ANALYSIS AND EXPERIMENTAL EXPLORATION OF A NANOFILTRATION MEMBRANE SYSTEM, IN THE CONTEXT OF POTENTIAL URINE TREATMENT PROCESSES

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ABSTRACT

This work examines the viability of a membrane system to recover salts in urine valuable for potential agricultural use, while separating these from any undesired components such as salts detrimental to plant growth. To this end, membranes were compared to processes currently in use, or those proposed for future use, for treating sewerage and desalinating brines, as urine is a saline solution. Membrane processes were considered favourable as they generally require less energy than the other processes, are modular and mechanically relatively simple, can be used in many environments, and usually require no chemical additives to achieve separation.

Many configurations involving membrane systems would be possible, the most promising involving a combination of microfiltration pre-treatment for organics and solids removal, a nanofiltration membrane to split the Nitrogen/Potassium/Phosphorous (NPK) from the sodium chloride, and a forward osmosis membrane for the final removal of sodium chloride to obtain potable water. The lack of detailed information regarding passing urine through nanofiltration membranes meant that the remainder of the project focussed on experimentally determining whether a nanofiltration membrane could perform the required salt separation. Three DOW-Filmtec polyamide membranes were chosen, namely the NF 270, NF 90 and XLE membranes, as they are readily available, commercially used membranes and polyamide seemed to be the most promising membrane material based on a review of the available literature.

Two solutions were tested using the membranes, namely stored urine and a synthetic urine solution. The flux achieved by the membranes, 80 – 100 $l/m^2.h$ for NF 270, 6 – 8 $l/m^2.h$ for NF 90, and 4 – 10 $l/m^2.h$ for XLE, followed the order of the Molecular Weight Cut-off (MWCO), would be sufficient for the *Reinvent the Toilet Challenge* (RTTC) purposes and was similar to literature values. The flux was still increasing with increasing Transmembrane Pressure (TMP) between 800 and 1250 kPa for the XLE membrane, indicating that higher TMP conditions are usable without loss of energy efficiency. Fouling resulted in negligible decrease in flux for the NF 270 membrane, a 15 % decrease in flux for the NF 90 and an 18 % decrease for the XLE membrane, all of which are within tolerable limits. The NF270 had the highest rejection for phosphorous (80%) and lowest for Na^+ (40%), which suggests that this membrane may be the most useful if phosphorous recovery was of primary importance.

The transport model suggests a high separation between phosphorous and sodium and ammonium and sodium, this was supported for phosphorous by previous work in literature but not during these trials. Neither literature nor these trials support the transport model with the ammonium/sodium split. The results suggest that perhaps using nanofiltration membranes for the recovery of phosphorous in conjunction with a second type of technology for the recovery of nitrogen, such as ammonia stripping, will be a viable process rather than membranes alone.

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SYMBOLS AND ABBREVIATIONS

AES	Air Evaporation System
COD	Chemical Oxygen Demand
DSPM	Donnan-Steric Pore Model
ES Model	Electrostatic and Steric-hindrance Model
FO	Forward Osmosis
HAP	Hydroxyapatite
IBDU	Isobutylaldehyde-diurea
MBBR	Moving Bed Biological Reactor
MP-AES	Microwave Plasma – Atomic Emission Spectrometer
MF	Microfiltration
MWCO	Molecular Weight Cut-off
NF	Nanofiltration
NP	Nernst-Planck
NPK	Nitrogen, phosphorous and potassium
PRG	Pollution Research Group
RO	Reverse Osmosis
RTTC	Re-invent the Toilet Challenge
SHP Model	Steric-hindrance Pore Model
TIMES	Thermoelectric Integrated Membrane Evaporation System
TMP	Transmembrane Pressure
TMS Model	Teorell-Meyer-Sievers Model
UDT	Urine Diverting Toilet
UF	Ultrafiltration
VCD	Vapour Compression Distillation

1 INTRODUCTION

1.1 Context of the study

The Bill & Melinda Gates Foundation has provided funding, to the University of KwaZulu-Natal, Pollution Research Group (PRG), amongst others, for a program known as the *Reinvent the Toilet Challenge* (RTTC). The engineering challenge to be addressed by the project is to re-think the entire toilet concept, eventually aiming to produce a system that can replace existing flush toilet technology with an equal, or better, user experience while treating the waste as a valuable resource. The outcome of this project would help to alleviate sanitation problems for approximately a third of the world's population, living in poverty in developing countries, who do not currently have access to modern sanitation systems. Further details regarding this project and others pursuing the same goal can be viewed on the Bill & Melinda Gates Foundation website [1].

Conventional water-flush toilets connected to waterborne sewerage systems and centralised treatment facilities rarely recover any potentially useful components from the waste being processed. It has been shown by agricultural experiments that waste can be a safe and valuable fertiliser [2, 3]. Due to the relatively low concentration of nutrients in the waste, compared to commercial fertiliser, the products would need to be used within a limited radius to be financially viable and environmentally profitable.

Thus the primary objective of the challenge is to produce a self-sustaining toilet that is able to convert human waste into sterilized fertilizer, potable water, mineral salts and energy suitable for powering the process. The waste processing facility will be off-grid, with no connection to water or electricity infrastructure.

1.2 Purpose of the study

Most of the nutrients present in human excreta are concentrated in the urine [2, 4], while most of the pathogens, apart from micro-pollutants, are concentrated in the faeces. Treating urine and recovering these nutrients is a key process of ensuring the economic viability of the new toilet system.

This study focuses solely on the processing of urine, most likely entering the proposed excreta treatment system via urine diverting toilets. Membrane filtration has been identified as being potentially useful to process urine in the RTTC context, as membrane systems are generally lower in energy consumption and in mechanical complexity than corresponding thermally or biologically based systems currently used in waste water treatment and desalination processes. The purpose of this study was to place membranes in the context of other possible treatment options and to identify the key knowledge gaps and fill in the knowledge through experimentation, focussing specifically on nanofiltration.

Some research has been done in this area but lacks details necessary for design purposes, such as fouling, flux, water recovery as well as the operating pressures used. The research was also only conducted on fresh urine or a synthetic version thereof. This study will help to quantify these to some degree.

1.3 Research Outcomes

Analysis and experimental exploration of a nanofiltration membrane system, in the context of potential urine treatment processes

i. Place membrane processes in the context of urine treatment by conducting a literature review of the current and developing methods used for urine processing, as well as for general solid-liquid and salt extractions from saline solutions, both on small and large scale.

ii. Identify the knowledge gaps which currently prevent the use of membrane systems for processing urine successfully beyond bench scale.

iii. Explore the knowledge gaps for the use of nanofiltration membranes through suitable experimentation. Specifically, to determine if nanofiltration membranes are capable of achieving an adequate separation between nutrients useful for fertilizer and sodium chloride, achieve a reasonable flux and water recovery while exhibiting acceptable fouling characteristics.

1.4 Significance of the study

While membranes have been used extensively to treat and desalinate water their application in the treatment of sewage water, and urine in particular, has not been widely explored. This study aims to determine if using membranes to treat urine is currently feasible, given available membrane technology. The study provides data regarding the separation potential of nanofiltration membranes in the key area of separating valuable components from unwanted components in the urine feed. The flux achievable and the fouling observed will also be discussed. In addition, the work will be done on both synthetic urine, as a basis for comparison, and stored urine, which has not been done before. This study will help to determine if a membrane system is worth pursuing or if another approach should be considered.

1.5 Limitations of the study

The processes considered here for treating urine are limited to those which have already been used for the treatment of waste water or the desalination of salt water, as these are seen as processes currently having the greatest potential for urine treatment.

The prospective treatment system also has limits set by the Bill & Melinda Gates Foundation as follows: the system should be robust and modular; consumables, including chemical additives, should be kept to a minimum; little to no energy should come from outside the treatment plant, although the exact energy source is not considered here; and the system should be universally applicable in terms of geography.

The membrane processes considered for experimentation are further restrained by those currently widely available and technically possible within the PRG laboratory. These are reverse osmosis, nanofiltration, forward osmosis, ultrafiltration and microfiltration.

The variation of feed properties and experimental parameters, such as temperature and pressure, for the experiments are limited both by time, in terms of amount of variability and number of chemical analyses possible, and analytical equipment availability, in terms of number of chemical analyses kits. The parameters and number of measurements conducted are also limited by the operating characteristics of the equipment used, specifically the low permeate flow due to limited membrane area.

2 LITERATURE REVIEW

2.1 Introduction

This literature review is broken down into five major research areas that together fulfil the first two objectives stated in section 1.3 and form the basis of the experimental section of the study.

The first area covered is a background on urine, focusing on the composition of urine and why it can be a valuable resource. This section will serve as a further explanation of why this study is taking place and what value it may have.

Next the various objectives considered, when selecting a suitable process to treat urine, are explained. Identifying objectives of treating urine allows for an easy grouping of the treatment processes and aids in comparing processes and deciding on the most advantageous processes for the purposes of this study. Included here are also comments on the types of processes associated with the treatment objective as well as the degree that this is covered in literature and industry.

This then leads to an explanation of the current and proposed processes of treating urine. A brief explanation of each process is given, along with a summary of the processes, compiled from an analysis of the literature in a table format for ease of comparison.

This summary is then used, along with experimental constraints stipulated in section 1.5 and explained in section 2.5.1, to define the membrane processes considered for the final treatment system. The processes to be considered are found by eliminating the unsuitable processes and selecting the most favourable combination of the remaining processes to achieve the necessary separation of valuables from the urine.

The last section provides further details on nanofiltration to provide a basis for the design of the experiments that follow.

2.2 Urine Background

Urine is a by-product of the body, formed in the kidneys, and is a means of excreting excess salts, by-products of cellular metabolism and any other soluble wastes that may be present in the body. Many of these soluble substances are rich in nitrogen resulting in urine containing, on average, 80 % of the nitrogen and 50 % of the phosphorus excreted from the human body [2, 4].

All nutrients not used for energy or cell generation in the human body are expelled as waste; nitrogen, phosphorous and potassium (NPK) are present in urine in forms that can readily be taken up by plants [5]. Recovery of these components provides the opportunity to (i) produce

an agricultural product with an economic value, for example total phosphorous produced from urine was approximately 1.68 million tons in 2009 [6], and (ii) close the nutrient cycle.

The waste components that must be separated from urine before it can be used as a fertiliser product include: sodium chloride, as too much sodium chloride has a negative impact on plant growth; and pharmaceutical compounds and endocrine disruptors, which build up in the environment and can be hazardous if consumed by humans. Urine diverting toilets (UDTs), which are toilets that collect urine separately from faeces, are seen as potential collection points for a toilet system [1]. Due to the human element in the use of UDTs, some cross contamination of the urine is to be expected. This means that some faecal matter may also be present in the urine stream and this will also have to be removed before use as fertiliser.

Table 1 shows the variation in urine composition. This variance is due to many factors including diet, which has a significant effect on pH and nutrient composition, and health, along with the accompanying medication taken, of the source [7, 8].

Grouping	Item	Molecular Weight	Range [mg/l]		
			Low	High	% *
	Total Solutes		36 700	46 700	
Nutrient - Potassium	Potassium	39.1	750	2 610	4.6%
Nutrient - Phosphorous	Phosphorous	31	410	1 070	1.9%
Nutrient - Nitrogen	Urea	60.1	9 300	23 300	40.7%
	Ammonia	17	200	730	1.3%
Nutrient - Other	Bicarbonate	61	20	560	1.0%
	Calcium	40.1	30	390	0.7%
	Other				0.3%
Pharma/Organics	Creatinine	113.1	670	2150	3.8%
	Hippuric Acid	179.2	50	1670	2.9%
	Citric Acid	192.1	90	930	1.6%
	Other				13.7%
Undesirable	Sodium	23	1170	4390	7.7%
	Chloride	35.5	1870	8400	14.7%
	Sulphur	32.1	163	1800	3.1%
	Other				1.5%

Table 1: Compounds forming approximately 80% of fresh urine solutes [7, 8]

*** Using values at maximum end of range (dry basis)**

2.3 Treatment Objectives

Research on urine processing indicates that the treatment processes, described in section 2.4, can be broken down into seven objectives [5]. The methods considered would aim to fulfil one or more of these objectives. Each of these objectives is explained below:

2.3.1 Disinfection

Urine can contain pathogenic organisms and prions, pathogenic agents resulting from protein in a misfolded form, which are undesirable when urine or any by-products may come into contact with humans, directly or indirectly, as these pathogens could spread disease [5]. Contamination with faecal matter is also possible, depending on the separation system used at the urine source, and this is undesirable not only because of the risk of disease but also because the faecal particles may interfere and hinder processes used downstream, such as causing fouling in membrane processes.

Although work has been done in the wastewater field to disinfect water, not much research has been done with regards to purely urine disinfection. Many processes will have some disinfecting potential but the effect of storage time on indicator pathogen deactivation has been studied most extensively [5]. The disinfection is achieved due to the rise in pH during urea hydrolysis resulting in the production of ammonia, which is a biocide thereby deactivating pathogens.

2.3.2 Volume Reduction

As discussed in section 2.2 urine contains nitrogen and phosphorous and has potential use as a fertiliser. The nitrogen and phosphorous concentrations in urine are far lower than commercial fertilizer. Therefore, storage, transport and application costs for commercial fertiliser would be far lower for commercial fertiliser than for urine. Unless this cost difference can be offset by the purchasing cost of the fertiliser, it will not be feasible to use urine without concentrating the nutrients. As an illustration of this problem: an average urine sample may contain around 0.4 – 1 g/l Phosphorous and around 7 – 9 g/l Nitrogen [2], while an average fertiliser may contain 38 g/l Phosphorous and 35 g/l Nitrogen [9]. A by-product of any volume reduction would be water, which, if processed properly within the treatment system, could be re-introduced into the local water system or used directly to fulfil any water requirements.

Volume reduction has been studied thoroughly for waste water and brines, with some studies done on urine [5]. The most promising technologies for this area include: evaporation, which can further be broken down into various possible technologies; or some type of high rejection membrane processes, such as reverse or forward osmosis.

2.3.3 Stabilisation

The single largest component of fresh urine, other than water, is urea, with a concentration between 9.3 and 23.3 g/l [7]. Urea contains much of the nitrogen in urine, it can be used directly as a fertiliser, if clean enough, and is relatively easily granulated. Therefore, for many applications it is desirable for the urea to remain in this form. However microbial activity causes organic matter to degrade, generating odours, and causing urea to hydrolyse [5]. The hydrolysis reaction is catalysed by the enzyme urease and the reaction is as follows:

$$CO(NH_2)_2 + 3H_2O \xrightarrow{urease} 2NH_4^+ + HCO_3^- + OH^-$$

Equation 1: Urea Hydrolysis [10]

The ammonium is in equilibrium with the dissolved ammonia in the urine following the hydrolysis reaction:

$$NH_4^+ + OH^- \leftrightarrow NH_3(aq) + H_2O \qquad pK_a = 9.3 \text{ @ } 25°C$$

Equation 2: Ammonium equilibrium [10]

Urea hydrolysis, depicted in Figure 1, causes the pH of the urine to increase to around 9.2 [11]; the precipitation of low solubility compounds such as struvite and hydroxyapatite (HAP) then occurs [11]; and nitrogen volatility, in the form of ammonia NH_3, increases [5]. The formation of volatile ammonia could lead to significant nitrogen losses, which would decrease the amount of recoverable nitrogen, thereby decreasing profitability.

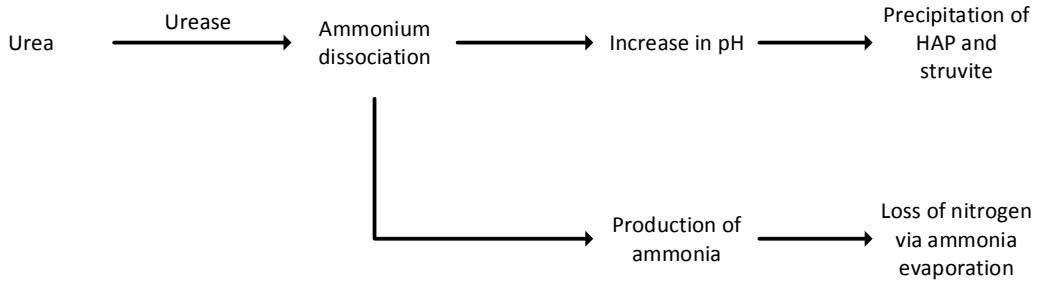

Figure 1: Urea Hydrolysis

Various processes to stabilise urea have been studied with the most promising processes including: acidification, partial nitrification, and micro and ultrafiltration [5].

2.3.4 Phosphorous Recovery

Phosphorous is mainly used in the fertiliser industry and is obtained through mining phosphorite, phosphate rich ore. This means that phosphate is a limited resource, although depletion of the ore is not as much a cause for concern as the decrease in the ore quality, in terms of phosphate concentration, which will lead to a possible price increase [6]. This means that recovering phosphorous from urine could be reasonably profitable in the near future, if suitable infrastructure for mass collection of urine is developed [6].

The recovery of phosphorous from urine is currently being studied quite extensively, with the main process under investigation being struvite and HAP precipitation [5].

2.3.5 Nitrogen Recovery

Nitrogen is an abundant resource, as it makes up 78% of the atmosphere. The problem is that the Haber-Bosch process, the process currently used in capturing nitrogen, and reacting it with hydrogen to convert it to ammonia, is energy intensive. Urine contains nitrogen in an already bonded form, urea. Recovering this could be useful in offsetting any other operating costs [5].

Nitrogen recovery from urine has not been studied as extensively as phosphorous recovery but some promising processes exist. Processes include ion-exchange, ammonia stripping and isobutylaldehyde-diurea (IBDU) precipitation [5].

2.3.6 Nutrient Removal

Even when urine is depleted of organics and pathogens, releasing the urine into the aquatic environment, be it river, dam or sea, can still cause problems for the local ecosystem. Urine has high nutrient concentrations, in the forms of nitrogen and phosphorous, which can cause problems such as excess algae growth, shifts in local species populations, dissolved oxygen deficit, production of toxins and excess nitrates in drinking water [5]. To control water pollution, it is possible to remove these nutrient without recovery. An example of this would be the conversion of any nitrogen present in the urine to N_2, which can be safely released into the atmosphere.

The processes used to achieve this include: biological oxidation of ammonia, with nitrite as the electron acceptor (the anammox process), and electrochemical oxidation of ammonia as well as the processes for phosphorous removal, such as precipitation with lime or biological removal [5].

2.3.7 Micro-pollutant Removal

Any of the pharmaceuticals and chemicals humans consume are excreted via urine along with many excess compounds the body produces. These are known as micro-pollutants and if they are released, along with fertiliser or into the water system, they can accumulate in the environment and may cause health problems for humans, who would consume them indirectly via plant and animal uptake [5]. Unless the micro-pollutants have a short life-cycle, or the ground or water will not be used by humans directly or indirectly, the micro-pollutants in urine should be removed or eliminated before use as fertiliser, or release into the water system.

Elimination of micro-pollutants uses processes similar to that of nutrient removal, mainly oxidation and adsorption, which are adversely affected by high Chemical Oxygen Demand (COD). Removal of micro-pollutants uses membranes and precipitation with processes such as electrodialysis, nanofiltration, ozonation and advanced oxidation [5].

2.4 Treatment Processes for Urine Disposal or Beneficiation

The treatment processes can be split into four general categories and are explained below:

2.4.1 Evaporation

2.4.1.1 Vapour Compression Distillation

In a vapour compression distillation (VCD) process, shown in Figure 2 below, saturated steam, coming from the evaporation of water from urine in the boiling chamber, is compressed to increase its temperature. This superheated steam is sent through the boiling chamber in a heating element. Here it releases latent heat through condensation into the surrounding urine, which results in further water evaporation and formation of saturated steam [12]. The now condensed steam then flows through the feed tank to preheat the feed solution. This method recovers 96 % of the water with an energy requirement of between 277 and 396 MJ/m^3 of water recovered [5]. The brine could potentially be used as a fertilizer but the presence of sodium chloride and pharmaceuticals would necessitate further treatment.

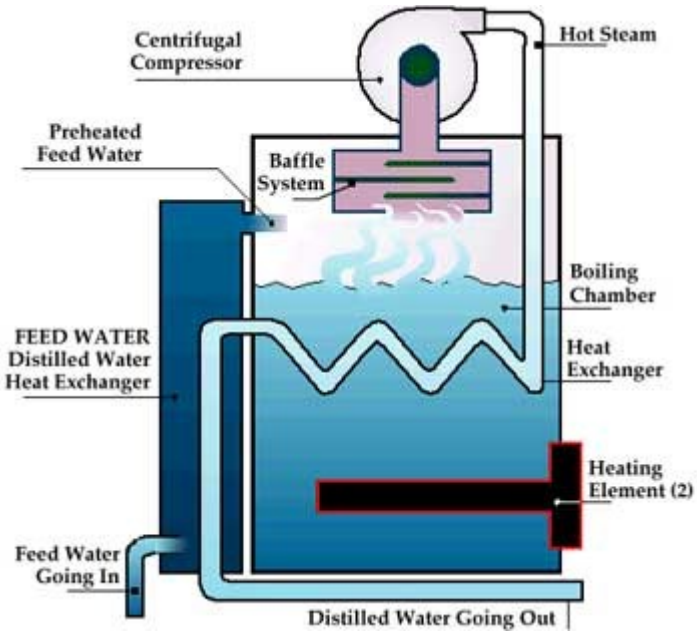

Figure 2: Vapour Compression Distillation [13]

The problems with this process include high energy requirements and the loss of ammonia during evaporation. Ammonia evaporation can be controlled through urine acidification or using fresh non-hydrolysed urine [5].

2.4.1.2 Thermoelectric Integrated Membrane Evaporation System

The TIMES process involves the pre-treatment of the urine, with either ozone or ultra-violet light and sulphuric acid. The urine is then heated and sent through hollow fibre membranes in a low pressure chamber, which promotes evaporation through the membrane. The now clean water vapour is condensed [5, 14]. As with VCD the brine could potentially be used as a fertilizer but the presence of sodium chloride and pharmaceuticals would necessitate further treatment.

With proper heat integration the energy requirements for the process can be well controlled. The main challenges come with the selection of the proper membrane, achieving the desired separation and controlling fouling [5].

2.4.1.3 Air Evaporation System

When treating urine or any salt solution, one of the problems is the resulting brine, which then has to be disposed or sent for further treatment. In an air evaporation system (AES), the urine is pre-treated to prevent hydrolysis and sent to a wick evaporator. Hot air is then used to evaporate the water in the urine and leave behind a solid, thereby negating the problem of brine disposal or treatment [5].

This process results in a near 100% removal of the water in the urine and an easily manageable solid. The challenges with the process include: urine pre-treatment to ensure only water and no other volatiles, such as ammonia, evaporate; removal of sodium chloride from the other salts; and the large amounts of energy required for heating the air, with difficult heat and water recovery options.

2.4.1.4 Lyophilisation/Freeze-thaw

One method to concentrate the nutrients in urine, which is beneficial to transport costs, is lyophilisation. The urine is frozen and the water is allowed to sublimate at a slightly elevated temperature.

Although the process can concentrate about 80% of the nutrients in 25% of the original volume, the energy requirements are prohibitively large, especially in hot climates, and there is a possibility of some nitrogen loss through ammonia evaporation, if the urine is not pre-treated [5].

2.4.1.5 Multi-stage Flash Evaporation

Multi-stage flash is a process where the liquid, in this case urine, is evaporated in chambers with successively lower pressure. To achieve this, urine is heated to the boiling point in the first stage. The urine and resulting steam at the boiling point enter the second chamber which is at a lower pressure. The steam from the first effect is condensed in the second stage, releasing latent heat which is used for further evaporation. Therefore the liquid requires a lower temperature to effect evaporation. This procedure continues for the required number of stages and the remaining brine is pumped out after the last stage [5].

Based on the usage of this process in water desalination it is expected that only around 15% of the urine entering the system would be converted to water, although this has yet to be investigated [15]. The process is reasonably energy efficient, using about 90 $MJ.m^{-3}$of water produced [5]. The main energy loss comes from the exit condensate. Besides the energy required, some of the volatiles in the urine, such as ammonia, may be lost if the urine is not pre-treated properly.

2.4.1.6 Solar Evaporation

Solar humidification-dehumidification is a process currently used to desalinate seawater by successive heating, evaporating, and condensing of the humid air. The process takes place in a solar still, with basic units having the solar heat section and condensation section together, and more advanced units separating the two. If designed properly, significant heat can be recovered

from the condensation step and returned to the heating chamber. This process mimics the natural water cycle over a much shorter period [16–18].

There have been some studies using solar energy to recover nutrients form urine [16, 18] but the problems in all the systems include difficulty in efficiently capturing and storing the solar energy. This inefficiency means that the system would have a relatively high capital cost to be large enough to achieve sufficient flow to process urine from a significant number of homes. The other problem is that any solar process would be inherently tied to areas with a suitable climate and plenty of direct sunlight, ruling out many countries and geographies.

2.4.1.7 Passarell Process

The Passarell process is a new technology used to desalinate seawater [20]. The process combines accelerated distillation and advanced vapour compression to produce potable water. The process allows for high-energy integration and recovery, and pilot plants show that this method is currently the most cost effective, industrially viable desalination process, as seen in the charts from Figure 3. Figure 4 shows the scheme of the process. The sea water is pre-heated and sent to the evaporator where the evaporation is achieved by low pressure rather than high heat. This low pressure is induced by the compression and subsequent condensation of the water vapour. The heat from the condensed liquid is recovered by heat interchange with the sea water feed [19, 20].

This technology has not been tested with urine but problems that may arise include ammonia loss through evaporation, unless the urine is pre-treated, and the need of large amounts of electricity to power the compressor.

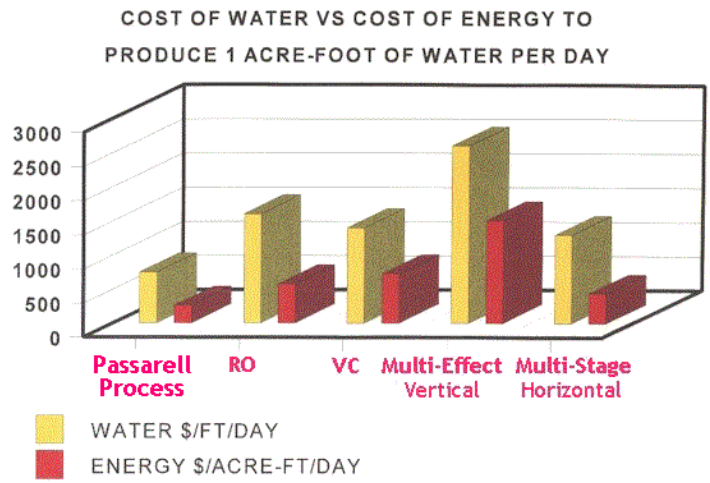

Figure 3: Relative Comparison of Desalination Costs and energy requirements [20]

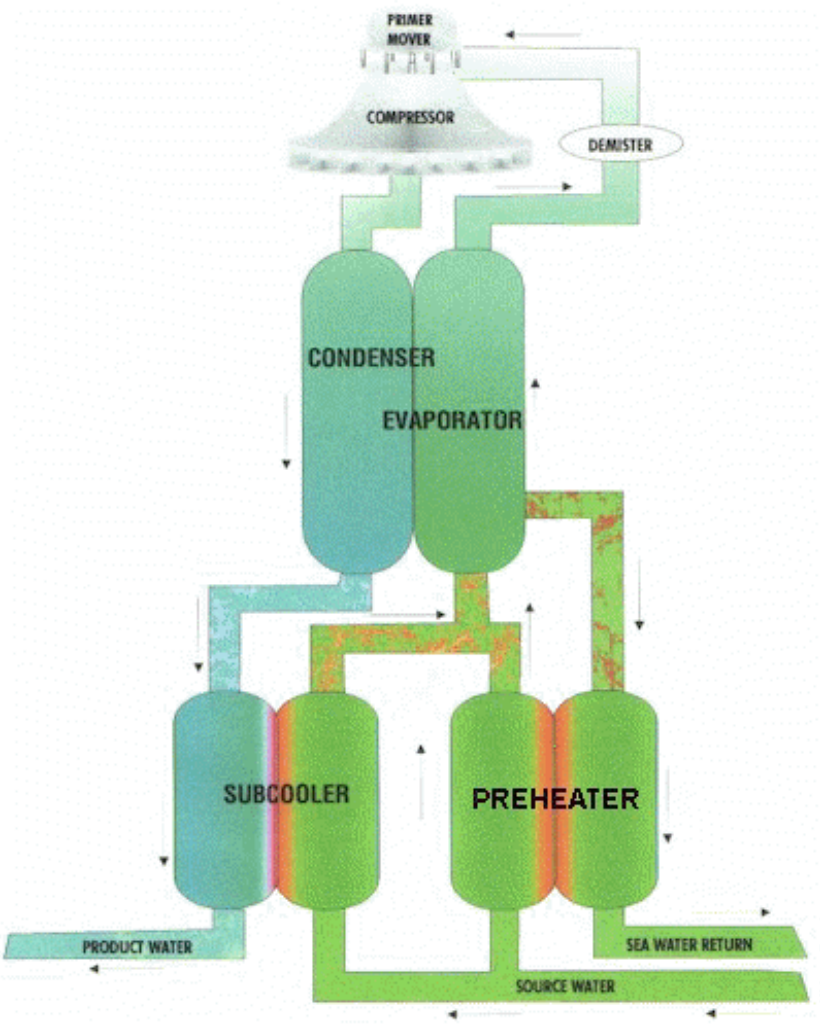

Figure 4: Passarell Process [19]

2.4.2 Membrane Filtration

2.4.2.1 Micro and Ultrafiltration

Micro and ultrafiltration, used in waste water treatment, use membranes with pore sizes ranging from 0.1 to 10 μm and 0.001 to 0.1 μm respectively [21]. Microfiltration is able to remove large particles, suspended solids, all bacteria and many viruses but for complete virus removal ultrafiltration is required [21].

These membranes have been used as pre-treatment steps in some experiments dealing with urine but they were not the focus of the experiments and no details of their effectiveness have been found [5, 22]. It is expected that they will perform similarly well with urine treatment, in removing bacterial and viral contaminants, as they have with wastewater treatment. The only concern is fouling potential, which would have to be investigated [5].

2.4.2.2 Nanofiltration

Nanofiltration (NF) membranes are frequently used in wastewater filtration to remove contaminants, as well as in desalination for salt removal. The membranes have pore sizes around 1nm, which enable the rejection of dissolved molecules while allowing small ions and un-charged molecules to pass through. The pressures used in nanofiltration are far lower than reverse osmosis, and therefore the electricity costs are lower. Nanofiltration can be used for polyvalent, divalent and, depending on the membrane, monovalent ion removal [22].

The problem with using nanofiltration to achieve this separation is that with completely or partially hydrolysed urine a significant amount of ammonia passes through the membrane, thus losing nitrogen unless further recovery is attempted [22].

2.4.2.3 Forward Osmosis

Forward osmosis is the process whereby the solvent diffuses through a semi-permeable membrane from a volume of low solute concentration, the feed, to a volume of high solute concentration, the draw solution, until the solute concentrations on either side are equal. This process requires no added energy and is driven solely by the concentration difference, i.e. the osmotic pressure difference. Forward osmosis has been studied extensively for use in sea water desalination [23] and to some extent in urine treatment [24, 25]. The process produces a concentrated solution, which must be treated to produce potable water.

A draw solution which can be separated from water with minimal energy input, such as an ammonium carbonate solution, is important as this will be the major energy requirement. An ammonium carbonate draw solution, used in previous urine and sea water experiments [26], requires a low heat input to separate the ammonia and carbon dioxide from the water. The only possible problems when treating urine would be low water flux through the membrane.

2.4.2.4 Reverse Osmosis

During reverse osmosis, the transfer of water is against the osmotic pressure difference across the membrane. This transfer is induced by applying a hydrostatic pressure larger than the osmotic pressure on the highly concentrated solution side of the membrane [5]. Reverse osmosis has successfully been used to desalinate sea water industrially and in various laboratory tests to treat urine [24, 25, 27].

The main problem is that the process requires large pressures, and thus high energy requirements to achieve the necessary water fluxes across the membranes. Other problems are the poor micro-pollutant retention; and the high sodium chloride retention and scaling, both of which can only be controlled through pH control of the urine, requiring chemical addition.

2.4.2.5 Electrodialysis

In electrodialysis, as seen in Figure 5, a current is applied across an electrodialysis stack consisting of alternating anion and cation ion-exchange membranes between two electrodes. The anion and cation ion-exchange membranes allow passage to only negatively and positively charged ions respectively. The ions move toward the oppositely charged electrode, passing through an ion-exchange membrane of opposite charge, but are stopped by the next membrane of the same charge. This movement of ions dilutes the concentrated feed stream while producing a concentrated salt solution.

The process is used in sea water and various other brine desalination processes, and has been tested on urine at a laboratory scale. Electrodialysis can achieve high product purity but works most economically on highly concentrated solutions and most effectively on solutions containing low molecular weight ionic components [28].

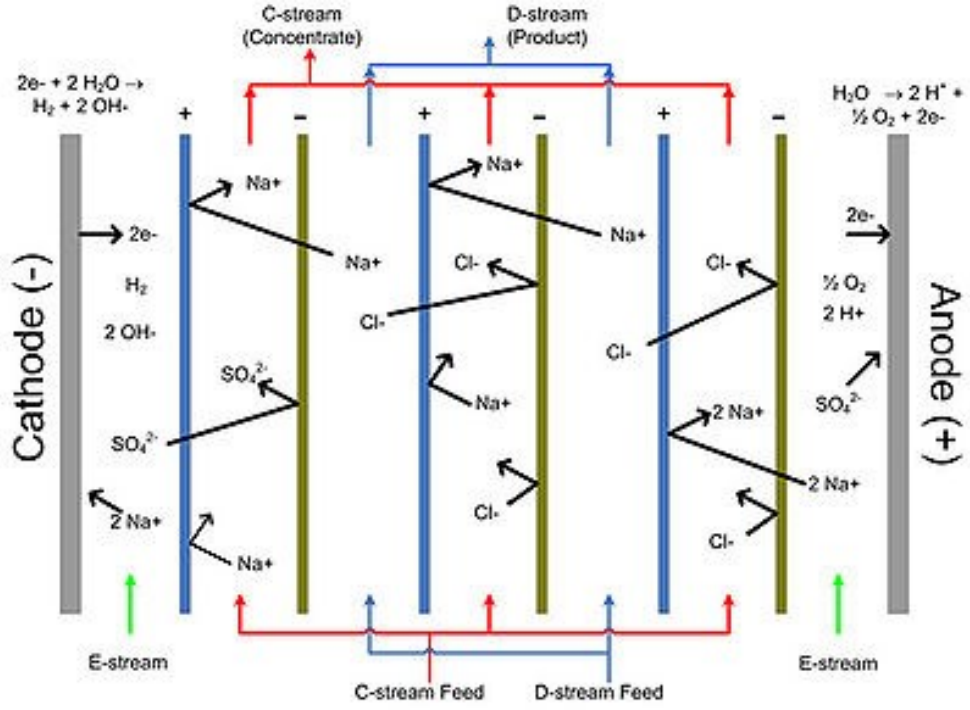

Figure 5: Electrodialysis Stack [29]

2.4.2.6 Osmotic and Membrane Distillation

Osmotic and membrane distillation are two very similar membrane separation processes. Both these processes use hydrophobic membranes with pore sizes between 0.1 and 0.5 μm and achieve separation by phase change. The feed and permeate solutions flow over the membrane but due to the membrane being hydrophobic the water cannot pass through in liquid form, except if a high enough pressure is exerted. The water from the feed side then evaporates and passes through the pores to the permeate solution. The driving force for the transfer in osmotic distillation is a difference in water activity, caused by the difference in solute concentration between the feed and draw solutions at the pores. In membrane distillation there is an added driving force of partial pressure difference induced by heating the feed [30–32].

Both these processes have been used in water treatment and desalination [30], and have been tested with urine [24, 25]. The problems with these processes are their very low fluxes and the requirement for heating in the case of membrane distillation. The other problem is that there may be fouling issues when used with urine.

2.4.2.7 Nanotube Membranes

At a very early stage of development, nanotube membranes are membranes made of an array of nanotubes orientated perpendicular to an impermeable film [33, 34]. The nanotube membrane can be used to desalinate sea water and allows for much higher fluxes than other membranes achieving comparable separation [33]. Theses membranes operate with a similar principle to porous membranes but the paths the permeate travels offers very little resistance and thus fluxes can be much higher. They have not been tested with urine and the technology must still be developed further before this can be done.

2.4.2.8 Biomimetic Membranes

Biomimetic membranes attempt to mimic bio-membranes already present in living organisms. These membranes are very selective about which chemicals may pass through, and can be highly efficient [35, 36]. Biomimetic membranes are still in development and are constantly improving.

Some experiments have been conducted in sea water desalination and the membranes have proven effective [36]. No tests have yet been conducted on urine.

2.4.3 Nitrogen and Ammonia Recovery

2.4.3.1 Ammonia Stripping

Stripping is common in the chemical industry as a process to recover a component from a liquid by mixing with a vapour and transferring the component to the vapour phase. The stripping of ammonia from urine has been reported in various papers and can achieve around 95% ammonia removal from urine [5]. The problem is that the liquid product, 10% ammonia solution, is unstable at atmospheric pressure [5].

2.4.3.2 Anammox Process

The Anammox, or anaerobic ammonium oxidation, process is a biological process which converts ammonium, nitrites and nitrates to nitrogen gas under anaerobic conditions [5]. This process has been tested extensively to treat digester supernatant but few tests on urine have been done. The aim of this process is mainly to eliminate nitrogen from urine and thereby lessen possible detrimental effects the nutrients in urine would have on the ecosystem [5].

The problem is that further processing is required to convert nitrogen to fertiliser and therefore it would be more beneficial to produce a product containing a Nitrogen/Phosphorous/Potassium (NPK) mixture.

2.4.3.3 Acidification

In some cases, it may be necessary to ensure that urea in fresh urine does not hydrolyse. The reasons for this are that urea may be the favoured compound for further reactions or processes and is easier to collect, for fertiliser production, than ammonia. To achieve this, the urine can be acidified by adding a strong acid, such as sulphuric acid, to ensure the pH stays below 4, the point where the urine begins hydrolysing. The low pH will also cause deactivation of many pathogens and, if low enough, can degrade pharmaceuticals [5].

It is important that the urine is acidified before significant hydrolysis takes place as neutralising hydrolysed urine requires approximately four times more acid due to the buffer effect of hydrolysed urine, which would drive up costs [5]. Besides the costs and added danger involved with purchasing and using the acid, early acidification is strictly required as the urea will begin to hydrolyse as it is transported through the pipelines or stored, before reaching the treatment work plant [11].

2.4.3.4 Nitrification

Nitrification has been tested extensively for the treatment of high strength industrial waste water and animal waste slurries, and has been found to be effective [5]. A few studies have been done on the treatment of urine by nitrification [37, 38]. To nitrify urine, oxygen is introduced and reacts with the urine in a moving bed biological reactor (MBBR) where the ammonium is converted to ammonium nitrate [5].

Only the MBBR has been found to produce ammonium nitrate, other reactors would produce ammonium nitrite, a less preferable chemical for fertilisers. The resulting solution is stable, giving off none of the odour typical of stored urine. The main problem with this method is that only half the nitrogen present in the urine can be converted to ammonium nitrate as the nitrification will stop when the pH becomes too low [5].

2.4.3.5 Struvite Precipitation

Magnesium ammonium phosphate ($MgNH_4PO_4.6H_2O$), also known as struvite, contains phosphate and ammonium, two nutrients in urine, and can be used as a slow release fertiliser [5]. To form struvite, magnesium is added to hydrolysed stored urine in the form of magnesium oxide (MgO), magnesium hydroxide ($Mg(OH)_2$), magnesium chloride ($MgCl_2$), or bittern, the magnesium-rich brine formed as a by-product of table-salt production [5].

The problem with using struvite precipitation in urine is that much of the nitrogen contained in the urine is left unrecovered and the urine would have to undergo further treatment to remove this [5].

2.4.3.6 IBDU Precipitation

Isobutylaldehyde-diurea (IBDU) is a commercially available, slow release fertiliser. IBDU can be made from urea by adding isobutylaldehyde (IBD). The urea forms a complex with the IBD and precipitates out of solution. This method could be used to form IBDU from urine but will leave a large fraction of urea unconverted as it requires highly concentrated urea to be effective. This would mean most of the water contained in the urine would have to be extracted beforehand. The cost of concentrating the urine, and purchasing IBD means that this process would be more expensive than other options [5].

2.4.4 Other

2.4.4.1 Urine Storage

The main aim of urine storage for long periods would be to disinfect the urine, deactivating any pathogens. The disinfection is achieved due to the rise in pH during urea hydrolysis resulting in the production of ammonia, which is a biocide thereby deactivating pathogens. The degree of disinfection is a function of mainly time, temperature and pH. The disinfection mechanism is the disruption of the cell metabolism by causing a rise in internal cell pH. This rise in pH is due to the travel of various components of urine, especially NH_3, into the cell. This is facilitated by the increase in cell membrane permeability due to elevated temperature. The simplest and cheapest way to implement this disinfection method would be the source separation and storage of the urine on site. The now safe urine could then be used directly for fertiliser, although the effect of storage on pharmaceuticals must still be investigated; processed on-site; or transported via tankers or pipelines for further processing in a treatment plant.

The most important parameters to take into account when using this method would be, in order of greatest effect: temperature, pH and time. The deactivation rate increases with an increasing temperature, with no deactivation below 4°C recorded with storage times below 6 months; and increases at pH extremes due to acid dosage or urea hydrolysis. A suitable temperature of 20°C can be easily achieved by underground storage, with no need for temperature control in warmer climates [5].

The main problem with this disinfection method is the evaporation of ammonia if the tank is not properly sealed. The precipitation could be controlled, while still achieving disinfection, with acid dosing [5].

2.4.4.2 Electrochemical Oxidation of Urea

By using a nickel catalyst, the urea in urine can be electrochemically oxidised to form hydrogen, nitrogen and carbon dioxide gases [5, 39]. The hydrogen can then be captured for use as fuel.

This process is still experimental but results have been positive [39] and the collection of hydrogen could prove economically viable. The problems are that all other nutrients are wasted, and the hydrogen may not be easy to collect and use in the areas the treatment facility would be located.

2.4.4.3 Ion-Exchange

Ion-exchange has been used in waste water treatment to remove unwanted salts. A complex, usually an ion-exchange resin or zeolite, is added to the solution and the desired ions are exchanged by attaching to the surface of the complex.

Ion-exchange has been tested with urine, and clinoptilolite, a naturally occurring zeolite with high affinity for ammonium, has been found to be quite effective [5]. The problem with using ion-exchange alone is that only the nitrogen would be recovered.

2.4.4.4 Ozonation and Advanced Oxidation

Ozonation and advanced oxidation can be used to remove micro-pollutants in waste water and has been used experimentally to treat urine [3, 5]. The micro-pollutants are oxidised using chlorine, chlorine dioxide, ozone, or hydroxide radicals. Using this treatment, micro-pollutants can be mostly, or completely, removed [5]. The problem is that chemicals must continually be added to achieve this.

2.4.4.5 Ultraviolet Treatment

Ultraviolet (UV) treatment is used in waste water treatment to deactivate pathogens. The treatment works by exposing the water to ultraviolet radiation which alters the genetic structure of bacteria, viruses and other pathogens, rendering them harmless and incapable of reproduction [40, 41]. This treatment results in no chlorine or ozone disinfection by-products, no chemical residues, and is low risk [41].

Although this process is frequently used to treat wastewater, it has not been tested on pure urine. The main problems with UV treatment include frequent maintenance and replacement of the UV lamps, and the need for highly treated feed to ensure no solids are present which could shield the micro-organisms from the radiation.

2.5 Process Selection

2.5.1 Treatment Process Analysis

Table 2 and Table 3 are a qualitative analysis of the information collected during a review of the literature available on the various processes. Table 2 shows all the processing methods considered while Table 3 shows the membrane processes in more detail. These tables and the design constraints, provided by the RTTC, will help in the process selection.

Table 2 took a broad overview and, through a weighting systems, indicates which processes were the most promising to compare in further detail in Table 3. Table 2 covers the following sections:

- Disinfection
    - ability to exclude or inactivate pathogens in urine
- Water Recovery
    - Indication of the fraction of water recovered, which links to volume reduction but focuses on the recovery of water in addition to reducing effluent or concentrate volume.
- Stabilisation
    - Ability to prevent or inhibit urea hydrolysis
- P, K Recovery
    - Indication of the recovery of phosphorous and potassium in a usable form
- N Recovery
    - Indication of the recovery of nitrogen in a usable form
- Micro-pollutant/NPK Separation
    - Ability to separate NPK form micro-pollutants, such as pharmaceuticals and compounds naturally excreted in urine, such as creatinine.
- Micro-pollutant Elimination
    - Ability to breakdown micro-pollutants.

Once the most promising technologies had been identified, a more in depth comparison was undertaken, summarised in Table 3. This table follows a similar format to Table 2 with a weighting system geared toward finding technologies that will achieve a split between the NPK and the unwanted components, namely pathogens, micro-pollutants and sodium chloride. In addition, some important design considerations are represented which helped to provide a better feel for which technologies showed the most promise.

Group	Process	Disinfection	Water Recovery	Stabilisation	P,K Recovery	N Recovery	Micro-pollutant /NPK Separation	Micro-pollutant Elimination	References
Evaporation	VCD	2	3	2	3	3	1	1	[5, 12, 42]
	TIMES	2	3	2	3	3	1	1	[5, 14]
	AES	2	3	2	3	3	1	1	[5]
	Multi-stage Flash	3	3	2	3	3	1	1	[5]
	Freeze-thaw	2	2	1	3	3	1	1	[5]
	Solar Evaporation	3	2	2	3	3	1	1	[16, 18, 43, 44]
	Passarell Process	3	2	2	3	3	1	1	[19, 20]
Membrane	Membrane Distillation	4	2	1	4	4	4	1	[24, 25, 30, 31]
	Reverse Osmosis	4	3	1	3	3	4	1	[5, 24, 25]
	Forward Osmosis	4	3	1	3	3	4	1	[23–26]
	Electrodialysis	3	2	2	2	2	2	1	[5, 28, 39]
	Micro/Ultra Filtration	2	1	3	1	1	1	1	[5, 21]
	Nanofiltration	3	1	2	1	1	3	1	[5, 22]
Nitrogen/ Ammonia recovery	Ammonia Stripping	1	2	1	1	3	3	1	[5]
	Anammox Process	2	1	3	1	1	2	1	[5]
	Acidification	2	1	3	1	1	1	1	[5]
	Nitrification	2	1	3	1	1	1	1	[5, 37, 38]
	Struvite	1	3	1	3	3	3	1	[5]
	IBDU Precipitation	1	2	1	1	3	2	1	[5]
Other	Ion-Exchange	1	2	1	1	3	2	1	[5]
	Advanced Oxidation	2	1	2	1	1	1	4	[5]
	UV Treatment	4	1	3	1	1	1	4	[40, 41]
	Storage	2	1	1	1	1	1	2	[5]

Table 2: Processing Methods

No effect or Not Feasible / Low	Some Effect / Medium	Strong Effect / High	Most Effect / Very High
1	2	3	4

No effect or Not Feasible / Low	Some Effect / Medium	Strong Effect / High	Most Effect / Very High
1	2	3	4

Function	Unit operation					
	Membrane/Osmotic Distillation	Reverse Osmosis	Forward Osmosis	Microfiltration	Ultrafiltration	Nanofiltration
Pathogen Removal	4	4	4	2	2	3
Enzyme/Microbe Rejection	1	1	1	3	3	1
P, K Retention	4	3	3	1	2	3
Urea Retention	4	3	3	1	1	2
Micropollutant/P, K Separation	1	1	1	1	2	1
Micropollutant and Pharmaceuticals Rejection	4	4	4	1	1	4
Requirement for pre-treatment	2	4	3	1	1	3
Flux (actual value in brackets) [$l/m^2.h$]	1	3 (20)	2 (12)	4	4	4 (100)
Available Literature	2	2	3	1	1	2
Extent Tested on Urine	2	2	2	2	2	3
Energy Required [kWh/m^3 water]	2	1 (24)	2 (6)	4 (0.3)	3	2 (6)
Primary energy source	Heat	Pressure	Heat	Pressure	Pressure	Pressure
Cost	2	3	2	1	2	2
Simplicity of System	3	4	2	1	2	3
Requirement for Chemical Addition	1	2	1	1	1	1
Nutrient Product Stream Usability	3	3	3	1	1	2
Product Water Stream Quality	4	3	3	1	1	2
References	[24, 25, 30, 31]	[5, 24, 25]	[23–26]	[5, 21]	[5, 21]	[5, 22]

Table 3: Membrane Processes

2.5.2 Design Constraints

The main design constraints for the urine treatment section of the system, taken from the design constraints from the RTTC requirements, include:

2.5.2.1 Energy requirements

Energy is often one of the largest costs of any industrial plant and so every effort should be made to use energy optimally. The RTTC toilet systems have to be designed to be self-sufficient and so minimising energy usage is vital. One of the best ways to achieve this is to use readily available energy sources. The main energy source in the proposed system could be low grade heat, for example from the combustion of faeces. Thus any process that could utilise this energy, and does not require high calorific energy usage, high electricity usage or high pressures, is preferable.

According to this constraint, the following processes are unfavourable:

- Electrodialysis → high electricity
- Lyophilisation →high electricity
- Vapour Compression Distillation → high electricity
- Passarell Process → high calorific/electricity
- Multi-stage Flash → high electricity
- Air Evaporation System → high calorific energy
- Reverse Osmosis → high pressure

2.5.2.2 Minimal Consumables

Many processes require the addition of chemicals to function correctly. These chemicals may be reagents, catalysts or some type of inhibitor. Regardless of the purpose, the chemicals would have to be bought and dosed correctly, which would require skilled technicians to ensure correct operation. Both the purchasing and monitoring would increase operating costs. Minimising consumables is therefore a good way of lowering operating costs.

According to this constraint, the following processes are unfavourable:

- Struvite precipitation → chemical addition
- IBDU precipitation → IBD addition
- Ozonation and Advanced oxidation → ozone addition
- Acidification → acid addition
- Ion Exchange → chemical addition
- Thermoelectric Integrated Membrane Evaporation → chemical addition
- Electrochemical oxidation and ammonia stripping → catalyst addition

2.5.2.3 Robust and Modular System

The treatment system will be installed in remote areas and would function largely autonomously and thus should be low maintenance and easily repairable. The units should be easy to replace, and be robust enough to handle varying feeds and possibly regular start-ups and shut-downs. Thus, biological systems, which are feed specific and do not handle large flow changes, and systems which are technologically complex or new, should be avoided.

According to this constraint, the following processes are unfavourable:

- Anammox Process → biological system
- Biological reduction of nitrates → biological system
- Thermoelectric Integrated Membrane Evaporation → technologically complex
- Ultra-violet treatment → technologically complex
- Nanotube membranes → new technology
- Biomimetic membranes → new technology

2.5.2.4 Universal Applicability

The last constraint is that the system must be able to function in varying climates and geographies as the system is meant for global use. This means that relying on a specific resource from an area, such as plentiful direct sunlight, is undesirable.

According to this constraint, the following processes are unfavourable:

- Solar evaporation

2.5.3 Urine Treatment Process Selection

Once various processes are disregarded due to not adhering to the design constraints of the RTTC, the result is that a combination of membrane processes, excluding electrodialysis, nanotube and biomimetic membranes, seems to be a promising solution. Table 3, in section 2.5.1, shows a summary of the information gathered regarding the various viable membrane processes. To decide on which processes to select, the primary aims of the system must be considered. These are: pathogen removal; separation of phosphorous, potassium and nitrogen sources from sodium chloride; minimal energy usage; and the production of water suitable for irrigation. Using Table 3 and the stated aims, various membrane process configurations are possible with a combination of microfiltration, nanofiltration and forward osmosis seeming to be very promising. The following configurations were considered:

(1) Recovery of water; secondary separation of concentrate into combustibles and small ions

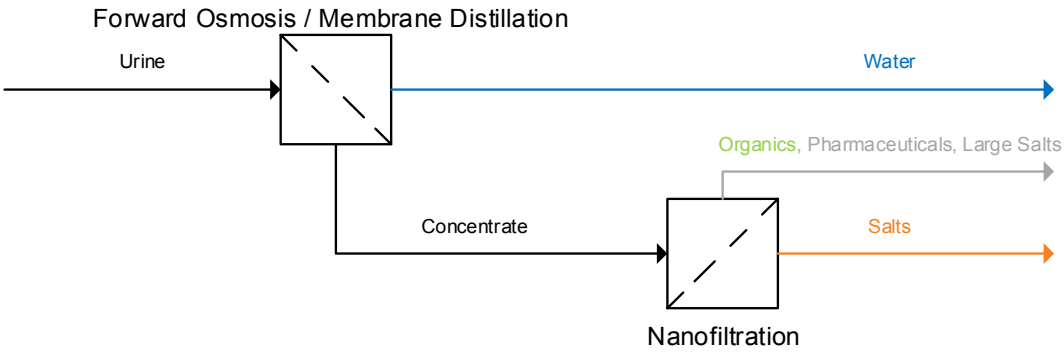

Figure 6: Process Flow Diagram 1

The first approach, shown in Figure 6, would concentrate the urine by separating the water from the urine, possibly using a combination of forward osmosis and membrane distillation. The aim behind this was to concentrate all the nutrients into one stream making fertiliser production easier. The problem with this process flow would be the potential of significant fouling at the first stage, as all the organics and salts would still be present. Lower flux across the membrane due to fouling would lead to lower recovery rates and higher energy requirements.

(2) Primary separation of waste components, secondary separation of water and brine

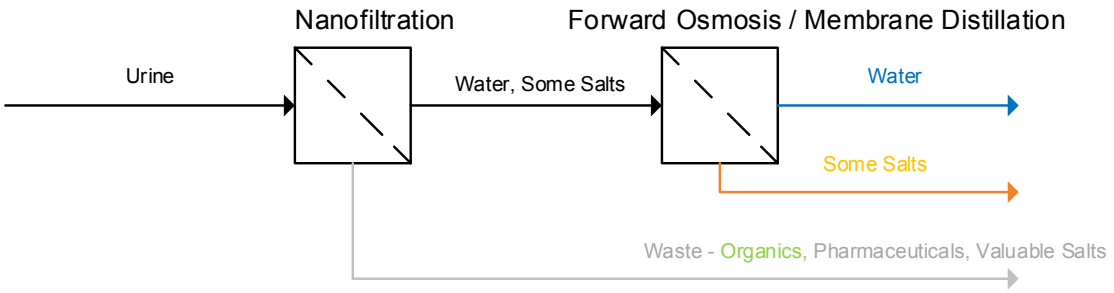

Figure 7: Process Flow Diagram 2

The second approach, shown in Figure 7, would decrease the fouling potential at the first stage by using a nanofiltration membrane, which is more resistant to fouling. The benefits of the scheme would be the concentration of the desired salts for fertiliser production in one stream. The problem with the second process would come with the separation after the nanofiltration stage, between the waste components and the salts with agricultural value in the concentrate.

(3) Removal of potential fouling components, secondary separation of waste components, tertiary separation of water and brine

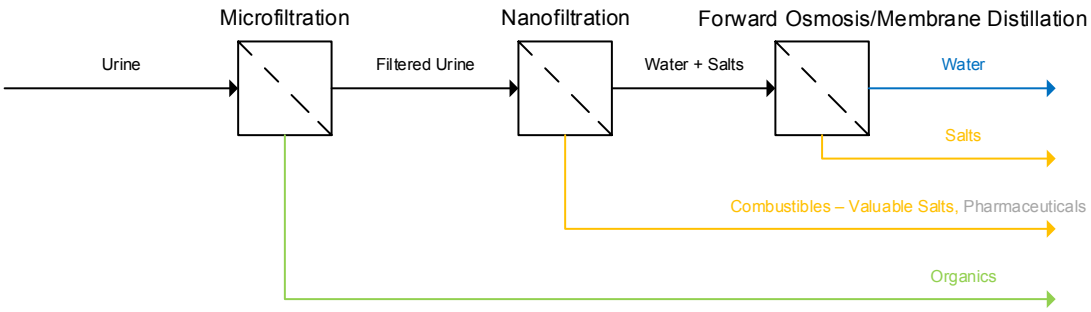

Figure 8: Process Flow Diagram 3

The third approach, shown in Figure 8, uses three different membrane units in series. The first stage – microfiltration (MF) or loose ultrafiltration (UF) - acts as a screening step to remove organic components which could cause fouling downstream.

According to literature [22], nanofiltration is capable of rejecting at least: 80 % of micro-pollutants; 95 % of the phosphate; 70 % of the potassium; and 65 % of the ammonia. Urea, which made up the bulk of the total nitrogen, has a rejection rate of around 10%. The concentrate from the NF stage, containing the nutrients phosphorous and potassium, will be sent for further processing, for instance the combustion unit dealing with solid waste, to deactivate the micro-pollutants before being used to make fertiliser. A final forward osmosis (FO) stage (or combination of forward osmosis and membrane distillation) separates the remaining salts from water.

The whole process should be able to accept the various feed solutions that might flow into the urine processing unit within the toilet - including fresh and stored urine, as well as urine contaminated with faecal matter.

2.6 Nanofiltration

As the remainder of the study focuses on nanofiltration, as reasoned in Section 2.7, further literature on nanofiltration is detailed in this section.

2.6.1 Definition

Nanofiltration is a membrane operation which separates a feed stream into a permeate stream, containing material which has passed through the membrane, and a retentate stream, containing the components rejected by the membrane. By using a membrane operation, a solvent-solute solution or solid-liquid suspension can be concentrated or purified and a solute-solute mixture can be fractionated [46].

Some of the advantages of using membrane operations to effect a separation are as follows [46]:

- Separation can take place at ambient, or near ambient temperature, without a net phase change. This saves energy compared with separation processes which require phase change to occur, requiring a heat input.
- Membrane operations are well suited to continuous operation, and need only be washed if fouling layers form and lower flux below acceptable levels.
- No chemicals are required to effect the separation. This means that products will not contain additional pollutants or contaminants and consumable costs for operation decrease.

2.6.2 Separation Mechanism

Nanofiltration membrane operations use a combination of three different separation mechanisms to affect the separation. The mechanisms involved are as follows: size exclusion or sieve-effect, dominant when the molecular weight of the solute is much greater than the Molecular Weight Cut-off (MWCO), which is a measure of the molecular weight of a compound above which it would be 90 % rejected by the membrane; solution-diffusion and electrostatic interaction [47], which is unique to nanofiltration and dominant when the molecular weight of the solute is much lower than the MWCO [48].

The sieve-effect excludes compounds based on their size in relation to the pore size of the membrane and the driving force for the separation is an induced pressure difference. This is usually characterised by the MWCO of the membrane. However, this is by no means a definitive measure of rejection potential as many compound rejections do not follow this trend [47]. A better measure of this mechanism, although harder to quantify, would be pore size distribution or effective number of pores, and membrane porosity [47].

The operation of the sieve-effect is also influenced by: the hydrophobicity of the membrane and molecules, with MWCO being overestimated for hydrophilic molecules and underestimated for hydrophobic molecules of the same size [49]; and the surface morphology of the membrane, with discrete small-pore structure giving a better membrane selectivity [49].

The solution-diffusion mechanism achieves separation based on the solubility and diffusivity of the compounds and the permeability of the membrane. Transport takes place in the free volume of the membrane between the macro-molecular chains of the material [46] and is induced by the concentration difference between the permeate and retentate. Operating temperature plays a role in solution-diffusion, with an increase in temperature causing an increase in convective flux, diffusivity of molecules and water flux, thereby reducing retention [49].

The significant influence of electrostatic interactions, between molecules and between molecules and membrane, on the rejection performance is unique to nanofiltration and can heavily influence the rejection of ions. Most nanofiltration membranes have a negative charge, due to the sulphonic or carboxylic acid groups in the membrane deprotonating at neutral pH. This is why negative molecules will be better rejected than neutral and positive molecules of comparative size [49].

The relationship between membrane surface charge and pH also means that the charge will change with pH, with an increase in pH leading to a larger negative charge and therefore an increase in rejection, of negatively charged molecules. In addition to the pH affecting the membrane charge, it may also influence the dissociation state, orientation and solubility of the solutes. By changing a solute dissociation state, the rejection of the solute can be changed. Lastly, the ionic strength of the solution influences rejection by increasing the relative pore size of the charged membrane pores which results in a rejection decrease, particularly of monovalent ions [49].

Based on the nanofiltration mechanisms and the pore size of the membranes, this process can be used to remove salts, hardness or minerals, pathogens, turbidity, disinfection by-product precursors, synthetic organic compounds, pesticides and other water contaminants [46]. Although not all contaminants can be removed using nanofiltration, it has the potential to remove a wider range of contaminants than many other treatment technologies [46].

2.6.3 Membrane Types

Synthetic nanofiltration membranes can be manufactured from a large number of materials but can be classed as either organic or inorganic.

Organic membranes are manufactured using polymers. Many types of polymers can be used to manufacture the membranes but, due to difficulties in processing, economic considerations and membrane durability, only a few are used in practice. The most widely used polymers are cellulose and its derivatives, due to their low cost and low absorption tendency [46]. These polymers make hydrophilic membranes which are used in all pressure driven membrane operations as well as haemodialysis and gas permeation [46].

Cellulose ester membranes, although sensitive to acid or alkaline hydrolysis, are relatively resistant to chlorine, temperature and biological degradation, making them popular in water treatment [46]. These membranes should transfer well to treating a highly saline solution such as urine.

Polyamide membranes, which are hydrophilic and more chemically, thermally and hydrolytically stable than cellulose membranes, are also used in water treatment, although these membranes are highly sensitive to oxidative degradation and cannot tolerate chlorine even in trace quantities [46].

Inorganic membranes generally have greater chemical, mechanical and thermal stability relative to organic membranes. The disadvantages to these membranes are their high cost and brittle nature. The main materials used in inorganic membrane manufacture are ceramics, including oxides, nitrides or carbides of various metals [46].

2.6.4 Fouling

An important factor during NF membrane operation is the reduction in permeate flow due to fouling. There are three causes of fouling in pressure driven membrane processes:

2.6.4.1 Cake Formation

Cake formation occurs when the material rejected by the membrane accumulate on the membrane surface. The resistance to permeation of this cake layer can be quite significant and will increase with decreasing particle size [46]. Cake fouling can be reduced by increasing the cross-flow velocity of the solution across the membrane, in an attempt to carry any caking material away, as well as by pre-treating the feed to remove fouling agents [46].

2.6.4.2 Precipitative Fouling

Precipitative fouling or scale formation occurs when the salt concentration near the membrane surface is higher than the salt solubility. The concentration of the salts increases either, because of the increase of the bulk concentration of the salts as a result of the removal of water from the solution, or because of concentration polarisation. The latter refers to the concentration gradient between the boundary layer near the membrane surface and the bulk of the feed solution, due to the selective permeation of ions through the membrane causing a build-up of the rejected ion species [46]. Controlling precipitative fouling usually involves using anti-caking agents, dosing the feed with acid to control anionic species concentration or pre-treating the feed to remove scale-forming materials [46].

2.6.4.3 Adsorptive Fouling

Adsorptive fouling occurs when materials are deposited inside the membrane pores. This fouling is especially prevalent with feed solutions containing organic materials, can have a much greater effect on flux than other fouling, and is usually very difficult to remove [46]. There are three main ways to reduce adsorptive fouling. First, negatively charged membranes with a high surface charge density, associated with membrane hydrophilicity, can be used. Second, solution pH can be increased, as lower pH tends to favour adsorption. Last, the membranes can be cleaned with a caustic and enzymatic chemical wash, which can re-dissolve the adsorbed organic compounds [46].

2.6.5 Nanofiltration of urine

As mentioned, published data regarding nanofiltration in conjunction with urine is quite scarce. Pronk et al. [22] dealt with the ability of nanofiltration membranes to remove pharmaceuticals found in urine and is a primary reference for this study. The use of nanofiltration to remove pharmaceuticals is quite common in the water treatment systems but has not been used before with pure urine. Pronk et al. posited that, by passing urine through a nanofiltration membrane, the permeate stream would be a nutrient rich and micro-pollutant free liquid suitable for use as an agricultural fertiliser.

Although the focus was on micro-pollutant removal, tests were done on the retention of various salts necessary for the permeate stream to be used as fertiliser. The researchers started with three different membranes with varying fresh water permeability and molecular weight cut-offs. The membranes were the NF 270 by Dow-Filmtec, the NF 30 by Microdyn-Nadir and the DS 5 by Osmonics. After testing the membranes, it was decided to focus on the NF 270 as this membrane gave the most desirable rejections profile for the involved nutrients. A detailed breakdown of the rejections by the NF 270 membrane, for both synthetic and fresh urine, can be found in Figure 9. Most importantly, the urea rejection is never higher than 20 %, the phosphate and sulphate are almost entirely rejected at all pH values, sodium and potassium have similar rejections usually around 60 % and the ammonia rejection varies widely with pH, between 5 and 65 %. Unfortunately, the paper does not go into detail regarding the pressures, water recoveries or corresponding water fluxes obtained, or how the rejections changed with these variables.

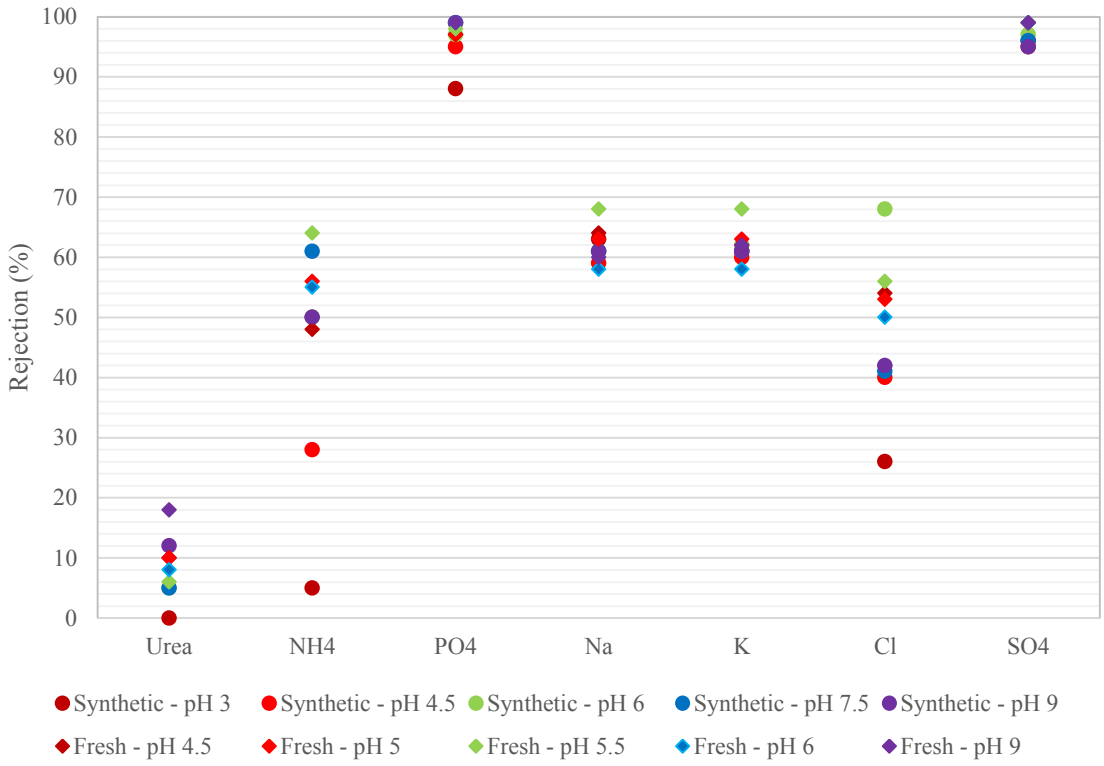

Figure 9: Fresh and synthetic urine rejections from Pronk et al. [22]

The majority of the remaining publications in this field were from a modelling perspective. These papers attempt to formulate a suitable model for mixed salt solutions, which reverse osmosis models cannot adequately represent due mainly to the electrostatic interactions that take place during nanofiltration. The most useful models would be those that represent a mixed salt solution with ions present in urine, such as sodium, chloride, phosphate, nitrates and potassium. Many of the papers, through experiments attempting to validate the proposed models, can provide information regarding rejection for various ions through a selection of membranes.

Four different research papers [50-53] have been published which contained experimental work concerning solutions containing more than three ions, some or all of which are present in urine. Three of them [50–52] detail effort into developing a model specifically for nanofiltration of mixed salt solutions containing 3 or more ions. The research uses the same experimental setup and conditions in each paper, with three different nanofiltration membranes and various salts to test the model. The membranes used were the ESNA 1, ESNA 1-LF and LES 90 membranes made by the Nitto Denko Corporation. These papers give the details of only some of the experimental conditions, namely: membrane area; mean pore radius; pure water permeability; feed concentrations and flux of the solutions. Some of the important variables left out include the solution pH and temperature, the cross-flow rate or tangential velocity, and the pressure applied.

Although some important variables are left out, the results are still useful in presenting the rejection for the membranes. Figure 10 shows results from the three papers, with marker colour indicating the various solution compositions and marker shape indicating membrane. The results indicate that the rejection of the various ions can vary by as much as 20 % just by changing the solution composition, as indicated by the sodium rejection rates. The rejection rates for all ions were quite high compared to the other papers, all being around 75 % and higher. The researchers were able to accurately model the transmission rates of the ions, with errors of between 10 and 20 % for the various ions modelled, including solutions containing multivalent ions. Still to be investigated is the effect permeation flux and pH have on the accuracy of the model.

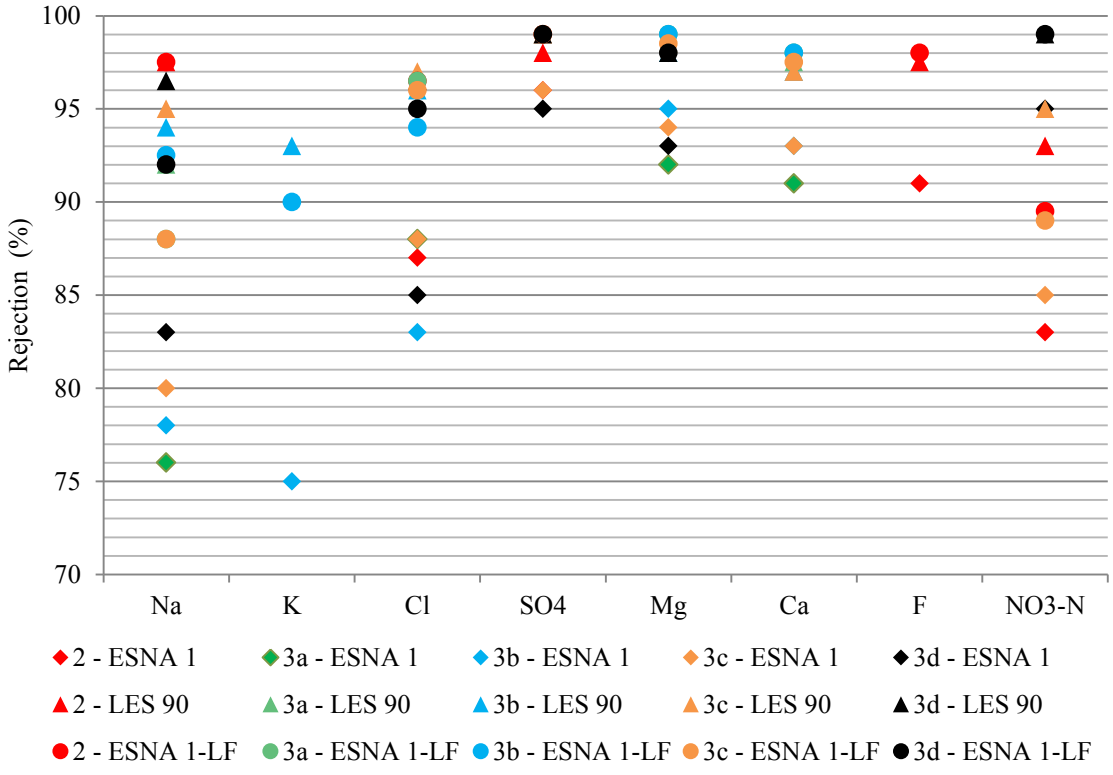

Figure 10: Salt solution rejections from Wang et al. [50–52]

Hayryen et al. [53] carried out a study concerning the concentration of mine water by nanofiltration and reverse osmosis. The research only used one nanofiltration membrane, as reverse osmosis was found to be better suited to their requirements, and details the pH, temperature, tangential velocity, cross-flow velocity, membrane area and flux but gives no information about the membrane pore size or MWCO and pure water permeability. The data from the studies [50-53] is summarised in Figure 11. The figure shows the rejections of several compounds including the distribution of the results around the mean value, from the all papers discussed.

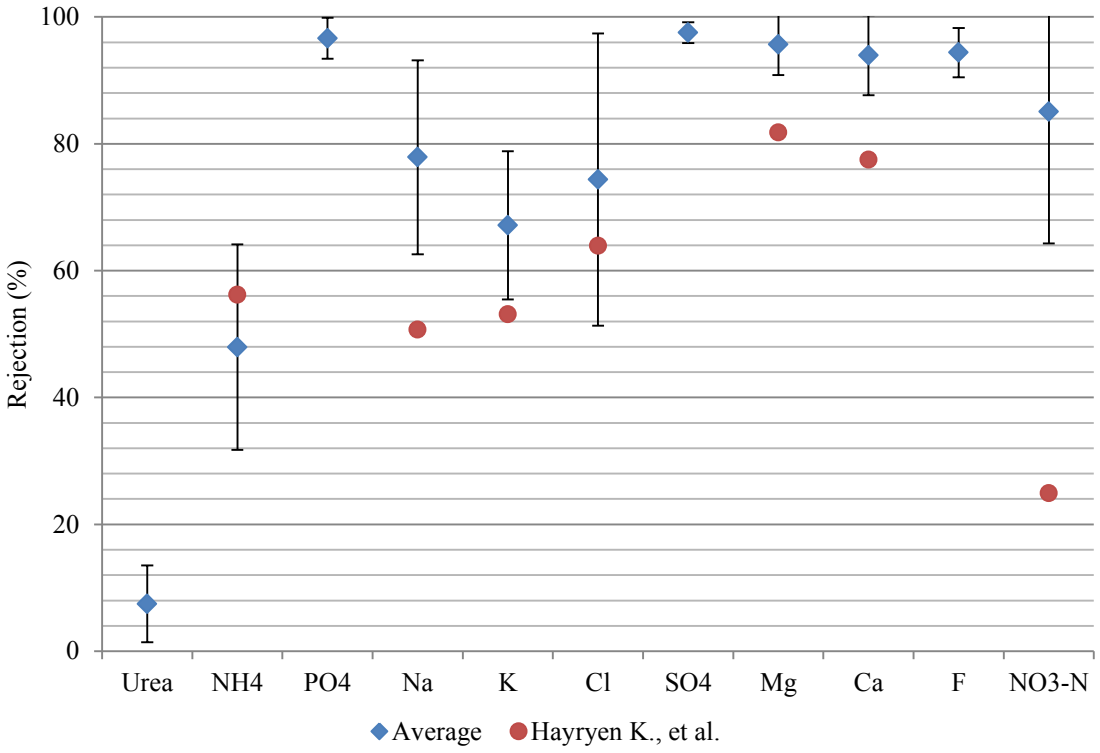

Figure 11: Summary of Experimental Results [22, 50–53]

In order for a nanofiltration step to be useful in the treatment system, there must be a separation between the unwanted compounds and the desired nutrients in the urine. From the literature, it can be seen that the rejection of many of the ions shown vary considerably. Through Figure 9, Figure 10 and Figure 11, it seems that a favourable set of conditions may exist where the possibility of waste and nutrient separation exists.

To achieve this, either: most of the sodium chloride in the urine must be retained, which is possible with rejection as high as 97%, while allowing nitrogen passage, which would be possible with urea rejection consistently below 20% for fresh urine; or, depending on the sodium chloride limits in fertiliser, there must be sufficient sodium chloride permeation through the membrane with retention of sufficient phosphate and nitrogen, from hydrolysed urea, to produce a retentate economically and agriculturally viable for processing into fertiliser.

As there are many variables affecting the rejection of ions, a model of the ion permeation would be useful resource to find the optimum conditions to achieve these goals.

2.7 Gap Analysis

The membrane system chosen in section 2.5.3 must be designed to (i) separate components as desired; (ii) achieve a sufficient level of throughput and (iii) not use excessive amounts of energy. The following design data are required to achieve this:

- Expected compositions of different urine feeds (fresh, aged, contaminated with faecal material)
    - Data on segregated fresh and aged urine is readily available [7] but the possibility of faecal contamination in the toilet must still be accounted for.
- Fouling and reduction in fouling after cleaning
    - No data was found in the literature on fouling for membrane processes used with a urine feed. An investigation into the use of a forward osmosis process for the desalination of sea water found that fouling rates were low, due to low-pressure operation, but greater fouling rates would be expected with a urine feed [26, 45].
- Recovery of water and rejection of solutes
    - Microfiltration is commonly used to treat waste water instead of granular media filtration combined with ozone treatment units [21]. Using microfiltration to remove particulates from urine is likely to be effective but no data has been found in the literature on rejection of organic particulates with a pure urine feed.
    - The use of nanofiltration with fresh and synthetic urine feeds has been investigated and showed potassium and phosphate rejections of 65 % and 95 % respectively, and various pharmaceutical compound rejections upwards of 80 % at a pH of 5 [22]. There were very few specifications given, regarding the operating conditions required to achieve this separation, and only fresh urine and synthetic fresh urine were tested.
    - Production of potable water from sea water [23, 26, 45] and waste water using forward osmosis is well documented, with water recovery of up to 70 %, salt rejections of 95 % and fluxes up to 25 $l/m^2.h$ [23], but the use of forward osmosis for urine treatment has not been widely studied. A few papers [24, 25] using forward osmosis with urine feed indicate that there are some promising results with rejection of urea upwards of 99 % when used in conjunction with membrane distillation.

- Flux through the membranes
    - The flux through the forward osmosis stage will be the rate limiting factor to the process.
    - According to one paper flux in the NF stage is expected to increase with pH but no further specifics were given [22].

The knowledge gaps in the use of nanofiltration for urine treatment are seen as the most pertinent missing information as achieving the required salt separation is a key factor in all three proposed configurations. Therefore, the remainder of this work focuses on nanofiltration, specifically investigating the salt rejections and water flux.

3 MATERIALS AND METHODS

As stated in section 2.6.1, the focus of the experimental section of the project was on nanofiltration. Through quantitative experimental analysis, the salt rejections', reported in section 2.6.5, were checked for repeatability. Then the results of the experiments were used to determine if it is possible to retain the majority of the valuable minerals, including potassium, phosphorous and nitrogen, while allowing permeation of sodium chloride in hydrolysed urine.

3.1 Research Design

The experiments used two different solutions to investigate the focus area of the salt split through nanofiltration. The solutions consist of: a synthetic solution developed by Udert et al. [11] which is seen as an accurate approximation of completely hydrolysed urine; and fully hydrolysed urine, which has been in storage for over 6 months. In Table 4 below the composition of the synthetic urine is detailed. In the case microfiltration was chosen as pre-treatment, it would have been ideal to pass the stored urine through a rigorously defined microfiltration system beforehand but time did not permit this and so fouling was expected to be higher than necessary.

Substance	Mass	Vol.	Conc.	Moles
	[g]	[ml]	[g/mol]	[mol]
Na_2SO_4 anhydrous	9.2		142.0	0.06
NaH_2PO_4 anhydrous	8.4		120.0	0.07
NaCl	14.4		58.4	0.25
KCl	16.8		74.6	0.23
NH4Ac	38.4		77.1	0.50
NH_4OH solution (25% NH_3)		52	22.3	2.33
NH_4HCO_3	85.6		79.1	1.08
H_2O Distilled		4000	18.0	222.04

Table 4: Udert et al. synthetic urine recipe [11]

List of chemical names:

Na_2SO_4 anhydrous	Anhydrous Sodium Sulphate
NaH_2PO_4 anhydrous	Anhydrous Sodium Dihydrogen Phosphate
NaCl	Sodium Chloride
KCl	Potassium Chloride
NH4Ac	Ammonium Acetate
NH_4OH solution (25% NH_3)	Ammonium Hydroxide Solution
NH_4HCO_3	Ammonium Bicarbonate
H_2O Distilled	Distilled Water

The membranes used were 3 different DOW-Filmtec membranes, the NF270, NF90 and XLE membranes, as these are easily obtainable and are in widespread commercial use. According to the membrane characteristics given by the supplier, their performance and quality are of acceptable levels. Another reason to use DOW-Filmtec membranes is that the NF 270 membrane was used in the work of by Pronk et al. [22], discussed in the literature review, which enabled a direct comparison of the results with the same membrane. The membranes have MWCOs ranging from 100 to 400, with XLE having the smallest and NF 270 the largest, and are made of polyamide. This type of membrane is frequently used in wastewater and brine treatment and, therefore, it may perform well in urine treatment operations.

3.2 Experimental Design

This section details the nanofiltration rig, experimental procedure and the analytical equipment.

3.2.1 Nanofiltration Equipment

Figure 12 on page 40 illustrates the high-pressure membrane-testing unit.

The high pressure cross-flow membrane laboratory set-up consisted of 3 cells in series, each containing a flat sheet membrane with a 38 mm diameter, held in place by a sintered steel disc. The equipment can reach pressures gradients across the membrane cells of up to 6000 kPa, equivalent to a transmembrane pressure (TMP) of 3000 kPa, and a maximum flow rate of around 3 l/min. The individual cell area was 0.0011 m^2 and the feed channels had a cross-sectional area of 9.82 mm^2, resulting in a cross-flow velocity of about 1.698 m/s at a flow of 1 l/min.

The feedstock was pumped from the feed tank, which has a 20 l capacity and can be heated or cooled via a coil, and fed into the cells. The permeate stream from each cell was sent to a sample container (cylinders 1 – 3), where the mass was recorded. The retentate was sent to the next cell in series and was fed back to the feed tank after the third cell, with the flow rate measured on the return line.

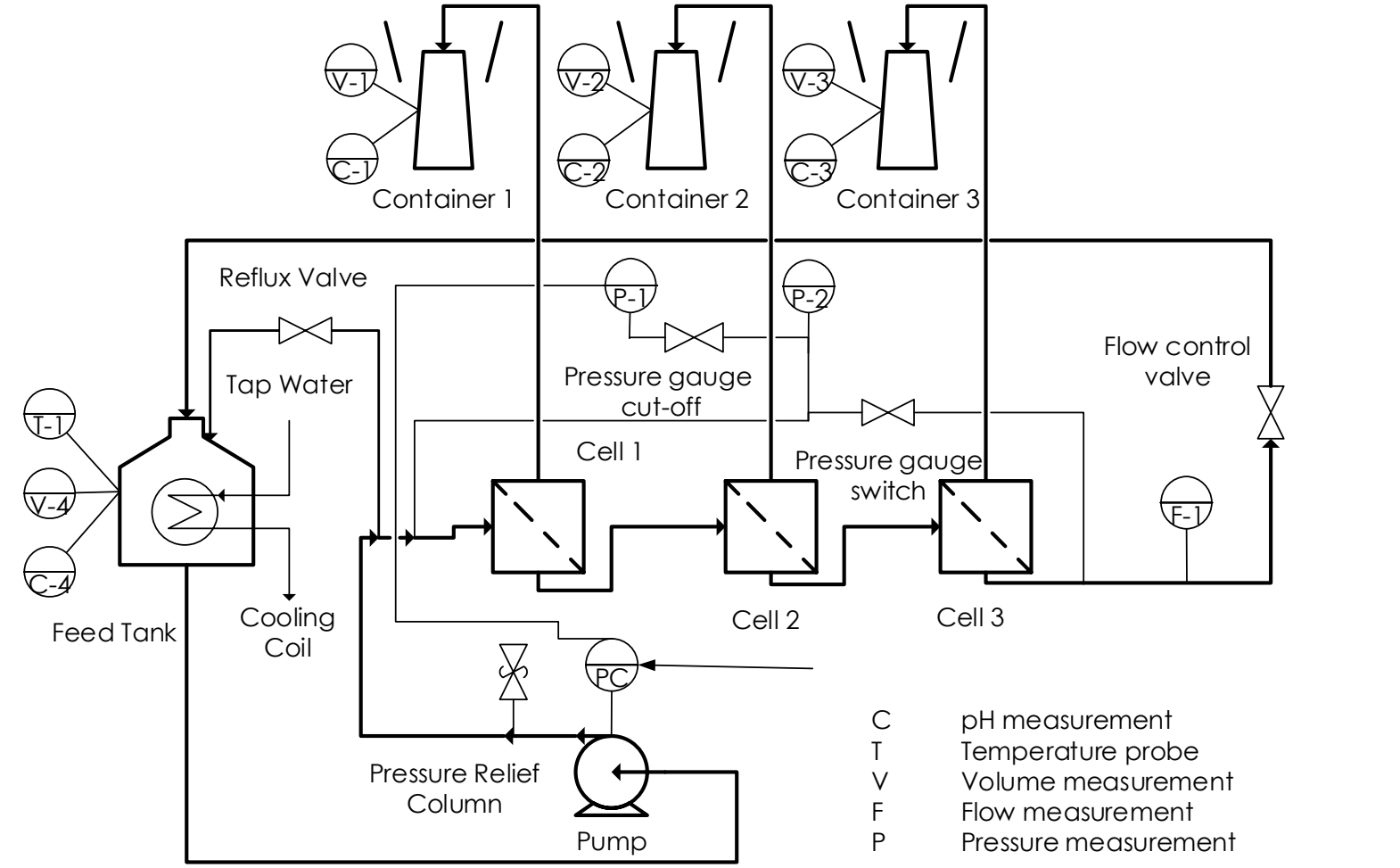

Figure 12: High Pressure Cross-flow Membrane Laboratory Rig

The pressure drop could be measured across the cells using 2 pressure gauges with accuracies of ± 3%, one for lower pressure readings up to 2500 kPa and the other for reading up to 6000 kPa. The pressure drop across the cells and the flow rate were controlled using a combination of opening the valve on the return line and the pump speed. The reflux valve was used for rapid changes in flow rates, and thereby pressures, and also served as a means to reduce hydrostatic shock across the membranes during start-up and shutdown.

3.2.2 Experimental Procedure

The procedure for conducting the experiments was as follows:

1) The mass of water passing through each of the clean membranes was measured, at 5-minute intervals over a period of 20 minutes, with a constant cross-flow velocity. This was done at four TMPs, namely 375, 500, 625 and 750 kPa. The mass measurements along with the density of water and the membrane area allowed for the calculation of the water flux.
2) Similarly, the flux of the two solutions, synthetic urine and stored urine, were calculated. A fresh membrane was used each time and the tests used a constant retentate flow of 1.6 l/min at a pressure of 800 kPa, with 15 minute intervals over a period of 45 minutes. This was done for both the NF 270 and NF 90 membranes. The salt rejections were calculated from these samples using the equipment detailed in section 3.2.3.
3) The same procedure was followed for the XLE membrane but the TMP was increased after each interval starting at a TMP of 800 kPa, increasing to 1000 kPa and finally 1250 kPa. The time interval between each measurement was 20 minutes to allow sufficient volume for analysis. This increase in TMP allowed for further analysis of membrane behaviour.
4) Lastly the water flux experiment, detailed in point 1, was repeated for each of the used membranes. The difference in flux allowed for an analysis of the fouling caused by the solutions.

3.2.3 Analytical Equipment

Evaluating the membrane performance requires the calculation of the rejections. To do this the concentrations of the various elements and ions in the feed and permeate were analysed. The Spectroquant Nova 60 and Agilent 4100 MP-AES were used for this analysis.

3.2.3.1 Spectroquant Nova 60

The Spectroquant Nova 60 is a spectrophotometer, which uses the absorbance of light by the solution to calculate the concentration of the ions present. To do this, specific wavelengths of light, in this case from a tungsten-halogen lamp, are passed through the test solution. The solution would absorb only certain wavelengths of light depending on the substances present. The concentration of the substance will affect the loss of intensity of the light passing through the solution. This is characterised by the transmittance, denoted by T, shown in Equation 3[55]:

$$\mathbf{T} = {}^{\mathbf{I}}/_{\mathbf{I_0}}$$

Equation 3: Transmittance

$I = intentisty\ of\ the\ transmitted\ light$

$I_0 = intial\ intensity\ of\ the\ light$

Where intensity is measured in lux (lx) or lumens per square meter (lm/m^2).

However, the concentration is generally determined using the absorbance, denoted by A. This is the measure of the light absorbed and is used as the absorbance correlates directly with concentration of the absorbing substance. Equation 4 shows the relationship between the absorbance and transmittance:

$$\mathbf{A} = -\mathbf{log}\,\mathbf{T}$$

Equation 4: Absorbance

The concentration can be calculated by using the measured transmittance and the Beer-Lambert law, shown in Equation 5[55]:

$$\mathbf{A} = \sum_{\mathbf{i=1}}^{\mathbf{N}} \mathbf{A_i} = \sum_{\mathbf{i=1}}^{\mathbf{N}} \boldsymbol{\varepsilon_i c_i l}$$

Equation 5: Absorbance, Concentration relationship

$i = chemical\ species$

$\varepsilon_i = Molar\ attentuation\ coefficient\ \left({}^{m^2}/_{mol}\right), species\ specific\ constant$

$c_i = Molar\ concentration\ \left({}^{mol}/_{l}\right)$

$l = path\ length\ of\ light\ through\ the\ sample\ (m)$

Knowing the expected concentration range of the desired species enables the preparation of the sample so that the Nova 60 can accurately measure the intensity of the transmitted light. This preparation uses reagents to convert the species to be measured into a coloured compound and includes masking agents that reduce the interference of other ions in absorbing light at the specific wavelengths used. The Nova 60 will then calculate the concentration of the selected species. Section 8.2.1 details this procedure for various species.

A limited number of testing kits for the Nova 60 were available and therefore the tests were limited to one pressure per membrane for each of the solutions.

3.2.3.2 Agilent 4100 MP-AES

The Agilent 4100 MP-AES is a Microwave Plasma – Atomic Emission Spectrometer. In general, an atomic emission spectrometer (AES) allows for the calculation of the concentration of specific elements by measuring the intensity of light emitted at a wavelength particular to each element. This emission is the result of atoms at an excited energy state releasing their energy when moving back to their ground energy states. The way in which the atoms achieve this excited state differs for each type of AES. These include using a flame, inductively coupled plasma, and electric arc or in this case microwave energy. The industrial magnetron charges nitrogen plasma with microwave energy and this is then used to excite the atoms of the sample. When the excited atoms return to the ground state the emitted light is split by a prism and detected by a spectrometer. [56]

The 4100 MP-AES is capable of measuring the concentration of a wide range of metals as well as phosphorous, silicon and sulphur accurately and the measurements for each element are not as prone to spectral interference from other elements as other AES methods, such as inductively coupled plasma AES (ICP-AES) machines. [56]

The ion concentrations measured by the Nova 60 were of total nitrogen, ammonium and chloride. The element concentrations measured by the 4100 MP-AES were phosphorous, potassium and sodium.

3.2.3.3 Other

The pH of the solution was measured using pH strips, resulting in an accuracy of ±0.5. The conductivity of the permeate streams could not be measured using the available conductivity meters as the volume obtained during the operating time was insufficient.

3.3 Data Analysis

3.3.1 Transport Model

Solute flux can be calculated directly using the water flux and solute concentration:

$$\boldsymbol{J_i = J_w C_{i,P}}$$

Equation 6: Solute Flux from Water Flux

But can also be calculated using the definition of flux; the mass of solute moving across the membrane area per unit of time, shown in Equation 7:

$$\boldsymbol{J_i = \frac{1}{A} . \frac{dm_i}{dt}}$$

Equation 7: Solute Flux

$m = mass\ flowrate$

$A = membrane\ area$

The solute rejection can be calculated using permeate and feed concentrations, by Equation 8:

$$\boldsymbol{R_i = 1 - \frac{C_{i,P}}{C_{i,F}}}$$

Equation 8: Solute Rejection

$C_{i,P} = permeate\ concentration\ solute\ i$

$C_{i,F} = feed\ concentration\ solute\ i$

Rearranging Equation 8 to solve for permeate concentration gives:

$$\boldsymbol{C_{i,P} = C_{i,F}(1 - R_i)}$$

Equation 9: Solute Concentration in Permeate

Equating Equation 6 and Equation 7 and replacing the solute permeate concentration with Equation 9 results in:

$$\boldsymbol{\frac{1}{A} . \frac{dm_i}{dt} = J_w C_{i,P} = J_w C_{i,F}(1 - R_i)}$$

Equation 10

Rearranging to solve for the mass flowrate gives:

$$\frac{dm_i}{dt} = J_w C_{i,F}(1 - R_i)A$$

Equation 11: Solute Mass Flowrate

The water flux is a function of the TMP and is effected by fouling, but can be assumed constant at a constant TMP for the purposes of modelling the data:

$$J_w = -\frac{dV}{dt}.\frac{1}{A}$$

Equation 12: Water Flux at constant TMP

$V = feed\ volume$

The transmembrane pressure, defined in Equation 13, is the driving force for the solute flux:

$$|\Delta P| = \frac{P_F + P_R}{2} - P_P$$

Equation 13: Transmembrane Pressure

$P_F = feed\ pressure$

$P_R = retentate\ pressure$

$P_P = permeate\ pressure$

3.3.2 Effect of Membrane Area

The flow of solute from the feed to the permeate would decrease the solute concentration in the feed over time. Although this change is negligible for this system, due to the small permeate flowrate compared to the feed volume, the effect can be shown by increasing the area in the above model

3.3.3 Water Recovery

A high water recovery is vital for the nanofiltration treatment to be viable. Having a good split between the NPK and Na does no good if this only happens at low water recoveries as this would mean most of the nutrients would be carried off in the retentate. Equation 14 is used to calculate the water recovery.

$$Water\ Recovery = \frac{Initial\ volume\ of\ water - Final\ volume\ of\ water}{Initial\ volume\ of\ water}$$

Equation 14: Water Recovery

3.3.4 Separation Factors

A good way to show the split between the different ions is to calculate the separation factors. These factors clearly show the degree to which the species are rejected by the membrane with reference to a chosen base species.

$$\alpha_{i,j} = \frac{\left[C_i / C_j \right]_{Permeate}}{\left[C_i / C_j \right]_{Feed}}$$

Equation 15: Separation Factor

A low separation factor (<1) shows that the membrane will reject species 'i' to a greater degree than species 'j' and a high separation factor shows the opposite. The further from 1 the separation factor is the larger the split will be between the two species. If the separation factor is equal to 1, this means there is no difference in the rejection of component '*i*' or '*j*'.

4 RESULTS AND DISCUSSION

The main objectives of this project, as stated in section 1.3, were to place membrane processes in the context of urine treatment, identify the knowledge gaps which prevent the usage of membrane systems for urine processing, and lastly to explore critical knowledge gaps for the use of nanofiltration through experimentation. The first two objectives have been addressed in the literature review and the subsequent analysis, and the last objective is explored here.

The specific knowledge gaps, identified in section 2.6.1 and specified in detail in section 3.1, can be summarised as follows: verifying salt rejections reported in literature and determining if it would be possible to retain the majority of the valuable minerals, including potassium, phosphorous and nitrogen, while allowing permeation of the undesired sodium chloride in hydrolysed urine.

4.1 Analytical Equipment

This section details findings for this project relating to the analytical equipment used to measure the samples. Included will be any concerns regarding the precision and accuracy of the measurements and how these were dealt with.

4.1.1 Spectroquant Nova 60 Operation

The Nova 60 has a few limitations when taking measurements. Firstly, the test kits are only available for selected species and for a specific concentration range. The concentration of the sample can be diluted to fall into this range but this introduces the possibility of dilution errors which are difficult to detect and prove when only one sample is prepared per sample taken. Second, as mentioned above, when measuring for one species another species may interfere with the readings if the concentration of the interfering species is too high for the masking agents to deal with. This was not a problem during this project as the appropriate kits were selected as the probable stream compositions were known and the actual compositions of interfering species was checked again once readings were done. Lastly, the Nova 60 relies on the sample being solids free for accurate concentration readings. Due to the samples coming from nanofiltration permeate this was not an issue.

For accurate measurements it is important to regularly calibrate the Nova 60, ensure that the species likely to interfere with the species being measured are within acceptable concentrations, carefully follow the preparation procedure for the various test kits and ensure that the sample cells are properly cleaned. In addition, multiple preparations of each sample would be ideal, however due to cost of test kits this is prohibitive.

During test work the readings of each prepared sample was repeated three times. These samples were prepared using dilution factor between 20 and 100, which accounts for the measurement ranges discussed being lower than the actual sample concentrations. The precision for these readings was excellent for the samples with lower concentrations, below 10 mg/l, and fairly good for higher concentrations, 10 to 100 mg/l. The precision can be found through first calculating the standard deviation of the repeated measurements of the same sample, the standard deviation can then be compared to the detection range. The standard deviations calculated this way were as follows: 0.1 mg/l for 0-5 mg/l, 0.578mg/l for 5 – 10 mg/l, 1.57 mg/l for 10-30 mg/l and 3.46 mg/l for 30-70 mg/l.

4.1.2 Analytical Precision

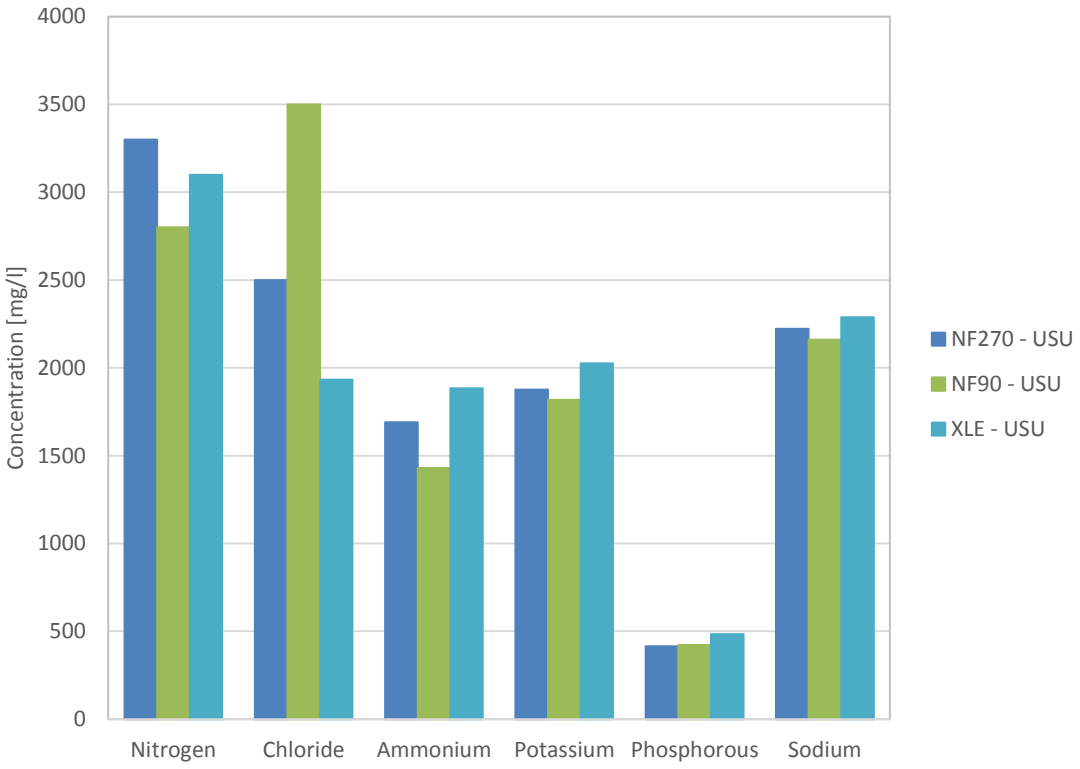

Figure 13: Initial Concentration of Synthetic Urine

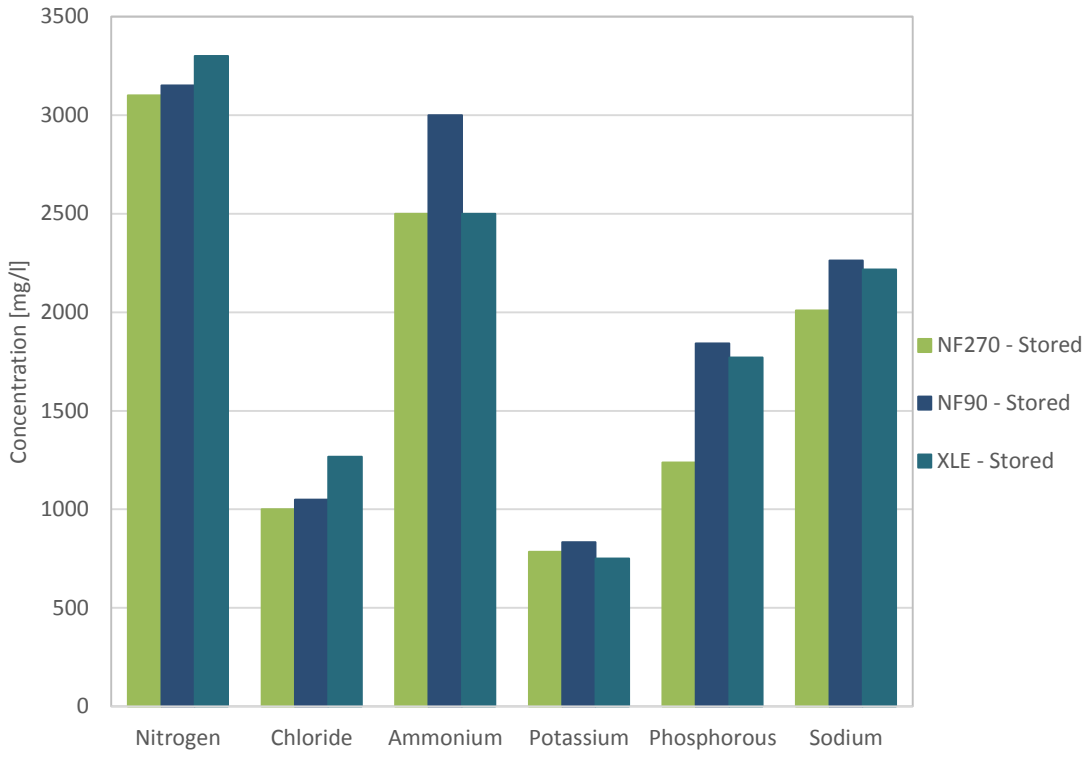

Figure 14: Initial Concentration of Stored Urine

Table 5: Stored and Synthetic Urine Composition and Analytical Precision

Urine Type		N	Cl	NH4	K	P	Na
USU	Average	3067	2644	1668	1907	440	2224
	S. Dev.	252	793	227	106	37	63
	% Error	8	30	14	6	9	3
Stored	Average	3183	1106	2667	790	1617	2162
	S. Dev.	104	142	289	42	50	135
	% Error	3	13	11	5	3	6

In an attempt to quantify the precision of the sample preparation and measurement method together, the initial measurements from each membrane run was compared on a per element basis for both the synthetic urine, Figure 13, and stored urine, Figure 14. These figures show the precision of both the Nova 60, which measured the nitrogen, ammonium and chloride, and the Agilent 4100, which measured the sodium, potassium and phosphorous. The measurements for each element should be the same if the analysis procedure was precise. As seen from the figures, this was not the case.

The easiest way to compare this error over the various concentration ranges was to use the standard deviation of the measurements and then take this as a percentage of the average value for that element. The results, shown in Table 5, shows that the measurements for the synthetic urine was less precise than for the stored urine. Perhaps because the more diverse mix of

species in the stored urine caused less interference during measurements than the limited, but more concentrated, chemicals in the synthetic solution. The table also shows that the Agilent 4100 is generally more precise than the Nova 60. This makes sense as the preparation procedure for the Nova 60 is more complex than the dilution necessary for the Agilent 4100.

4.2 Flux Results

4.2.1 Water Flux versus Transmembrane Pressure

The first step in the experimental phase was to find the clean water flux with varying transmembrane pressure for each of the membranes. This would allow a comparison to the water flux after the synthetic and stored urine had been passed through the membrane. This comparison would give an indication of the fouling potential of the two solutions.

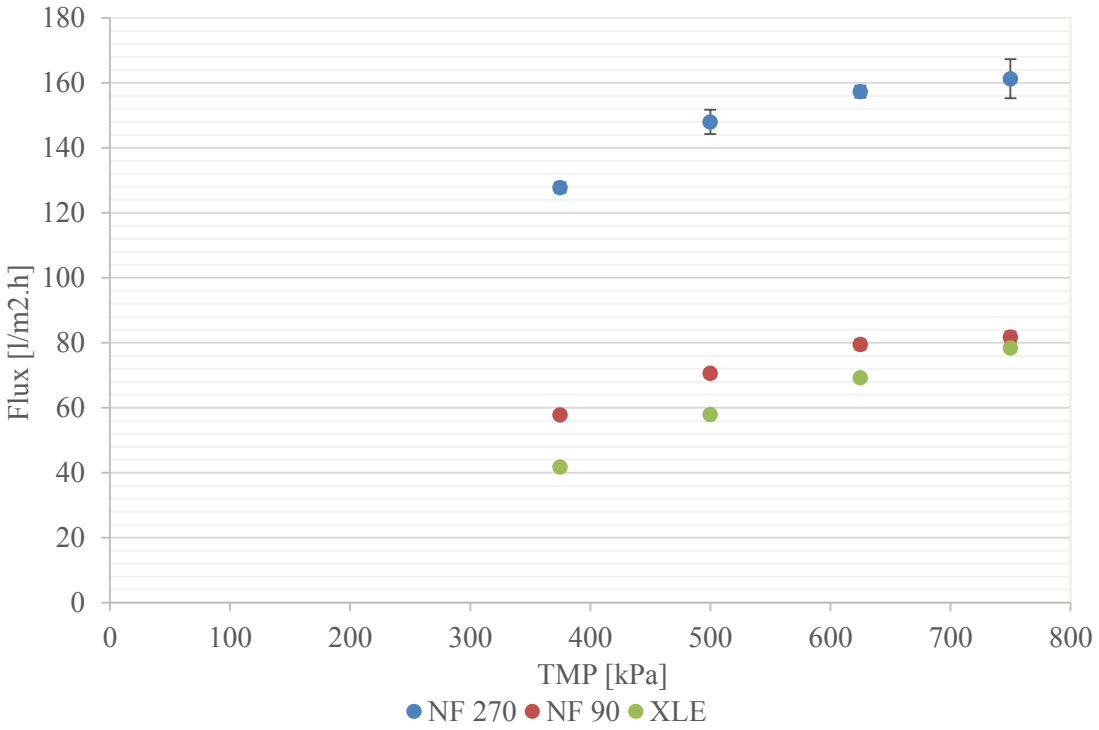

Figure 15: Water flux vs transmembrane pressure for the 3 membranes

As seen in Figure 15, the transmembrane pressure was varied from 375 kPa to 750 kPa and followed the expected trend of flux increasing with increasing membrane MWCO. The trend also shows that the flux increases with increasing transmembrane pressure. This follows the typical models for flux vs TMP, with a linear region proportional to the TMP, a transitional region, where most of the data points fall, and the mass transfer controlled region approached around the 800 kPa mark in these cases. The data for Figure 15 ca be found in Table 8, Table 7 and Table 9 in section 8.3.1

These plots also indicate that the membranes should be operated at a TMP of between 400 and 500 kPa for most efficient energy use, as in this region a change in flux is still proportional to a change in TMP. However, higher TMPs were chosen to ensure a high enough flux so that the operating time for the trials was reasonable. During industrial scale-up the choice of TMP will be a trade-off between the capital cost (CAPEX) due to the membrane size and the operating cost (OPEX) of increasing the TMP. In this case a higher CAPEX would be preferable due to the requirement for a low energy system.

4.2.2 Synthetic and Stored Urine Fluxes

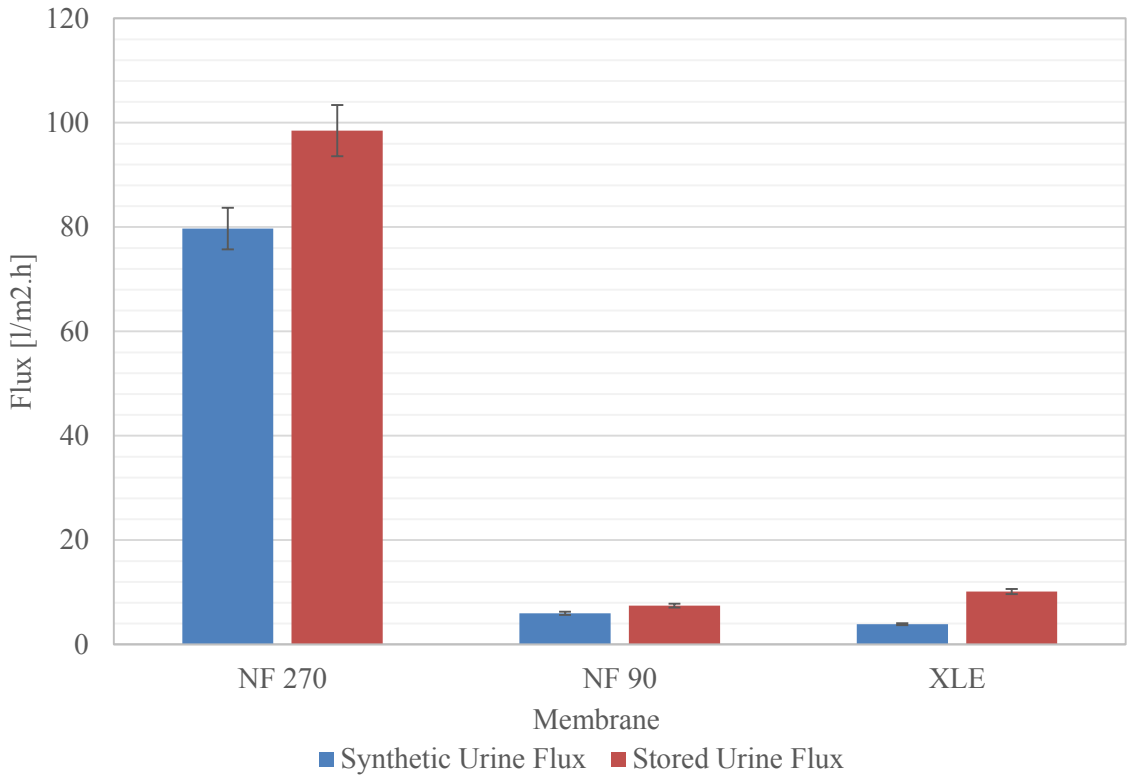

Figure 16: Average Synthetic and Stored urine flux for 3 membranes, at a TMP of 800 kPa

As seen in Figure 16, based on Tables 16-28, at a TMP of 800 kPa and flow rate of 1.6 l/min the synthetic urine flux is lower than the stored urine flux. It was expected that the stored urine flux would be lower as the fouling was expected to be higher than the fouling caused by the synthetic urine. This seemed the likely case as the synthetic was made of laboratory grade ingredients while the stored urine contained organics and particulates, however this is not the case. The lower flux for the synthetic urine could be due to the osmotic pressure difference between the synthetic and stored urine. The synthetic urine has a higher osmotic pressure due to the higher salt concentration, especially chlorine as seen in Figure 13 and Figure 14.

Another unexpected result was the higher flux for the stored urine through the XLE membrane than through the NF 90 membrane, while the synthetic urine and water flux follow the opposite trend. This may be due to the difference in chloride concentration between the stored and synthetic urine, 1100 vs 2600 mg/l, and the NF90 having a higher rejection of chloride ions.

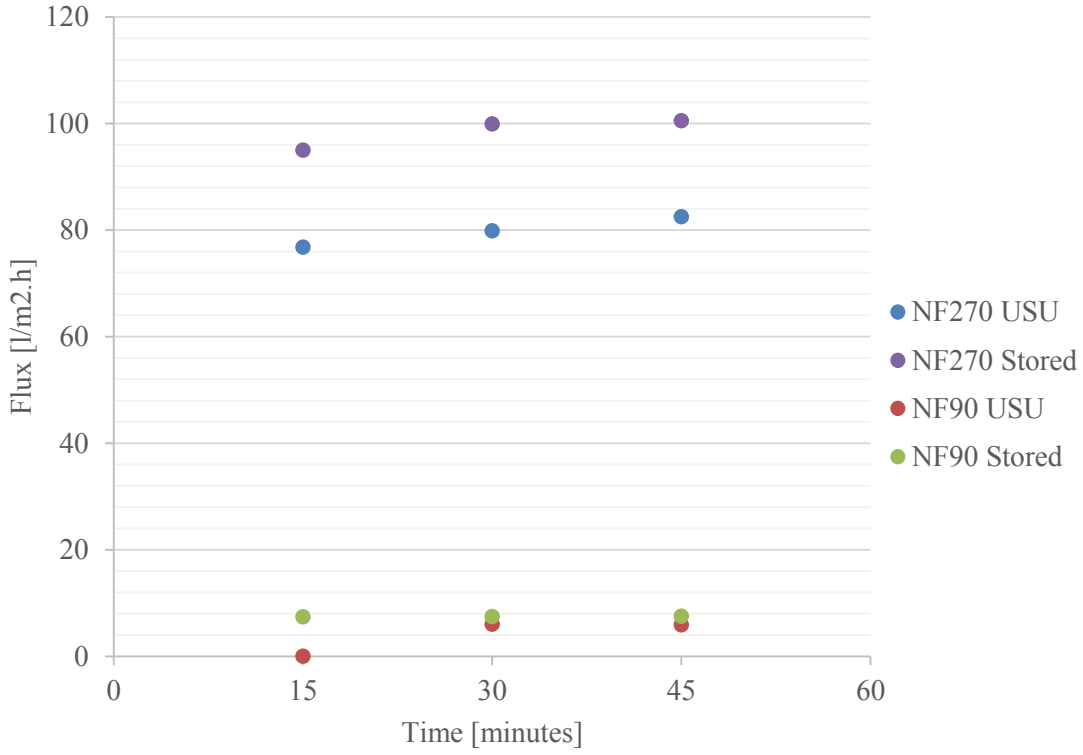

Figure 17: Flux vs Time at constant TMP

Figure 17 shows the water flux over time at constant TMP for the NF270 and NF90 membranes with both stored and synthetic urine. Generally, the plots follow a similar pattern, increasing over time before levelling off. This suggests that there is some initial period where the membrane changing before reaching a steady state, perhaps absorbing a certain amount of molecules before becoming saturated.

4.2.3 Solution Flux versus Transmembrane Pressure

To gauge the behaviour of the solution flux with varying transmembrane pressure, this was varied from 800 kPa to 1250 kPa during the experiments with the XLE membrane. The results obtained at a flow rate of 1.6 l/min, as seen in Figure 18, show a trend of increasing flux with transmembrane pressure. Interesting to note is that this trend differs from that seen with clean water and varying TMP in Figure 15, here the plot is still quite linear, although the flux is significantly lower. This suggests that a higher operating TMP is possible before the transitional area seen with the clean water before 800 kPa.

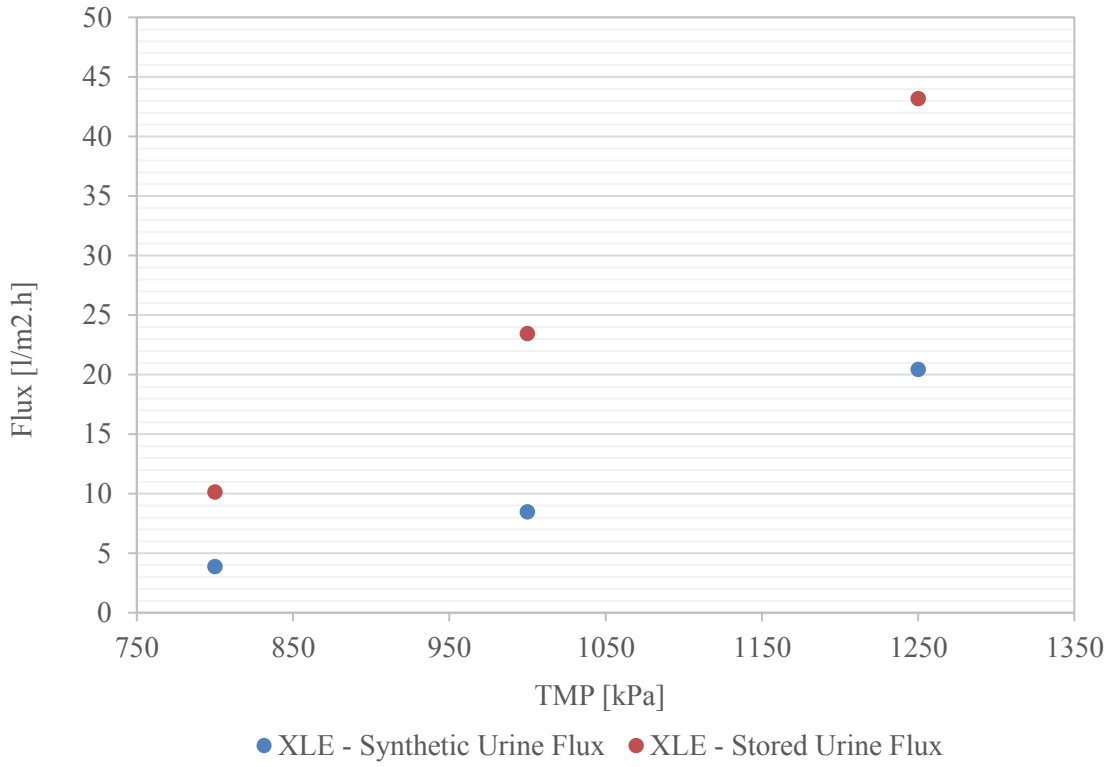

Figure 18: Synthetic and stored urine Flux vs TMP for XLE membranes

4.2.4 Fouling Potential

An important factor to consider when operating membrane systems is the degree to which the membrane can be fouled by the feed solutions. This could lead to drastically reduced flux, which would mean that the transmembrane pressure or more likely the membrane surface area would have to be increased to account for this decrease, which will lead to an increase in energy requirements. The fouling potential of the synthetic and stored urine is shown in Figure 19. This potential was determined by comparing the water flux through each of the membranes before and after nanofiltration experiments.

It can be seen that the degree of fouling increased with decreasing MWCO of the membranes. The flux difference on the NF 270 membrane was negligible, while the difference in the NF 90 and XLE membranes was quite noticeable. The only outlier is the flux in the XLE membrane used with the stored urine, where the water flux after running the urine through the membrane was higher than the water flux through a clean membrane. This illogical result was most likely due to an experimental error.

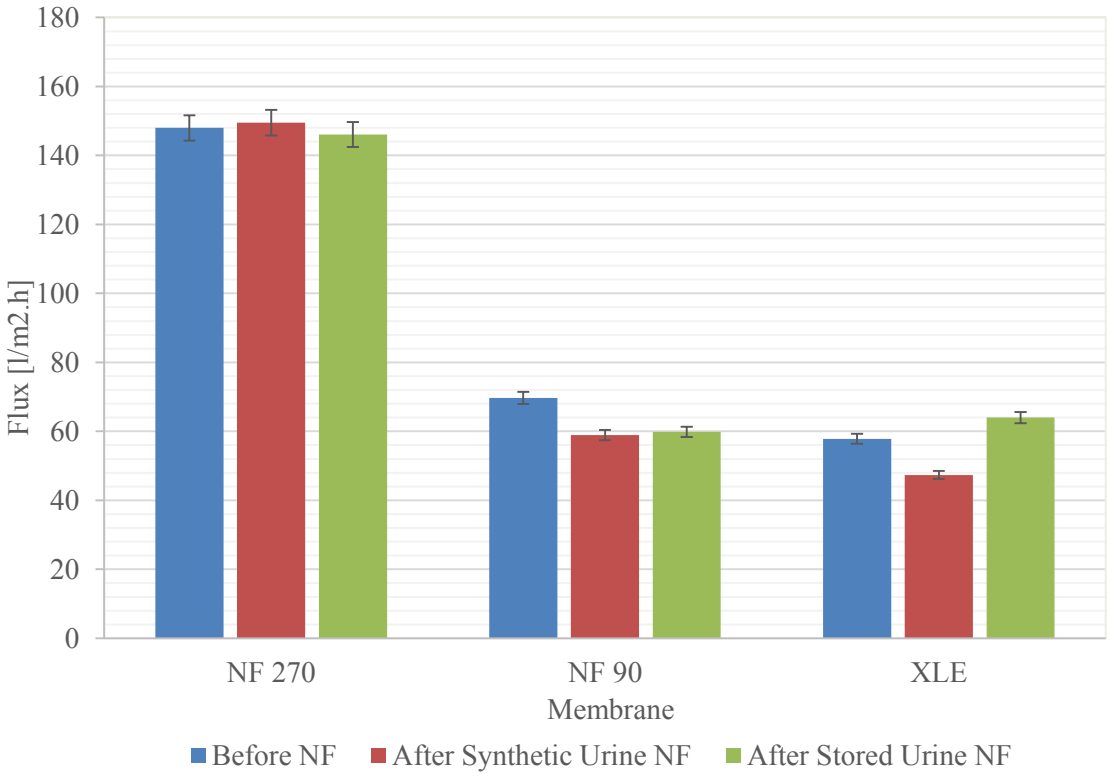

Figure 19: Water flux through the 3 membranes, before and after filtration

4.3 Rejection Results

4.3.1 Rejections by Membrane Type

According to Figure 20, which shows the rejections for the synthetic urine solution and the stored urine, there is a wide range of rejections for the various ions. Generally, rejection increases with decreasing MWCO, from NF 270 to NF 90 to XLE, as expected.

The first exception to this was for chlorine, where the XLE membrane had the lowest rejection, presumably due to electrostatic forces playing a more significant role here than in the other membranes. The second exception was for phosphorous, where the XLE membrane again had a lower rejection than the other membranes but was close enough to be caused by experimental error, within 10% for the stored urine.

The ammonium rejection results were rather peculiar, with opposite trends for the stored and synthetic urine. However, the measurements had high standard deviations and if it were possible further test runs would have been run to obtain more accurate data points.

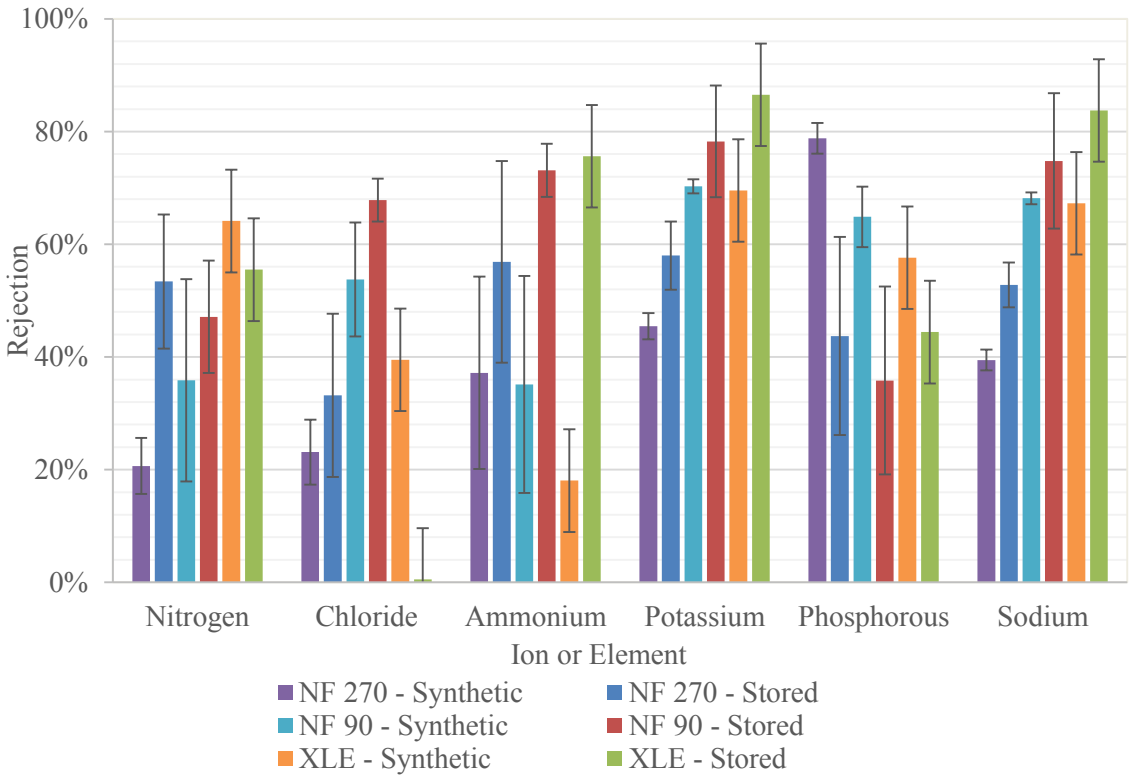

Figure 20: Rejections for Synthetic and Stored Urine for 3 membranes, at a TMP of 800 kPa

4.3.2 Rejection vs Transmembrane Pressure

The rejections for varying TMP, shown in Figure 21, were conducted using the XLE membrane and TMPs of 800, 1000 and 1250 kPa. Generally, the observed rejections increased with an increase in the transmembrane pressure, which is expected as the driving force for the water flux relies on the transmembrane pressure while the driving force for salt passage relies on the concentration difference across the membrane. The rejections of the other ions were approximately the same with changing transmembrane pressure but the inaccuracy in the measurements, as seen by the error bars, indicate more experiments with varying transmembrane pressure need to be conducted.

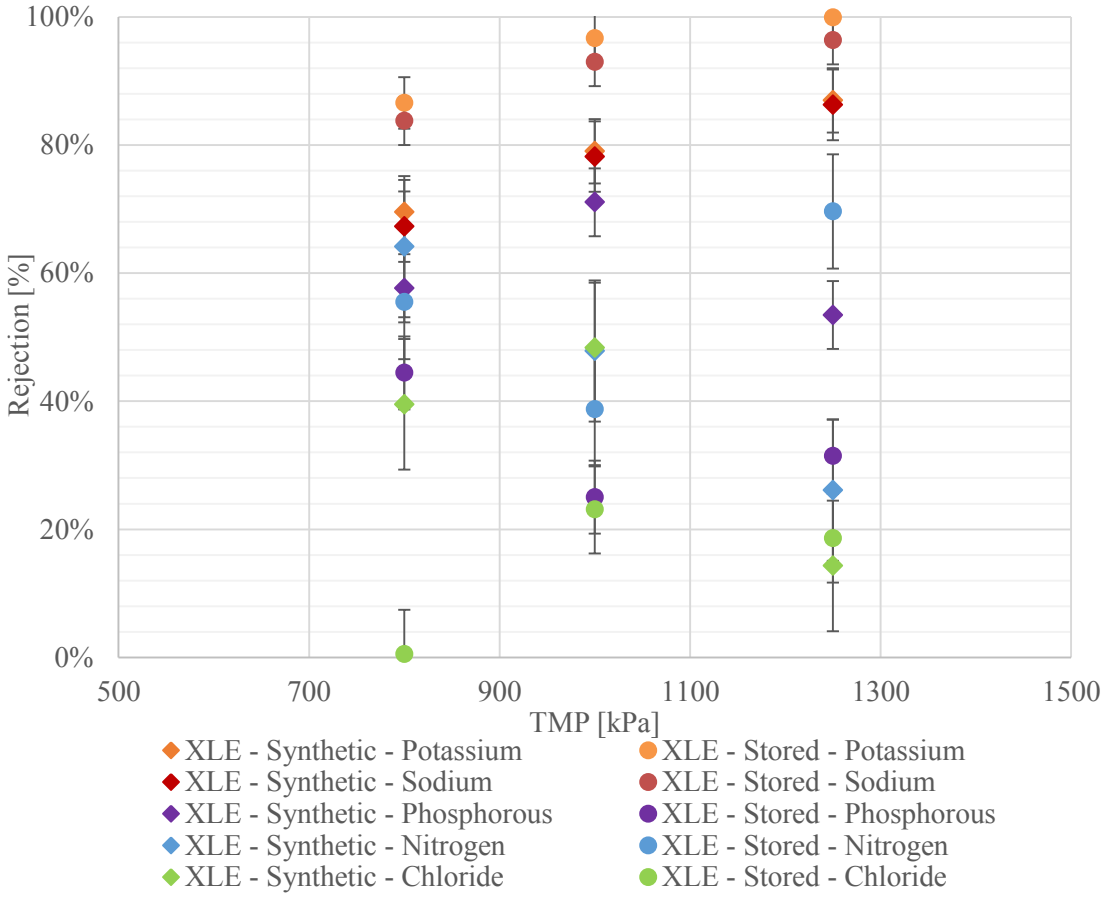

Figure 21: Rejection for synthetic and stored urine vs TMP for XLE membrane

4.3.3 Comparison to Literature

Comparing the average rejections reported by Pronk et al. [22], shown in Figure 22, to those experimentally found with the NF 270 membrane show similar rejections for ammonium but rejections were found to be lower for phosphorous, sodium, chloride and potassium at a transmembrane pressure of 800 kPa.

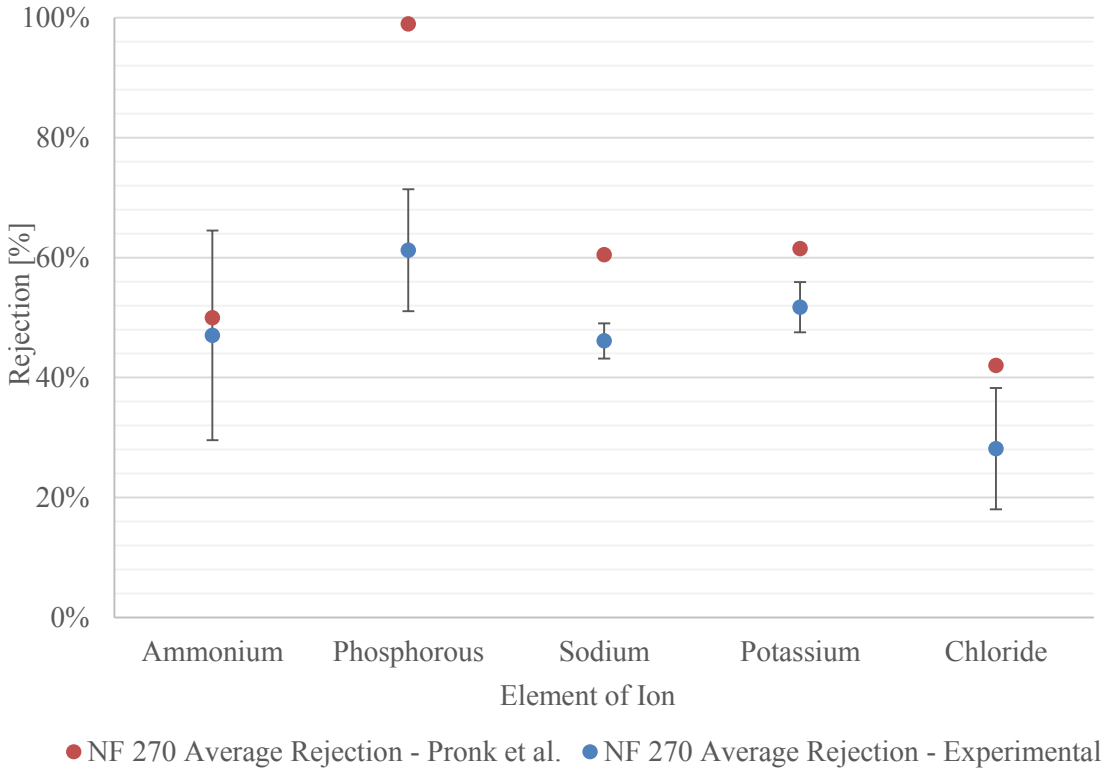

Figure 22: Literature Rejections and Experimental Rejections for NF 270 membrane

Using the increase in rejection with an increase in TMP found when using the XLE membrane it is possible to reach slightly higher rejections. However, it seems highly unlikely that the phosphorous rejection found by Pronk et al. [22] would be achievable using the NF270.

4.4 Nanofiltration Usage

In summary the flux of each of the membranes was found suitable for industrial application and fouling resulted in negligible decrease in flux for the NF 270 membrane, a 15 % decrease in flux for the NF 90 and an 18 % decrease for the XLE membrane, which is within the tolerable limits.

However, the rejection profiles of the membranes do not seem promising for use in the intended system as the split between nitrogen and sodium and potassium and sodium was not sufficient. Other membranes may produce more positive results but perhaps resources would be better spent by considering incorporating a nitrogen recovery process in conjunction with a nanofiltration system that would focus on phosphorous recovery.

4.5 Transport Model

A simulation was run using the model laid out in section 3.3.1 on the NF90 membrane using the synthetic urine composition. The aim of the simulation was to vary the membrane area to determine the effects on water recovery, separation factors, and feed and permeate concentrations. The results of this investigation are presented in this section.

4.5.1 Effect of increasing membrane area on feed concentration

From Figure 23, it can be observed that there is a marked increase in the solute concentrations on the feed as the membrane area was increased.

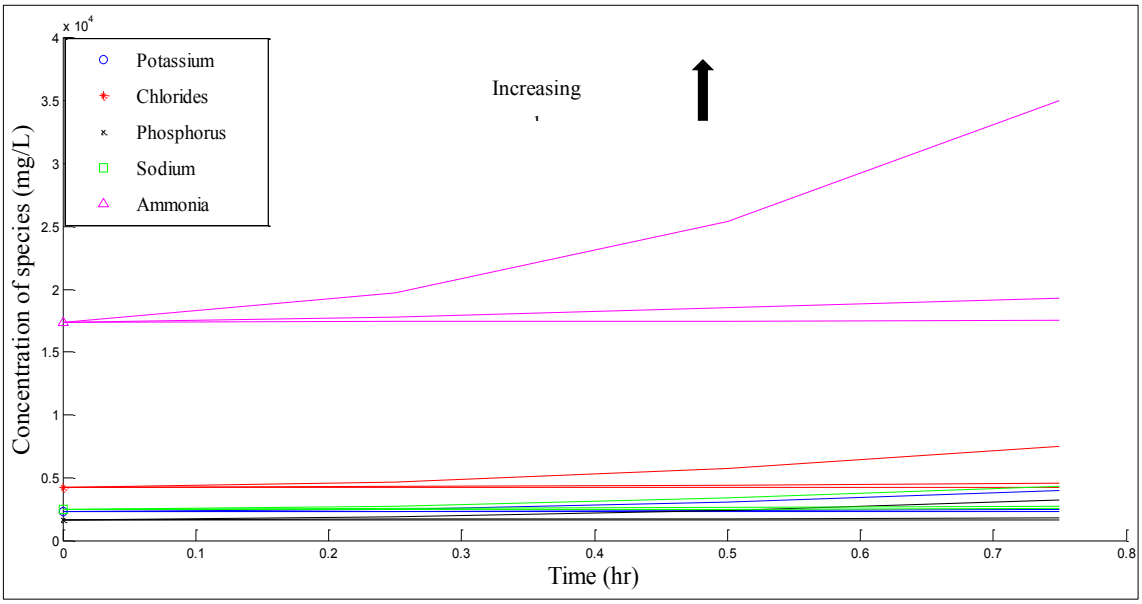

Figure 23: Concentration of species on the feed side

* It must be noted that this plot was done by Mr Brouckaert as part of a summary of various projects falling under the Bill & Melinda Gates Foundation. Explanation and interpretation of plot by the author of this thesis.

The increase in solute concentration indicates that the water flux was increasing much faster than solute flux. Even though the water flux remained unaltered, the volume of water moving across the membrane increased because the membrane area was larger.

4.5.2 Effect of increasing membrane area on permeate concentration

The solute concentration on the permeate side also increased when the membrane area was increased. This can be seen in Figure 24.

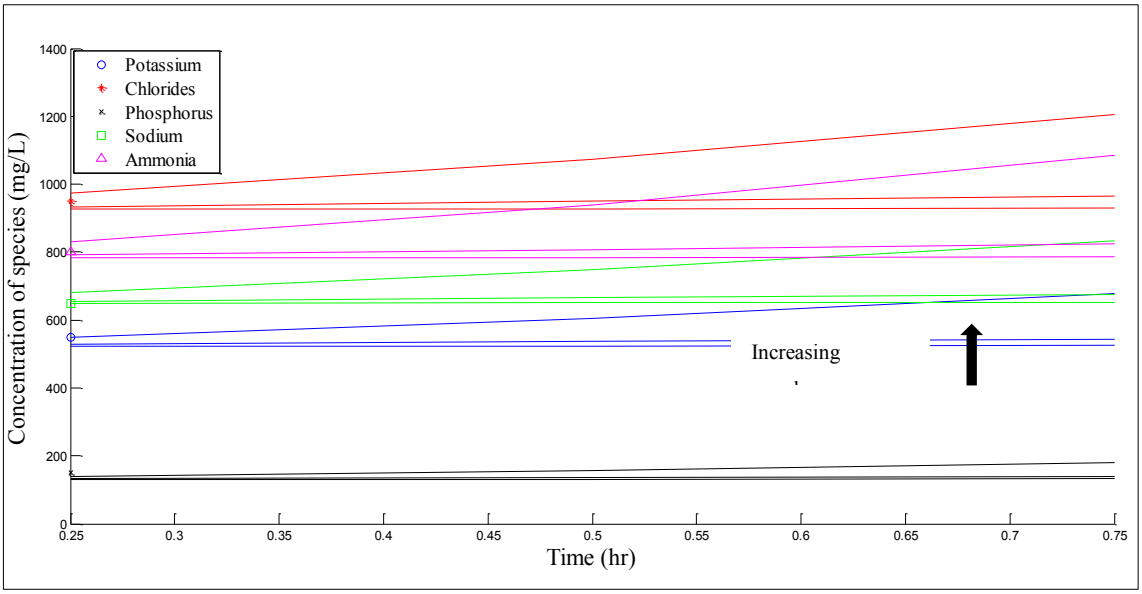

Figure 24: Concentration profile of species on permeate side

* It must be noted that this plot was done by Mr Brouckaert as part of a summary of various projects falling under the Bill & Melinda Gates Foundation. Explanation and interpretation of plot by the author of this thesis.

4.5.3 Water recovery

The system used is designed to have a low water recovery so that the assumption of negligible volume change is valid. So with a membrane surface area of only 0.0011m^2 the water recovery ranged from 0.12% for the NF90 membrane to 2% for the NF270 membrane. Using the transport model detailed in section 3.3.1 the area was increased by multiplying the initial area by 10 and 50, the recovery increased to 10% and 52% respectively for the NF90 membrane with the synthetic urine.

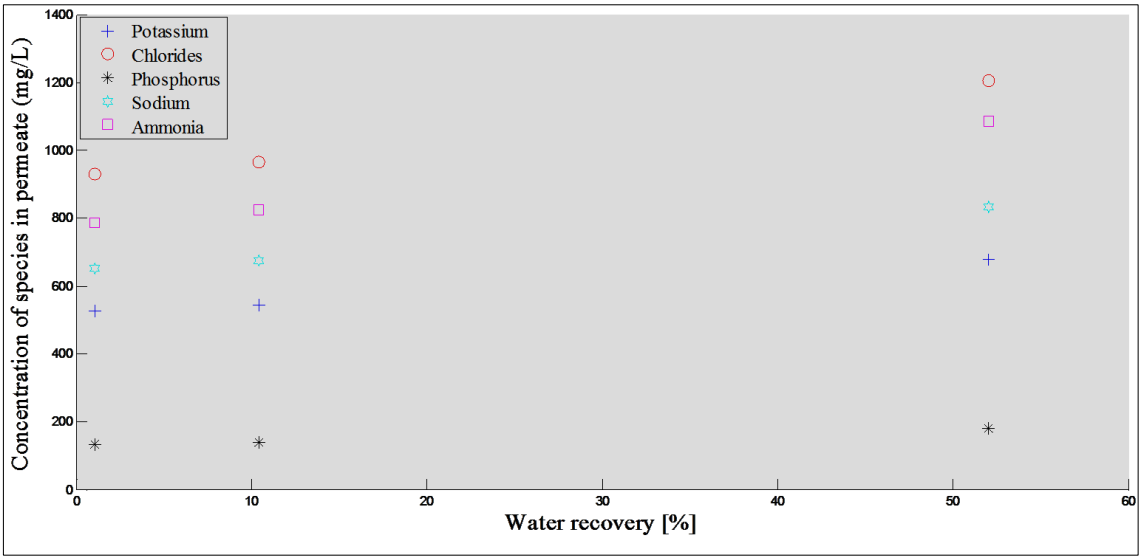

Figure 25: Concentration profile as a function of water recovery for NF90 with synthetic urine

* It must be noted that this plot was done by Mr Brouckaert as part of a summary of various projects falling under the Bill & Melinda Gates Foundation. Explanation and interpretation of plot by the author of this thesis.

Figure 25 shows the concentration of solute species with increasing water recovery based on the transport model. It should be noted that the concentrations in Figure 25 represents the solute concentrations at the end of 45 minutes. The concentrations of all species increased with increasing water recovery. This is expected but not ideal for the sodium. The increase in sodium concentration will have to be weighed against the water recovery when deciding the operating point for full-scale operation.

4.5.4 Separation factors

When choosing the base species for the separation factor it is most useful to choose a species that is desired and has a high rejection to better show if the desired split between the species is taking place. For this reason, the separation of the other ions was measured relative to the phosphate ion. Figure 26 shows the separation factors of various ions, relative to the phosphate ion, at different water recoveries.

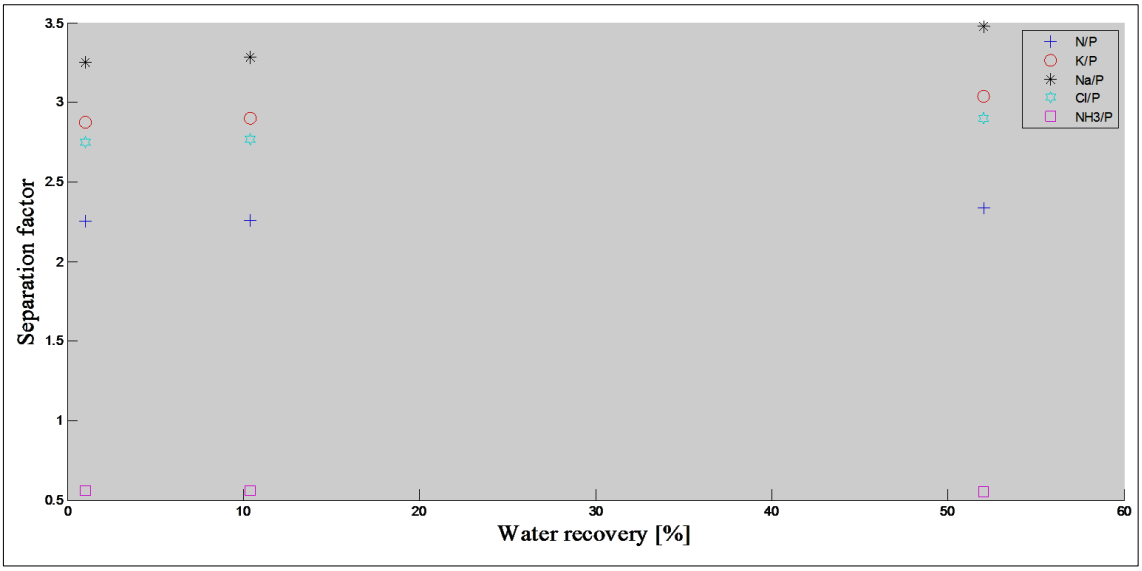

Figure 26: Separation factors relative to the phosphate ion for NF90 with Synthetic Urine

* It must be noted that this plot was done by Mr Brouckaert as part of a summary of various projects falling under the Bill & Melinda Gates Foundation. Explanation and interpretation of plot by the author of this thesis.

Figure 26 shows clearly that there is an excellent separation between phosphate and sodium ions, with a separation factor over 3.25. There is excellent separation and between the ammonium and sodium ions, as ammonium has a separation factor near 0.5. The separation between total nitrogen and sodium is reasonable, with a difference in separation factor of 1, but that the potassium ions tend to pass through the membrane at a similar degree to the sodium ions, having similar separation factors. A slight increase in the separation factors for sodium, potassium, chlorine and nitrogen is observed at high water recoveries, suggesting a greater split at higher recoveries.

5 CONCLUSION

The outcome of this project was twofold: firstly, to explore the use of membrane systems, within the current and potential processes for the treatment of urine in the context of the Re-invent the Toilet Challenge; secondly, to identify and explore knowledge gaps necessary for the implementation of such a membrane system, focussing specifically on the NF membrane stage.

The first outcome was performed by researching the reasons for treating urine and the processes currently in use, as well as those that may be used in future to treat urine and waste water and as well as for desalination. There are seven objectives for the treatment of urine, namely disinfection, volume reduction, stabilisation, phosphorous, nitrogen recovery, nutrient removal and micro-pollutant removal. The processes that can be considered for achieving these objectives can be broken down into 4 major categories, namely membrane filtration, evaporation, nitrogen and ammonia recovery and others. When assessing the treatment processes available, using the literature analysis along with the guidelines set in the RTTC, a combination of different membrane filtration units seemed to be an extremely promising path to pursue.

This finding lead to identifying three promising membrane separation scenarios, which could be used for the recovery of valuable materials from urine, all three involving nanofiltration separation. The most promising scenario was chosen and the scarcity of specific operating parameters and separation potential of the nanofiltration membrane was identified as the key knowledge gap. An experiment was designed, involving the nanofiltration of synthetic and stored urine through 3 different polyamide Dow-Filmtec NF membranes with MWCOs between 100 and 400. This experiment would determine whether a NF could achieve the required separation of the NPK from the sodium chloride. The separation of NPK from sodium chloride is important as sodium chloride inhibits plant growth. The findings lead to several conclusions:

- The precision of the Nova 60 was not as high as anticipated, with Cl^- and $NH4^+$ measurements being particularly problematic with errors between 10 and 30 %.
- The flux achieved by the membranes, 80 – 100 $l/m^2.h$ for NF 270, 6 – 8 $l/m^2.h$ for NF 90, and 4 – 10 $l/m^2.h$ for XLE, followed the order of the MWCO.
- The flux would be sufficient for the RTTC purposes and was similar to literature values.
- The flux was still increasing with increasing TMP between 800 and 1250 kPa for the XLE membrane, indicating that higher TMP conditions are usable without loss of energy efficiency.
- Fouling resulted in negligible decrease in flux for the NF 270 membrane, a 15 % decrease in flux for the NF 90 and an 18 % decrease for the XLE membrane, which is within the tolerable limits.

- The XLE membrane showed higher rejections for N, NH_4^+, K^+ and Na^+ and the lowest for Cl^-, suggesting electrostatic forces influencing rejection.
- The NF270 had the highest rejection for phosphorous and lowest for N, NH_4^+, K^+ and Na^+, which suggests that this membrane may be the most useful if phosphorous recovery was of primary importance.
- The rejections of all species save phosphorous was consistently lower with the synthetic urine as opposed to the stored urine. In the case of phosphorous this can be explained by the stored urine having a much higher concentration than the synthetic.
- The rejections of all species increases with increasing water recovery and there would be a trade-off between water recovery and sodium retention in the NP rich retentate.
- The transport model suggests a high separation between phosphorous and sodium and ammonium and sodium, this was supported for phosphorous by previous work in literature but not during these trials. Neither literature nor these trials support the transport model with the ammonium/sodium split.
- These results suggest that perhaps using nanofiltration membranes for the recovery of phosphorous in conjunction with a second type of technology for the recovery of nitrogen will be a viable process rather than membranes alone.

6 RECOMMENDATIONS

The project fulfilled the research outcomes set out in section 1.3, however there are a number of recommendations for future research in the area of membrane systems, in particular nanofiltration, and urine:

- Perform more experiments with this set of membranes to obtain a larger set of results so that a more accurate model can be fitted.
- Perform experiments with membranes of different membrane material which may provide a more beneficial separation between the desired and undesired salts.
- Investigate the impact of temperature and pH on rejections in order to attempt to achieve the desired separation.
- Consider incorporating the membrane system with a nitrogen recovery method such as ammonia stripping to recover a higher portion of the NPK.

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8 APPENDICES

8.1 Urine Composition

8.1.1 Fresh Urine

Table 6: Constituents of Human Urine [8]

Table I

CONSTITUENTS OF HUMAN URINE EXCEEDING 10 mg/1. FROM REFERENCE 12

Item	Formula	Formula Weight	Range mg/l	Range mg/l	Solubility Limit In A Binary Solution g/100g H_2O
Total Solutes			36,700	46,700	---
Urea	H_2NCONH_2	60.1	9,300	23,300	119
Chloride	Cl^-	35.5	1,870	8,400	---
Sodium	Na^+	23.0	1,170	4,390	---
Potassium	K^+	39.1	750	2,610	---
Creatinine	$C_4H_7N_3O$	113.1	670	2,150	8.7
Sulfur, Inorganic	S	32.1	163	1,800	---
Hippuric Acid	$C_6H_5CO{\cdot}NHCH_2{\cdot}CO_2H$	179.2	50	1,670	0.367
Phosphorus, Total	P	31.0	470	1,070	---
Citric Acid	$HOC(CH_2CO_2H)_2CO_2H$	192.1	90	930	208
Glucuronic Acid	$C_6H_{10}O_7$	194.1	70	880	S.
Ammonia	NH_3	17.0	200	730	---
Uric Acid	$C_5H_4O_3N_4$	168.1	40	670	0.00645
Uropepsin (as Tyrosine)	$HO{\cdot}C_6H_4{\cdot}C_2H_3(NH_2){\cdot}CO_2H$	181.2	70	560	0.04
Bicarbonate	HCO_3^-	61.0	20	560	---
Creatine	$HN{:}C(NH_2)N(CH_3){\cdot}CH_2{\cdot}CO_2H{\cdot}H_2O$	149.2	0	530	1.4
Sulfur, Organic	S	32.1	77	470	---
Glycine	$NH_2{\cdot}CH_2{\cdot}CO_2H$	75.1	90	450	23
Phenols	$C_6H_5{\cdot}OH$	94.1	130	420	8.2
Lactic Acid	$CH_3{\cdot}CHOH{\cdot}CO_2H$	90.1	30	400	∞
Calcium	Ca^{+2}	40.1	30	390	---
Histidine	$C_3H_3N_2{\cdot}CH_2{\cdot}CH{\cdot}(NH_2){\cdot}CO_2H$	155.2	40	330	S.
Glutamic Acid	$HO_2C{\cdot}CHNH_2{\cdot}(CH_2)_2{\cdot}CO_2H$	147.1	<7	320	1.5
Androsterone	$C_{19}H_{30}O_2$	290.5	2	280	i.;S.
1-Methylhistidine	$C_3H_3N_2CH_2CH(NH{\cdot}CH_3){\cdot}COOH$	169.2	30	260	
Magnesium	Mg	24.3	20	205	---
Imidazole Derivatives	$C_3H_4N_2$	68.1	90	200	S.
Glucose	$C_6H_7O_6(COCH_3)_5$	390.4	30	200	0.15
Taurine	$NH_2{\cdot}CH_2{\cdot}CH_2{\cdot}SO_3H$	125.2	5	200	6.4
Aspartic Acid	$C_4H_7O_4N$	133.1	<7	170	2.71
Carbonate	CO_3^{-2}	60.0	100	150	---
Cystine	$[HO_2C{\cdot}CH(NH_2){\cdot}CH_2S{\cdot}]_2$	240.3	7	130	0.01
Citrulline	$NH_2CONH(CH_2)_3{\cdot}CH{\cdot}(NH_2){\cdot}CO_2H$	175.2	0	130	S.
Threonine	$C_4H_9O_3N$	119.1	10	120	S.
Lysine	$(NH_2)_2C_5H_9{\cdot}CO_2H$	146.2	5	110	V.S.
Indoxylsulfuric Acid	$C_8H_7ON{\cdot}H_2SO_4$	231.2	3	110	
m-Hydroxyhippuric Acid	$C_6H_4COHC(CONH{\cdot}CH_2COOH)$	195.2	1	100	
p-Hydroxyphenyl-Hydrocrylic Acid			1	100	

Table I

CONSTITUENTS OF HUMAN URINE EXCEEDING 10 mg/1. FROM REFERENCE 12 (Concluded)

Item	Formula	Formula Weight	Range mg/l	Range mg/l	Solubility Limit In A Binary Solution g/100g H_2O
Aminoisobutyric Acid	$H_2N{\cdot}CH_2{>}CH{\cdot}CHOOH$ (CH$_3$)	103.1	3	120	
Inositol	$C_6H_{12}O_6$	180.2	5	100	
Formic Acid	$H{\cdot}CO_2H$	46.0	20	90	∞
Urobilin	$C_{33}H_{40}O_6N_4$	588.7	7	90	
Tyrosine	$HO{\cdot}C_6H_4{\cdot}C_2H_3(NH_2){\cdot}CO_2H$	181.2	10	70	0.04
Pyruvic Acid	$CH_3{\cdot}CO{\cdot}CO_2H$	88.1	2	70	∞
Albumin			7	70	
Asparagine	$HO_2C{\cdot}CH(NH_2){\cdot}CH_2{\cdot}CONH_2$	132.1	20	70	3.1
Tryptophan	$C_6H_4{\cdot}NH{\cdot}CH{:}C{\cdot}C_2H_3(NH_2)CO_2H$	286.8	5	60	25
Ketones (as Acetone)	CH_3COCH_3	58.1	10	50	∞
Serine	$HO{\cdot}CH_2{\cdot}CHNH_2{\cdot}CO_2H$	105.1	20	50	4
Alanine	$H_2N{\cdot}CH(CH_3){\cdot}CO_2H$	89.1	15	50	20.5
Purine Bases	$C_5H_4N_4$	120.1	0	50	i.
Glycocyamine			15	45	
Proline	$HN{\cdot}(CH_2)_3{\cdot}CH{\cdot}CO_2H$	115.1	<7	40	V.S.
Arginine	$H_2N{\cdot}C(:NH){\cdot}NH{\cdot}(CH_2)_3{\cdot}CH(NH_2){\cdot}CO_2H$	174.2	<7	40	15
Ascorbic Acid	$C_6H_8O_6$	176.1	3	40	V.S
Oxalic Acid	$HO_2C{\cdot}CO_2H$	90.0	1	30	10
Bilirubin	$C_{33}H_{36}N_4O_6$	584.7	3	30	i.
Valine	$(CH_3)_2CH{\cdot}CH(NH_2){\cdot}COOH$	117.2	<7	30	
Phenylalamine	$\beta{\cdot}C_6H_5{\cdot}CH_2{\cdot}CH(NH_2){\cdot}COOH$	165.2	6	30	
Allantoin	$C_4H_6O_3N_4$	158.1	2	25	0.76
Oxoglutaric Acid	$C_5H_6O_5$	146.1	13	25	
Leucine	$(CH_3)_2CH{\cdot}CH_2{\cdot}CH(NH_2){\cdot}COOH$	131.2	8	25	
Guanidinoacetic Acid	$HN{:}C(NH_2)(NH{\cdot}CH_2{\cdot}COOH)$	117.1	9	25	
Isoleucine	$CH_3{\cdot}CH_2{\cdot}CH(CH_3){\cdot}CH(NH_2){\cdot}COOH$	131.2	4	22	
Urobilinogen			0	17	
Ethanolamine	$NH_2{\cdot}CH_2{\cdot}CH_2OH$	61.1	3	15	∞
Guanidine	$(H_2N)_2C{:}NH$	59.1	7	13	V.S.
Methionine Sulfoxide			0	13	
Dehydroascorbic Acid	$C_6H_6O_6$	174.1	3	13	
Other Organics				285	

8.2 Analysis Equipment

8.2.1 Spectroquant Nova 60 SOPs

Standard Operation Procedure – Ammonium Test

(Cat. No. 1.00683)

1. Scope and Field of Application

Test measures both ammonium ions and dissolved ammonia in a concentration range of 2 – 150 mg/l NH_4-N

2. Principle

Ammonium nitrogen (NH_4-N) occurs partly in the form of ammonium ions and partly as ammonia. A pH-dependent equilibrium exists between the two forms. In strongly alkaline solutions NH_4-N is present almost entirely as ammonia, which reacts with hypochlorite ions to form monochloramine. This in turn reacts with a substituted phenol to form a blue indophenol derivative that is determined photometrically.

3. Interferences

Concentrations of foreign substances in mg/l or %					
Al^{3+}	1000	Mn^{2+}	100	EDTA	1000
Ca^{2+}	1000	Ni^{2+}	250	Primary Amines	0
Cd^{2+}	1000	NO_2^-	1000	Secondary Amines	250
CN^-	100	Pb^{2+}	1000	Aminophenols	10
Cr^{3+}	100	PO_4^{2-}	1000	Aniline	50
$Cr_2O_7^{2-}$	1000	S^{2-}	50	Triethanolamine	1000
Cu^{2+}	1000	SiO_3^{2-}	1000	Surfactants	1000
F^-	1000	Zn^{2+}	500	Na-acetate	10%
Fe^{3+}	25			NaCl	20%
Hg^{2+}	500			$NaNO_3$	20%
Mg^{2+}	500			Na_2SO_4	20%

4. Sampling

- Analyze immediately after sampling.
- Preferably collect samples in glass bottles.
- The pH must be within the range 4 - 13. Adjust, if necessary, with sodium hydroxide or sulfuric acid.
- Filter turbid samples.
- Check the ammonium content with the Merckoquant Ammonium Test. Samples containing more than 150 mg/l NH4-N must be diluted with distilled water.

5. Safety Precautions

- Handle concentrated acid with care
- Always use safety goggles, gloves, and laboratory coat while working in laboratory
- After the analysis clean the bottles and beakers with distilled water before for drying
- Dispose any used gloves after completion of analysis
- Clean hands using antiseptic soap and disinfect with ethanol solution
- Avoid spillage and contact with skin. In the latter case wash with copious amounts of cold water and call for medical attention.

6. Apparatus

- Spectroquant
- Pipettes for pipetting volumes of 0.10, 0.20, and 5.0 ml
- Rectangular cells 10 mm (2 pcs), Cat. No. 114946

7. Reagents

- Reagent NH4-1
- Reagent NH4-2 (contains granulate + desiccant capsule)
- Merckoquant® Ammonium Test, Cat. No. 110024
- Universal indicator strips pH 0 - 14, Cat. No. 109535
- Sodium hydroxide solution 1 mol/l
- Sulfuric acid 0.5 mol/l

8. Calibration

To calibrate test solutions of 5.0, 10, 50 and 100 mg/l NH_4-N.

9. Procedure

Measuring range of 2.0 – 75.0 mg/l NH4-N (2.6 – 96.9 mg/l NH4+):

1. Pipette 5.0 ml of reagent NH_4-1, stored between 20 – 30 °C, into a test tube
2. Pipette 0.2 ml of pretreated sample into the test tube and mix.
3. Add 1 level blue microspoon of reagent NH_4-2 and shake vigorously until the reagent is completely dissolved.
4. Leave to stand for 15 minutes, in a test tube rack, then fill the sample into a 10 mm cell and measure in the photometer.

Measuring range of 5 – 150 mg/l NH4-N (6 – 193 mg/l NH4+):

1. Pipette 5.0 ml of reagent NH_4-1, stored between 20 – 30 °C, into a test tube
2. Pipette 0.1 ml of pretreated sample into the test tube and mix.
3. Add 1 level blue microspoon of reagent NH_4-2 and shake vigorously until the reagent is completely dissolved.
4. Leave to stand for 15 minutes, in a test tube rack, then fill the sample into a 10 mm cell and measure in the photometer.

Notes on the measurement:

- Reclose the reagent bottles immediately after use.
- Due to the strong temperature dependence of the colour reaction, the temperature of the reagents should be between 20 and 30 °C.
- Ensure the cells are cleaned, with dry paper towel, for the photometric analysis.
- Measurement of turbid solutions yields false-high readings.
- Ammonium-free samples turn yellow on addition of reagent NH_4-2.
- The pH of the measurement solution must be within the range 11.5 - 11.8.
- The colour of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.
- In the event of ammonium concentrations exceeding 2500 mg/l, other reaction products are formed and false-low readings are yielded. In such cases it is advisable to conduct a plausibility check of the measurement results by diluting the sample (1:10, 1:100)

10. Data Quality

Measurement	2 – 75 mg/l NH_4-N	5 – 150 mg/l NH_4-N
Standard Deviation (mg/l NH_4-N)	± 0.49	± 1.0
Confidence Interval (mg/l NH_4-N)	± 1.2	± 2
Sensitivity (mg/l NH_4-N)	0.3	1
Accuracy (mg/l NH_4-N)	± 1.8	± 4.0

11. Chemical Waste Disposal

- Rinse glassware ammonium-free with distilled water, **do not use detergent.**

Standard Operation Procedure – Chloride Test (Cat. No. 1.14897)

1. Scope and Field of Application

Test measures the chloride concentration in the ranges of 2.5 – 25 and 10 – 250 mg/l Cl^-.

2. Principle

Chloride ions react with mercury(II) thiocyanate to form slightly dissociated mercury(II) chloride. The thiocyanate released in the process in turn reacts with iron(III) ions to form red iron(III) thiocyanate that is determined photometrically.

3. Interferences

Concentrations of foreign substances in mg/l or %					
Al^{3+}	100	Hg^{2+}	2 (10)	Free Chlorine	10
Ca^{2+}	1000	Mg^{2+}	1000	Surfactants	1000
Cd^{2+}	500	Mn^{2+}	1000	$NaNO_3$	20 %
Ag^{+}	5 (10)	Ni^{2+}	500	Na_2SO_4	0.25% (1%)
Cr^{3+}	500	Pb^{2+}	500		
$Cr_2O_7^{2-}$	250	PO_4^{3-}	100		
Cu^{2+}	500	SiO_3^{2-}	1000		
F^-	100	S^{2-}	0.5 (2.5)		
Fe^{3+}	250	Zn^{2+}	500		
Br^-	1 (5)	K^+	1000		
CN^-	0.2 (1)	$NH_4^=$	1000		

4. Sampling

- Preferably collect samples in glass bottles.
- Analyze immediately after sampling.
- The pH must be within the range 1 - 12. Adjust, if necessary, with dilute ammonia solution or nitric acid.
- Filter turbid samples.

5. Safety Precautions

- Handle concentrated acid with cares
- Always use safety goggles, gloves and laboratory coat while working in laboratory
- After the analysis clean bottles and beakers with water keep it for drying
- Dispose the used gloves after completion of analysis
- Clean hands using antiseptic soap
- Disinfect hands after washing with soap
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

6. Apparatus

- Spectroquant
- Pipettes for pipetting volumes of 0.50, 1.0, 2.5, and 5.0 ml
- Rectangular cells 10 mm (2 pcs), Cat. No. 114946
- Universal indicator strips pH 0 - 14, Cat. No. 109535

7. Reagents

- Reagent Cl-1
- Reagent Cl-2
- Nitric acid for 1 mol/l
- Ammonia solution 25%

8. Calibration

To check the photometric measurement system (test reagent, measurement device, and handling) and the mode of working, chloride solutions, 12.5 mg/l Cl^-, and 125 mg/l Cl^- can be used.

9. Procedure

1. Pipette 5 ml, for 2.5 – 25 mg/l Cl^-, or 1 ml, for 10 – 250 mg/l Cl^-, of pretreated sample into test tube.
2. Pipette 2.5 ml of reagent Cl-1 into tube and mix.
3. Pipette 0.5 ml of reagent Cl-2 into tube and mix.
4. Leave to stand for 1 min, then fill the sample into a 10 mm cell.
5. Measure in the photometer.

Notes on the measurement:

- Analyze immediately after sampling.
- Reclose the reagent bottles immediately after use.
- For photometric measurement the cells must be clean. Wipe, if necessary, with a dry paper towel.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be approx. 1.
- The color of the measurement solution remains stable for 30 min after the end of the reaction time stated above. (After 60 min the measurement value would have increased by 5 %.)

10. Data Quality

Measurement	2.5 – 25.0 mg/l Cl^-	10 – 250 mg/l Cl^-
Standard Deviation (mg/l Cl^-)	± 0.19	± 2.8
Confidence Interval (mg/l Cl^-)	± 0.5	± 7
Sensitivity (mg/l Cl^-)	0.3	1
Accuracy (mg/l Cl^-)	± 1.0	± 10

11. Chemical Waste Disposal

- Collect waste in a labeled 2.5L bottle for collection from Waste Tech.

Standard Operation Procedure – Nitrogen (Total) Cell Test

(Cat. No. 1.14763)

1. Scope and Field of Application

Test measures the total nitrogen, in a concentration range of 10 – 150 mg/l N, of solutions with a maximum of 2% sodium chloride.

2. Principle

Organic and inorganic nitrogen compounds are transformed into nitrate according to Koroleff's method by treatment with an oxidizing agent in a thermoreactor. In a solution acidified with sulfuric and phosphoric acid, this nitrate reacts with 2,6-dimethylphenol (DMP) to form 4-nitro-2,6-dimethylphenol that is determined photometrically.

3. Interferences

Concentrations of foreign substances in mg/l or %					
Al^{3+}	1000	Hg^{2+}	1000	Surfactants	500
Ca^{2+}	1000	Mg^{2+}	1000	CSB (K-Hydrogen phthalate)	3500
Cd^{2+}	1000	Mn^{2+}	1000		
Cl^-	10000	Ni^{2+}	1000	Na-acetate	10 %
Cr^{3+}	100	Pb^{2+}	1000	NaCl	2 %
$Cr_2O_7^{2-}$	100	PO_4^{3-}	1000	Na_2SO_4	10 %
Cu^{2+}	1000	SiO_3^{2-}	1000		
F^-	1000	Sn^{2+}	1000		
Fe^{3+}	1000	Zn^{2+}	1000		

When the quantity of reagent N-1K is doubled, the tolerable COD increases ``to 7000 mg/l. In the event of higher COD values false-low results are obtained.

4. Sampling

- Preferably collect samples in glass bottles.
- Analyze immediately after sampling.
- Check, where necessary, the COD with the Spectroquant® COD Cell Test. In the event of COD values of more than 7000 mg/l, the sample must be diluted with distilled water.
- Reclose the reagent bottles immediately after use.

5. Safety Precautions

- Handle concentrated acid with cares
- Always use safety goggles, gloves and laboratory coat while working in laboratory
- After the analysis clean bottles and beakers with clear water keep it for drying
- Dispose the used gloves after completion of analysis
- Clean the hands using antiseptic soap
- Disinfect hands after washing with soap
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

6. Apparatus

- Spectroquant
- Reaction cells
- Thermoreactor
- Pipettes

7. Reagents

- Reagent N-1K
- Reagent N-2K
- Reagent N-3K

8. Calibration

To check the photometric measurement system (test reagent, measurement device, and handling) and the mode of working, nitrogen (total) solutions, 10.0 mg/l N, and 100 mg/l N can be used.

9. Procedure

6. Pipette 1 ml of pretreated sample into an empty cell.
7. Add 9 ml of distilled water into cell and mix.
8. Add 1 level blue microspoon of reagent N-1K and mix.
9. Add 6 drops of reagent N-2K, close cell and mix.
10. Heat the cell at 120 °C in the preheated thermoreactor for 1 hour. Shake the cell briefly after 10 minutes.
11. Pipette 1 ml of the digested solution into a reaction cell. Do not mix.
12. Pipette 1 ml of reagent N-3K the reaction cell, close the cell and mix. Wear eye protection and hold the cell only at the top.
13. Leave the hot reaction to stand for 10 min (reaction time). Do not cool with water.
14. Measure in the photometer

Notes on the measurement:

- Analyze immediately after sampling.
- Reclose the reagent bottles immediately after use.
- For photometric measurement the cells must be clean. Wipe, if necessary, with a dry paper towel.
- The colour of the measurement solution remains stable for 30 min after the end of the reaction time stated above. (After 60 min the measurement value would have increased by 5 %.)

10. Data Quality

Measurement	10 – 150 mg/l N
Standard Deviation (mg/l N)	± 1.1
Confidence Interval (mg/l N)	± 3
Sensitivity (mg/l N)	2
Accuracy (mg/l N)	± 5

11. Chemical Waste Disposal

8.3 Result Sheets

8.3.1 Water Flux

Time	Vial	Mass of measuring vial [g]			Pressure	Retentate Flow		Flow	Flux
s		Initial	Final	Net	Average	Average	Variance	ml/min	$l/m^2.h$
0					375	1.15	0.02		
300	1A	16.68	19.86	3.17	375	1.2	0.02	0.63	58
600	2A	16.79	19.98	3.20	375	1.25	0.02	0.64	59
900	3B	16.43	19.60	3.16	375	1.26	0.02	0.63	58
1200	4A	16.25	19.37	3.12	375	1.28	0.02	0.62	57
1500	5A	16.60	19.72	3.12	375	1.28	0.02	0.62	57
1800	1B	16.66	19.79	3.13	375	1.3	0.02	0.63	57
								0.63	58
0					500	1.2	0.02		
300	1A	16.69	20.62	3.94	500	1.18	0.02	0.79	72
600	2A	16.79	20.69	3.90	500	1.2	0.02	0.78	72
900	3B	16.44	20.32	3.89	500	1.2	0.02	0.78	71
1200	4A	16.25	20.08	3.83	500	1.2	0.02	0.77	70
1500	5A	16.60	20.40	3.80	500	1.2	0.02	0.76	70
1800	1B	16.66	20.41	3.75	500	1.2	0.02	0.75	69
								0.77	71
0					625	1.12	0.02		
300	1A	16.68	21.14	4.46	625	1.12	0.02	0.89	82
600	2A	16.78	21.14	4.36	625	1.15	0.02	0.87	80
900	3B	16.44	20.74	4.30	625	1.15	0.02	0.86	79
1200	4A	16.26	20.48	4.22	625	1.15	0.02	0.84	77
1500	5A	16.60	20.98	4.38	625	1.15	0.02	0.88	80
1800	1B	16.66	20.93	4.27	625	1.15	0.02	0.85	78
								0.87	79
0					750	1.14	0.02		
300	1A	16.68	21.19	4.51	750	1.14	0.02	0.90	83
600	2A	16.79	21.24	4.45	750	1.2	0.02	0.89	82
900	3B	16.43	20.86	4.43	750	1.25	0.02	0.89	81
1200	4A	16.24	20.58	4.56	750	1.25	0.02	0.91	84
1500	5A	16.61	20.94	4.34	750	1.27	0.02	0.87	80
1800	1B	16.66	20.99	4.33	750	1.27	0.02	0.87	79
								0.89	82

Table 7: NF 90 Distilled Water Permeability

Time	Vial	Mass of measuring vial [g]			Pressure	Retentate Flow		Flow	Flux
s		Initial	Final	Net	Average	Average	Variance	ml/min	$l/m^2.h$
0					375	1.2	0.02		
300	1A	16.69	23.70	7.02	375	1.32	0.02	1.40	129
600	2A	16.79	23.88	7.09	375	1.36	0.02	1.42	130
900	3B	16.47	23.41	6.94	375	1.42	0.02	1.39	127
1200	4A	16.28	23.25	6.97	375	1.43	0.02	1.39	128
1500	5A	16.62	23.57	6.95	375	1.45	0.02	1.39	127
1800	1B	16.67	23.50	6.83	375	1.45	0.02	1.37	125
								1.39	128
0					500	1.38	0.02		
330	1A	16.68	25.75	9.06	500	1.28	0.02	1.65	151
630	2A	16.79	25.14	8.35	500	1.32	0.02	1.67	153
930	3B	16.44	24.57	8.13	500	1.35	0.02	1.63	149
1230	4A	16.25	24.20	7.95	500	1.38	0.02	1.59	146
1530	5A	16.61	24.47	7.87	500	1.38	0.02	1.57	144
1830	1B	16.66	24.53	7.87	500	1.38	0.02	1.57	144
								1.61	148
0					625	1.34	0.02		
300	1A	16.68	25.41	8.72	625	1.34	0.02	1.74	160
600	2A	16.79	25.41	8.62	625	1.34	0.02	1.72	158
900	3B	16.44	25.06	8.62	625	1.34	0.02	1.72	158
1200	4A	16.25	24.70	8.45	625	1.34	0.02	1.69	155
1500	5A	16.60	25.12	8.51	625	1.34	0.02	1.70	156
1800	1B	16.66	25.18	8.52	625	1.34	0.02	1.70	156
								1.71	157
0					750	1.28	0.02		
300	1A	16.69	25.65	8.97	750	1.28	0.02	1.79	164
600	2A	16.79	25.72	8.94	750	1.29	0.02	1.79	164
900	3B	16.43	25.15	8.72	750	1.3	0.02	1.74	160
1200	4A	16.25	24.78	8.75	750	1.28	0.02	1.75	161
1500	5A	16.66	25.79	9.13	750	1.34	0.02	1.83	168
1800	1B	16.60	24.79	8.19	750	1.35	0.02	1.64	150
								1.74	160
0					875	1.33	0.03		
360	1A	16.68	27.35	10.67	875	1.33	0.03	1.78	163
660	2A	16.79	25.68	8.90	875	1.33	0.03	1.78	163
960	3B	16.43	25.23	8.79	875	1.33	0.03	1.76	161
1260	4A	16.25	25.01	8.76	875	1.33	0.03	1.75	161
1560	5A	16.60	25.42	8.82	875	1.33	0.03	1.76	162
1860	1B	16.66	25.26	8.60	875	1.33	0.03	1.72	158
								1.76	161

Table 8: NF 270 Distilled Water Permeability

Time	Vial	Mass of measuring vial [g]			Pressure	Retentate Flow		Flow	Flux
s		Initial	Final	Net	Average	Average	Variance	ml/min	$l/m^2.h$
0					375	1.05	0.02		
300	1A	16.68	20.22	3.53	375	1.07	0.02	0.71	41
600	2A	16.79	20.38	3.59	375	1.1	0.02	0.72	42
900	3B	16.43	19.99	3.55	375	1.08	0.02	0.71	41
1200	4A	16.25	19.82	3.57	375	1.15	0.02	0.71	42
1500	5A	16.60	20.23	3.63	375	1.17	0.02	0.73	42
1800	1B	16.66	20.26	3.61	375	1.17	0.02	0.72	42
								0.72	42
0					500	2.1	0.02		
300	1A	16.69	21.60	4.92	500	2	0.02	0.98	57
600	2A	16.79	21.71	4.92	500	2	0.02	0.98	57
900	3B	16.44	21.39	4.96	500	2.15	0.02	0.99	58
1200	4A	16.27	21.26	5.00	500	2.22	0.02	1.00	58
1500	5A	16.60	21.50	4.90	500	2.08	0.02	0.98	57
1800	1B	16.66	21.71	5.06	500	2.25	0.02	1.01	59
								0.99	58
0					625	2.15	0.02		
300	1A	16.68	22.67	5.98	625	2.2	0.02	1.20	70
600	2A	16.79	22.68	5.89	625	2.15	0.02	1.18	69
900	3B	16.43	22.43	6.00	625	2.2	0.02	1.20	70
1200	4A	16.25	22.20	5.95	625	2.2	0.02	1.19	69
1500	5A	16.60	22.50	5.90	625	2.24	0.02	1.18	69
1800	1B	16.66	22.54	5.88	625	2.25	0.02	1.18	69
								1.19	69
0					750	1.28	0.02		
300	1A	16.69	23.53	6.85	750	1.28	0.02	1.37	80
600	2A	16.78	23.46	6.67	750	1.29	0.02	1.33	78
900	3B	16.44	23.11	6.68	750	1.3	0.02	1.34	78
1200	4A	16.25	22.84	6.82	750	1.28	0.02	1.36	80
1500	5A	16.60	23.32	6.72	750	1.34	0.02	1.34	78
1800	1B	16.66	23.23	6.57	750	1.35	0.02	1.31	77
								1.34	78

Table 9: XLE Distilled Water Permeability

8.3.2 Synthetic Urine

Membrane	NF 270	Units	Time [min]			
Solution	Synthetic		0	15	30	45
Pressure	Average	kPa	1600	1600	1600	1600
	Variance	kPa	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.6	1.6
	Variance	l/min	0.02	0.02	0.02	0.02
Mass	Total	g	29.13	35.22	35.89	36.79
	Cell	g	14.02	14.01	13.83	14.00
	Net	g	15.11	21.20	22.06	22.78
Permeate Flow		ml/min		1.41	1.47	1.52
Flux		$l/m^2.h$		76.79	79.88	82.51

Table 10: Experimental data of synthetic urine run through NF 270 membrane

Substance	Reference				Net		
	[g]	[ml]	[g/mol]	[mol]	[g]	[ml]	[mol]
Na_2SO_4 anhydrous	9.2		142.0	0.06	9.206		0.06
NaH_2PO_4 anhydrous	8.4		120.0	0.07	8.409		0.07
NaCl	14.4		58.4	0.25	14.404		0.25
KCl	16.8		74.6	0.23	16.804		0.23
NH4Ac	38.4		77.1	0.50	38.4		0.50
NH_4OH solution (25% NH_3)		52	22.3	2.33		52	2.33
NH_4HCO_3	85.6		79.1	1.08	85.6		1.08
H_2O Distilled		4000	18.0	222.04		4000	222.04

Table 11: Synthetic urine composition

Membrane	NF 270	Units	Time [min]				
Solution	Synthetic		0	5	10	15	20
Pressure	Average	kPa	1000	1000	1000	1000	1000
	Variance	kPa	50	50	50	50	50
Retentate Flow	Reading	l/min	1.65	1.65	1.65	1.65	1.65
	Variance	l/min	0.02	0.02	0.02	0.02	0.02
Mass	Total	g		30.20	30.48	30.28	30.25
	Cell	g		16.68	16.79	16.43	16.26
	Net	g	0.00	13.52	13.69	13.84	13.99
Permeate Flow		ml/min		2.70	2.74	2.77	2.80
Flux		$l/m^2.h$		147	149	150	152

Table 12: Water flux through fouled NF 270 membrane

Variable	Dilution	Units	Time [min]											
			0			15			30			45		
			1	2	3	1	2	3	1	2	3	1	2	3
Total N	1:100	mg/l	3.3	3.3	3.3	2.4	2.4	2.4	2.6	2.6	2.6	2.3	2.3	2.3
	1:1	mg/l	3300	3300	3300	2400	2400	2400	2600	2600	2600	2300	2300	2300
		Rej				27.27%			21.21%			30.30%		
Potassium	1:1	mg/l	1876.05			998.44			1033.27			1086.77		
	1:1	mg/l	1876.05			998.44			1033.27			1086.77		
		Rej				46.78%			44.92%			42.07%		
Chloride	1:100	mg/l	25	25	25	22	22	22	20	20	20	19	19	19
	1:1	mg/l	2500	2500	2500	2200	2200	2200	2000	2000	2000	1900	1900	1900
		Rej				12.00%			20.00%			24.00%		
Phosphorous	1:1	mg/l	415.6			96.35			103.68			80.21		
	1:1	mg/l	415.6			96.35			103.68			80.21		
		Rej				76.82%			75.05%			80.70%		
Sodium	1:1	mg/l	2223.35			1319.19			1325.59			1394.08		
	1:1	mg/l	2223.35			1319.19			1325.59			1394.08		
		Rej				40.67%			40.38%			37.30%		
Ammonium	1:100	mg/l	16.9	16.9	16.9	7.7	8	8	10	10	10	13.4	13.6	13.6
	1:1	mg/l	1690	1690	1690	770	800	800	1000	1000	1000	1340	1360	1360
		Rej				53.25%			40.83%			19.92%		

Table 13: Chemical analysis for synthetic urine through NF 270 membrane

Membrane	NF 90	Units	Time [min]			
Solution	Synthetic		0	15	30	45
Pressure	Average	kPa	1600	1600	1600	1600
	Variance	kPa	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.62	1.64
	Variance	l/min	0.02	0.02	0.02	0.02
Mass	Total	g	28.84	15.52	15.75	15.65
	Cell	g	13.94	13.85	14.09	14.02
	Net	g	14.90	1.67	1.66	1.63
Permeate Flow		ml/min		0.11	0.11	0.11
Flux		$l/m^2.h$		6.06	6.01	5.89

Table 14: Experimental data of synthetic urine run through NF 90 membrane

Membrane	NF 90	Units	Time [min]				
Solution	Synthetic		0	5	10	15	20
Pressure	Average	kPa	1000	1000	1000	1000	1000
	Variance	kPa	50	50	50	50	50
Retentate Flow	Reading	l/min	1.5	1.52	1.58	1.6	1.62
	Variance	l/min	0.02	0.02	0.02	0.02	0.02
Mass	Total	g		22.20	22.18	21.88	21.60
	Cell	g		16.69	16.79	16.44	16.25
	Net	g	0.00	5.51	5.39	5.44	5.35
Permeate Flow		ml/min		1.10	1.08	1.09	1.07
Flux		$l/m^2.h$		59.92	58.58	59.06	58.14

Table 15: Water flux through fouled NF 90 membrane

Variable	Dilution	Units	Time											
			0			15			30			45		
			1	2	3	1	2	3	1	2	3	1	2	3
Total N	1:100	mg/l	2.8	2.8	2.8	2.6	2.5	2.4	2	2	2	1.5	1.3	1.4
	1:1	mg/l	2800	2800	2800	2600	2500	2400	2000	2000	2000	1500	1300	1400
		Rej				10.71%			28.57%			50.00%		
Potassium	1:1	mg/l	1818.96			548.24			593.83			557.1		
	1:1	mg/l	1818.96			548.24			593.83			557.1		
		Rej				69.86%			67.35%			69.37%		
Chloride	1:100	mg/l	35	35	35	9	10	10	15	15	15	11	12	13
	1:1	mg/l	3500	3500	3500	900	1000	1000	1500	1500	1500	1100	1200	1300
		Rej				72.38%			57.14%			65.71%		
Phosphorous	1:1	mg/l	422.06			135.35			147.78			181		
	1:1	mg/l	422.06			135.35			147.78			181		
		Rej				67.93%			64.99%			57.12%		
Sodium	1:1	mg/l	2160.99			693.88			734.67			695.38		
	1:1	mg/l	2160.99			693.88			734.67			695.38		
		Rej				67.89%			66.00%			67.82%		
Ammonium	1:100	mg/l	14.3	14.3	14.3	8.1	8.1	8.1	10	10	10	14.2	14.4	14.5
	1:1	mg/l	1430	1430	1430	810	810	810	1000	1000	1000	1420	1440	1450
		Rej				43.36%			30.07%			-0.47%		

Table 16: Chemical analysis for synthetic urine through NF 90 membrane

Membrane	XLE	Units	Time [min]			
Solution	Synthetic		0	30	60	75
Pressure	Average	kPa	1600	1600	2000	2500
	Variance	kPa	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.58	1.55
	Variance	l/min	0.02	0.02	0.02	0.02
Mass	Total	g	30.88	16.07	18.76	19.62
	Cell	g	13.81	13.93	14.09	13.98
	Net	g	17.07	2.14	4.68	5.64
Permeate Flow		ml/min		0.07	0.16	0.38
Flux		$l/m^2.h$		3.87	8.47	20.41

Table 17: Experimental data of synthetic urine run through XLE membrane

Membrane	XLE	Units	Time [min]				
Solution	Synthetic		0	5	10	15	20
Pressure	Average	kPa	1000	1000	1000	1000	1000
	Variance	kPa	50	50	50	50	50
Retentate Flow	Reading	l/min	1.64	1.6	1.62	1.62	1.62
	Variance	l/min	0.02	0.02	0.02	0.02	0.02
Mass	Total	g		21.12	21.13	20.76	20.59
	Cell	g		16.69	16.79	16.43	16.25
	Net	g	0.00	4.43	4.34	4.32	4.33
Permeate Flow		ml/min		0.89	0.87	0.86	0.87
Flux		$l/m^2.h$		48.17	47.16	46.98	47.10

Table 18: Water flux through fouled XLE membrane

Variable	Dilution	Units	Time											
			0			30			60			75		
			1	2	3	1	2	3	1	2	3	1	2	3
Total N	1:100	mg/l	3.1	3.1	3.1	1.1	1.1	1.1	1.6	1.6	1.6	2.2	2.3	2.3
	1:1	mg/l	3100	3100	3100	1100	1100	1100	1600	1600	1600	2200	2300	2300
		Rej				64.52%			48.39%			26.88%		
Potassium	1:1	mg/l	2025.01			580.94			399.89			248.59		
	1:1	mg/l	2025.01			580.94			399.89			248.59		
		Rej				71.31%			80.25%			87.72%		
Chloride	1:100	mg/l	19	19	20	16	16	16	11	15	15	22	23	23
	1:1	mg/l	1900	1900	2000	1600	1600	1600	1100	1500	1500	2200	2300	2300
		Rej				17.24%			29.31%			-17.24%		
Phosphorous	1:1	mg/l	483.51			186.64			127.49			204.99		
	1:1	mg/l	483.51			186.64			127.49			204.99		
		Rej				61.40%			73.63%			57.60%		
Sodium	1:1	mg/l	2287.87			728.12			485.09			305.32		
	1:1	mg/l	2287.87			728.12			485.09			305.32		
		Rej				68.17%			78.80%			86.65%		
Ammonium	1:100	mg/l	18.8	18.9	18.8	13.7	13.6	13.7	7.9	8	7.9	5.7	5.7	5.7
	1:1	mg/l	1880	1890	1880	1370	1360	1370	790	800	790	570	570	570
		Rej				27.43%			57.88%			69.73%		

Table 19: Chemical analysis for synthetic urine through XLE membrane

8.3.3 Stored Urine

Membrane	NF 270	Units	Time [min]			
Solution	Stored		0	15	30	45
Pressure	Average	kPa	1600	1600	1600	1600
	Variance	kPa	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.62	1.6
	Variance	l/min	0.02	0.02	0.02	0.02
Mass	Total	g	29.00	40.26	41.43	41.75
	Cell	g	14.02	14.02	13.83	14.00
	Net	g	14.99	26.24	27.59	27.75
Permeate Flow		ml/min		1.75	1.84	1.85
Flux		$l/m^2.h$		95.03	99.93	100.50

Table 20: Experimental data of stored urine run through NF 270 membrane

Membrane	NF 270	Units	Time [min]				
State	Dirty		0	5	10	15	20
Pressure	Average	kPa	1000	1000	1000	1000	1000
	Variance	kPa	50	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.6	1.6	1.6
	Variance	l/min	0.02	0.02	0.02	0.02	0.02
Mass	Total	g		29.89	30.20	30.00	29.83
	Cell	g		16.68	16.79	16.43	16.25
	Net	g	0.00	13.20	13.41	13.57	13.59
Permeate Flow		ml/min		2.64	2.68	2.71	2.72
Flux		$l/m^2.h$		143.46	145.70	147.43	147.63

Table 21: Water flux through fouled NF 270 membrane

Variable	Dilution	Units	Time											
			0			15			30			45		
			1	2	3	1	2	3	1	2	3	1	2	3
Total N	1:50	mg/l	62	62	62	33	33	33	21	21	21	35	35	35
	1:1	mg/l	3100	3100	3100	1650	1650	1650	1050	1050	1050	1750	1750	1750
		Rej				46.77%			66.13%			43.55%		
Potassium	1:1	mg/l	785.33			385.67			295.18			314.53		
	1:1	mg/l	785.33			385.67			295.18			314.53		
		Rej				50.89%			62.41%			59.95%		
Chloride	1:50	mg/l	20	20	20	17	18	18	10	12	12	16	15	15
	1:1	mg/l	1000	1000	1000	850	900	900	500	600	600	800	750	750
		Rej				11.67%			43.33%			23.33%		
Phosphorous	1:1	mg/l	1237.39			696.17			1232.78			800.49		
	1:1	mg/l	1237.39			696.17			1232.78			800.49		
		Rej				43.74%			0.37%			35.31%		
Sodium	1:1	mg/l	2009.09			1107.4			935.02			1020.96		
	1:1	mg/l	2009.09			1107.4			935.02			1020.96		
		Rej				44.88%			53.46%			49.18%		
Ammonium	1:50	mg/l	50	50	50	22	22	22	14	14	14	33	33	33
	1:1	mg/l	2500	2500	2500	1100	1100	1100	700	700	700	1650	1650	1650
		Rej				56.00%			72.00%			34.00%		

Table 22: Chemical analysis for stored urine through NF 270 membrane

Membrane	NF 90	Units	Time [min]			
Solution	Stored		0	15	30	45
Pressure	Average	kPa	1600	1600	1600	1600
	Variance	kPa	50	50	50	50
Retentate Flow	Reading	l/min	1.62	1.6	1.62	1.64
	Variance	l/min	0.02	0.02	0.02	0.02
Mass	Total	g	35.46	15.92	16.14	16.09
	Cell	g	13.94	13.89	14.09	14.02
	Net	g	21.52	2.04	2.05	2.07
Permeate Flow		ml/min		0.14	0.14	0.14
Flux		$l/m^2.h$		7.37	7.43	7.51

Table 23: Experimental data of stored urine run through NF 90 membrane

Membrane	NF 90	Units	Time [min]				
State	Dirty		0	5	10	15	20
Pressure	Average	kPa	1000	1000	1000	1000	1000
	Variance	kPa	50	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.6	1.6	1.6
	Variance	l/min	0.02	0.02	0.02	0.02	0.02
Mass	Total	g		22.29	22.32	21.87	21.72
	Cell	g		16.69	16.79	16.44	16.25
	Net	g	0.00	5.60	5.53	5.43	5.47
Permeate Flow		ml/min		1.12	1.11	1.09	1.09
Flux		$l/m^2.h$		60.88	60.05	59.01	59.42

Table 24: Water flux through fouled NF 90 membrane

Variable	Dilution	Units	Time											
			0			15			30			45		
			1	2	3	1	2	3	1	2	3	1	2	3
Total N	1:50	mg/l	63	63	63	30	30	30	30	30	30	41	41	41
	1:1	mg/l	3150	3150	3150	1500	1500	1500	1500	1500	1500	2050	2050	2050
		Rej				52.38%			52.38%			34.92%		
Potassium	1:1	mg/l	834.14			235.07			195.85			84.18		
	1:1	mg/l	834.14			235.07			195.85			84.18		
		Rej				71.82%			76.52%			89.91%		
Chloride	1:50	mg/l	21	21	21	8	8	8	6	6	7	7	7	7
	1:1	mg/l	1050	1050	1050	400	400	400	300	300	350	350	350	350
		Rej				61.90%			69.84%			66.67%		
Phosphorous	1:1	mg/l	1841.62			1719.32			1414.42			1181.98		
	1:1	mg/l	1841.62			1719.32			1414.42			1181.98		
		Rej				6.64%			23.20%			35.82%		
Sodium	1:1	mg/l	2261.84			690.07			700			245.04		
	1:1	mg/l	2261.84			690.07			700			245.04		
		Rej				69.49%			69.05%			89.17%		
Ammonium	1:50	mg/l	60	60	60	14	14	14	12	12	12	17	17	17
	1:1	mg/l	3000	3000	3000	700	700	700	600	600	600	850	850	850
		Rej				76.67%			80.00%			71.67%		

Table 25: Chemical analysis for stored urine through NF 90 membrane

Membrane	XLE	Units	Time [min]			
Solution	Stored		0	20	40	60
Pressure	Average	kPa	1600	1600	2000	2500
	Variance	kPa	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.6	1.6
	Variance	l/min	0.02	0.02	0.02	0.02
Mass	Total	g	34.33	17.67	22.73	29.88
	Cell	g	13.81	13.93	14.11	13.99
	Net	g	20.52	3.73	8.63	15.89
Permeate Flow		ml/min		0.19	0.43	0.79
Flux		$l/m^2.h$		10.140	23.434	43.159

Table 26: Experimental data of stored urine run through XLE membrane

Membrane	XLE	Units	Time [min]				
State	Dirty		0	5	10	15	20
Pressure	Average	kPa	1000	1000	1000	1000	1000
	Variance	kPa	50	50	50	50	50
Retentate Flow	Reading	l/min	1.6	1.6	1.6	1.6	1.6
	Variance	l/min	0.02	0.02	0.02	0.02	0.02
Mass	Total	g		22.68	22.66	22.37	22.04
	Cell	g		16.70	16.79	16.44	16.26
	Net	g	0.00	5.98	5.87	5.93	5.78
Permeate Flow		ml/min		1.20	1.17	1.19	1.16
Flux		$l/m^2.h$		64.94	63.73	64.47	62.84

Table 27: Water flux through fouled XLE membrane

Variable	Dilution	Units	Time											
			0			20			40			60		
			1	2	3	1	2	3	1	2	3	1	2	3
Total N	1:50	mg/l	64	64	70	28	28	29	38	39	40	23	18	17
		mg/l	3200	3200	3500	1400	1400	1450	1900	1950	2000	1150	900	850
		Rej				57.07%			40.91%			70.71%		
Potassium	1:1	mg/l	750.23			106.27			26.3			0.77		
		mg/l	750.23			106.27			26.3			0.77		
		Rej				85.84%			96.49%			99.90%		
Chloride	1:50	mg/l	25	25	26	22	22	22	17	17	17	18	18	18
		mg/l	1250	1250	1300	1100	1100	1100	850	850	850	900	900	900
		Rej				13.16%			32.89%			28.95%		
Phosphorous	1:1	mg/l	1771.01			898.62			1212.13			1107.91		
		mg/l	1771.01			898.62			1212.13			1107.91		
		Rej				49.26%			31.56%			37.44%		
Sodium	1:1	mg/l	2216.22			351.48			152.63			78.84		
		mg/l	2216.22			351.48			152.63			78.84		
		Rej				84.14%			93.11%			96.44%		
Ammonium	1:50	mg/l	50	50	50	13	13	13	9	9	9	13	13	13
		mg/l	2500	2500	2500	650	650	650	450	450	450	650	650	650
		Rej				74.00%			82.00%			74.00%		

Table 28: Chemical analysis for stored urine through XLE membrane