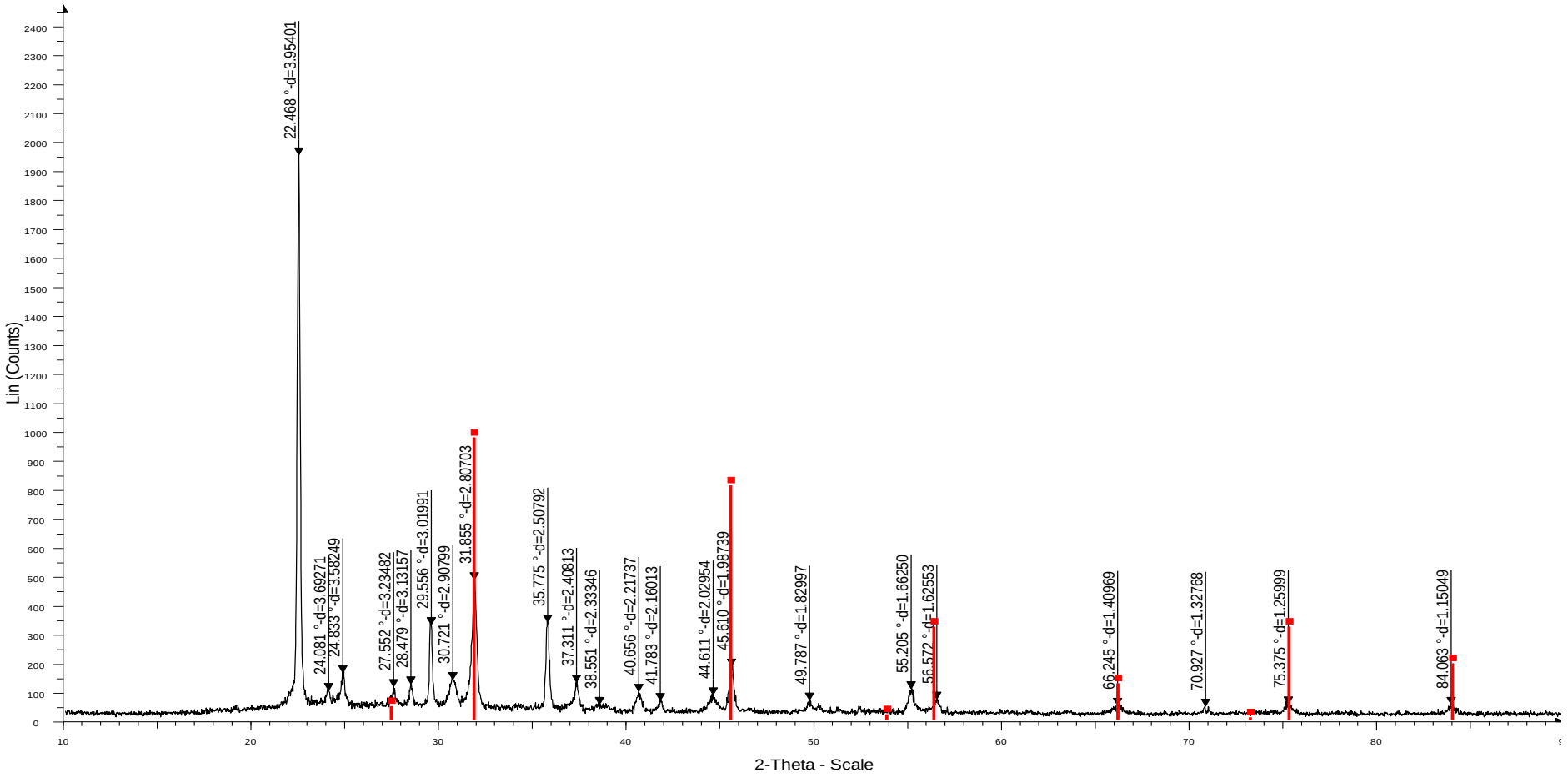

Figure 24: XRD spectrum halite- sample 4

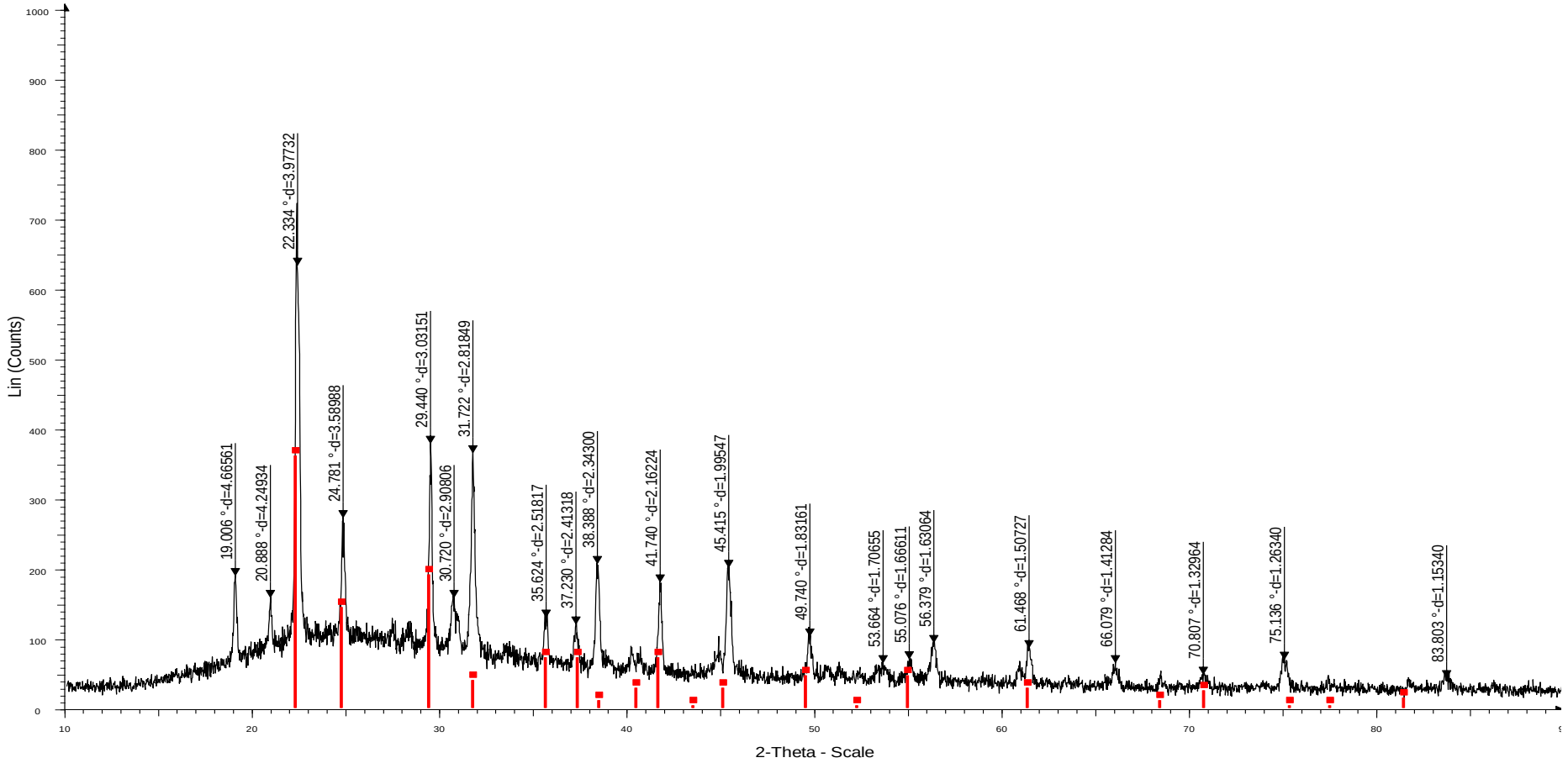

Figure 25: XRD spectrum urea- sample 5

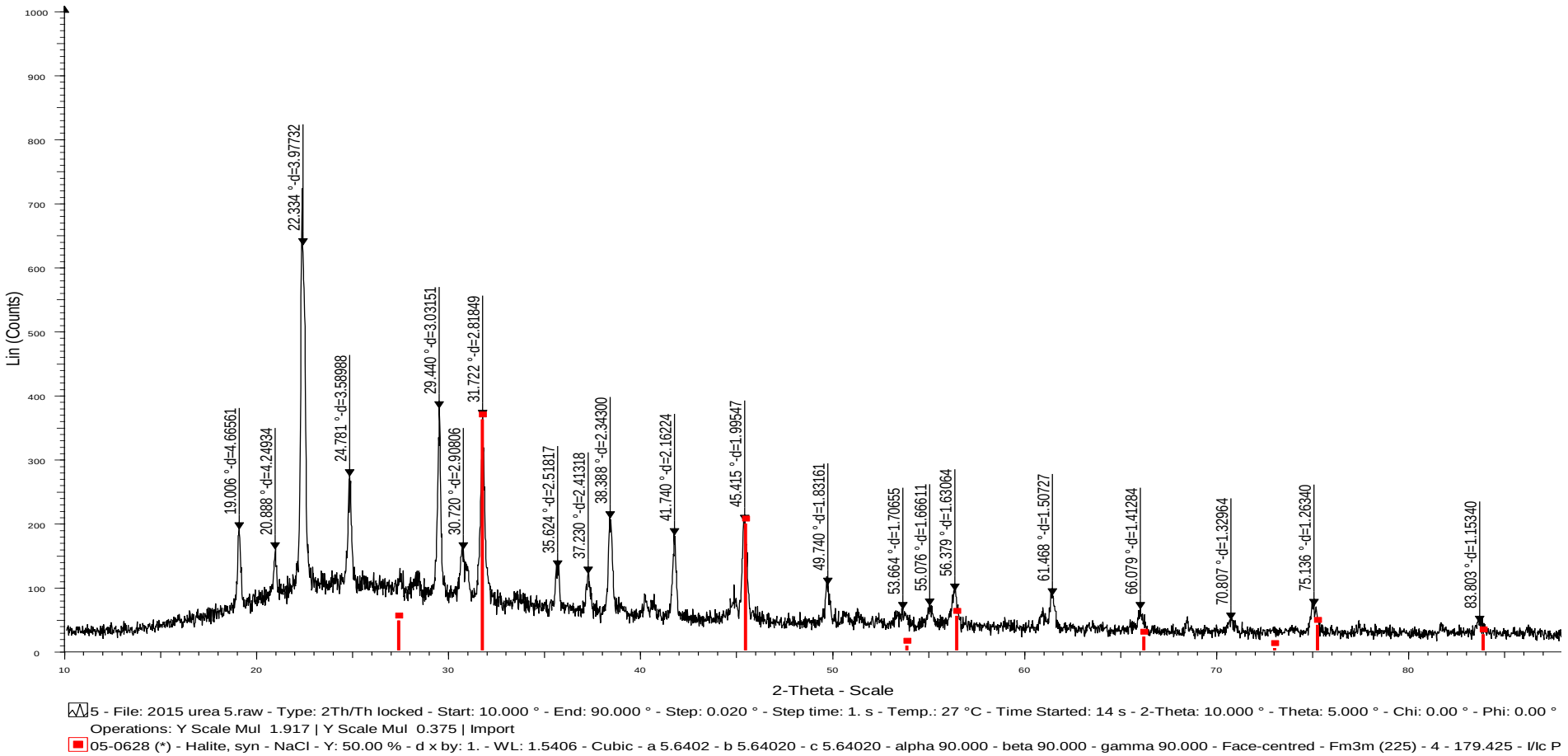

Figure 26: XRD spectrum halite- sample 5

Appendix 5. Flow chart

Stage of Process	Methods	Potential Issues	Solutions
Hygienisation	Storage	Hydrolysis Ammonia Evaporation	Acidification/Nitrification or Pasteurisation High pH, high temperature or no ventilation
Stabilisation	Pasteurisation Acidification Nitrification	Additional costs and risk of handling acid.	
Volume Reduction	Evaporation Free Thaw Reverse Osmosis	Not as efficient Expensive and not as efficient	
Nitrogen Recovery	Precipitation Crystallisation	Deliquescence	Keep the temperature below 18°C and the relative humidity below 80%
Dispersal	Crystal form liquid	Ammonia Evaporation	Harrowing of the soil whilst spreading the fertiliser

Figure 27: Flow diagram of the system

Appendix 6. Implementation calculations

4.4 kg = 4,400 g

4,400 g / 13 g/L = 338.5 L (of urine required to produce 4.4 kg fertiliser)

338.5 L / 1.5 L = 226 person's daily urine required to produce 4.4 kg fertiliser.

Figure 28: Urine required per hectare

66 children x (1.5 L / 2) = 66 x 0.75 L = 49.5 L/day

338.5 L / 49.5 L/day = 6.83 days (7 days)

Figure 29: School urine output

Appendix 7. Prototype Implementation

For the pasteurisation, the number of people is chosen depending on:

- The number of times the pasteurisation stage has to run (here: 45 times to pasteurise 338.5 L)
- The maximum surface area of 1 evaporation unit = 2.1 m²

The chosen temperature is the minimum annual temperature in Tanzania (23.5°C).

Table 15: Model for the collection and pasteurisation of the urine (Courtesy of Paul Foulds)

Phase 1 - Collection	For 1 pasteurisation	
Number of people	5	
Urine produced per person per day	1.5	kg
Total urine produced per day	7.5	kg
Volume of storage tank	**0.0075**	m^3
Phase 2 - Pasteurisation		
Specific Heat Capacity of Water	4.186	kJ/kg K
Pasteurisation Temperature	345	K
Ambient (Air) Temperature	296.5	K
ΔT	48.5	
Energy required to raise urine to pasteurisation temperature	1522.6	kJ
	1.5	MJ
Solar Conversion Efficiency of Metal Plate	0.3	
Solar Insolation Rate	2412	$kJ/m^2/day$
Solar Energy captured by plate	723.6	$kJ/m^2/day$
Plate area required to achieve pasteurisation temperature	**2.1**	m^2
Length of sides if square	1.5	m

Calculations are made for 1 evaporation batch only.

Table 16: Model for the collection and evaporation stage of the prototype (Courtesy of Paul Foulds)

Phase 1 - Collection	For 1 evaporation batch	
Number of people	11	
Urine produced per person per day	1.5	kg
Total urine produced per day	16.5	kg
Volume of storage tank	**0.0165**	m^3
Phase 3 - Evaporation		
Evaporation coefficient, Θ	0.5517	kg/m^2h
Maximum time allowed for evaporation process	140	h
	396000	s
Evaporation rate required, g_s	2.27273E-05	kg/s
Humidity ratio in saturated air at the same temperature as water surface, x_s	0.3141	
Humidity ratio in dry air, x	0.005387	
Area of evaporation trough	$=3600g_s/(x_s-x)\Theta$	m^2
	0.7	m^2

The number of people is chosen depending on:

- The number of times the evaporation units has to run in order to evaporate the total amount of urine (here: 20 times to evaporate 338.5 L)
- The maximum surface area of 1 evaporation unit = 0.7 m²

The evaporation coefficient used is the one found from the experimental work (the fastest evaporation coefficient from the 5 experiments was chosen: 974.8 g of urine were evaporated in 23.4 hours for a surface area of 0.0754 m² → 551.7 g/m²/h).