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**WATER ENGINEERING**

Presented by

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**Evolution of Macronutrients Content during Thermal  
Drying of Faecal Sludge**

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## **Dedication**

I dedicate this work to my family and friends who always believed in me and encouraged me throughout my studies.

## **Biographical Sketch**

Lone Morubisi completed his bachelor's degree BSc Soil and Water Conservation Engineering from Botswana College of Agriculture now Botswana University of Agriculture and Natural Resources in 2012. He previously worked as a mathematics teacher in the Ministry of Education then joined University of Botswana as a teaching assistant before joining Botswana International University of Science and Technology as teaching and research assistant before joining PAUWES for master's degree. In his study at PAUWES he was the president of student Community of Practice, where he was responsible for leading and mobilising students for various activities.

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## Abstract

Research has shown that an increase in urbanization is an increase in urban population, which relate to increase in waste production. Sub Saharan African countries are experiencing a lot of urbanization and most of the population is not connected to waste water treatment systems but instead using onsite sanitation systems. Statistics shows that 80% of the faecal sludge produced as waste is left untreated and not converted into any productive use. This study focused on the evolution of faecal sludge macronutrients due to thermal drying.

Faecal sludge was thermally dried using an oven at drying temperatures of 200°C, 100°C and 50°C to obtain moisture content of 50%, 25% and 0% for each drying temperature. Faecal sludge was tested for nutrients contents of nitrogen, ammonium, nitrite, nitrate, phosphate, orthophosphate and potassium at each condition. Thermal drying and loss of moisture content proved to have an effect on nutrient content of faecal sludge. It shows that with varying of drying temperature and loss of moisture content nitrogen mineralises, and changes form. Nitrogen fractions ammonium, nitrite and nitrate proved to be more volatile to change in drying temperatures and loss of moisture content. Phosphate and orthophosphate were also affected by varying of drying temperatures and loss of moisture as concentration kept changing at different conditions due speciation of phosphorus. Potassium was not significantly affected by change in drying temperatures and loss of moisture content because it is strongly bond to the dry matter.

*Keywords:* faecal sludge, thermal drying, drying temperature, moisture content, nutrient concentration.

## List of Abbreviations and Acronyms

FS	: Faecal Sludge
VIP	: Ventilated Improved Pit
MAPET	: Manual Pit Emptying Technology
MDP	: Motorized Diaphragm Pumps
DRE	: Deep Row Entrenchment
N	: Nitrogen
NH <sub>4</sub> <sup>+</sup>	: Ammonium
NO <sub>2</sub> <sup>-</sup>	: Nitrite
NO <sub>3</sub> <sup>-</sup>	: Nitrate
PO <sub>3</sub> <sup>4-</sup>	: Phosphate
K	: Potassium



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# 1. Introduction

## 1.1 Background of the Study

The increasing demands for food across the globe in the midst of concerns of over sustainable and environmentally friendly production methods have stimulated several global engagements and debates. While synthetic fertilizer-based systems for crop production has dominated commercial and subsistence production activities particularly in rural and urban Africa, recent advancement in research and technology has demonstrated significant potentials of the viability of commercialising organic sources of nutrition for production (Fließbach et al., 2007; Singh & Agrawal, 2008). While literature has been dominated by a plethora of evidence on the use of organic materials derived from animal and plants waste, the potential of human faecal sludge conversion to crop nutrient source has recently been widely established.

With the Sub-Saharan African region has been rapidly urbanised, the evaluation of the potential of human fecal sludge in this context is even far more justified than elsewhere (Kihara et al., 2016; Zake et al., 2010), compared to European countries which statistics show that by the year 2005 an average of two thirds of the population was already connected to waste water treatment systems (World Health Organization, 2009). As noted by researches on the social dimensions of urbanisation, an increasing rate of population comes with an increasing generation of waste, dominant of which includes human excreta (Oelofse & Nahman, 2013; Scarlat et al., 2015). Human excreta accumulated through on-site collection and storage processes, in its raw or semidigested, semi-solid state and treated with water, is herein conceptualised as sludge (Olden, 2016). The nature of the sanitation facilities of most African housing systems allows to accumulate this waste and potentially convert it into a significant nutrient source for plants. However, available evidence suggests that about 80% of the faecal sludge produced from most households in Sub Saharan African ends up untreated and unconverted into productive uses (Moya et al., 2019; Schoeman et al., 2017; Snyman, 2007).

In Botswana, with an increasing population and an urbanization rate of 70.17% in 2019, evidence has shown that limited treatment of faecal sludge has led to

increasing rates of underground water contamination (Mosha, 2020). The conversion process of faecal sludge into organic fertiliser has been obscured due to limited technological ease and cost-effectiveness of the process, insufficient knowledge of the physiochemical properties of the sludge and their application for plant health and growth. In South Africa, increase in urban population has led to mushrooming of either informal settlements where sanitation facilities are overloaded or non-existent. To address the problem of sanitation South African cities are providing on site sanitation systems, but management is overstretched as some onsite sanitation systems are full or less maintained which poses a health hazard. However, the process of thermal drying has been described as one that does not significantly alter the nutrient content and the calorific values of the final product. Preliminary evidence on the ability of the drying process to produce organic biosolids that are rich in nutrients that can sustain crop production has been established extensively in the works of Septien et al (2019). It is against this background that this study seeks to advance their findings by investigating further, the final nutritional composition of sludge treated through thermal drying and assess its suitability for crop cultivation

## 1.2 Problem Statement

It is well known that faecal sludge is suitable for reuse in agriculture. However, of all the diverse treatment processes for the treatment of faecal sludge, there is insignificant evidence on how the nutrient composition vary with thermal drying, and if the sludge nutrient quality is preserved after this treatment process. Several studies have tried to establish the chemical composition and suitability of faecal sludge for crop production (see Krueger et al., 2020; Ofori-Amanfo, 2018; Septien et al., 2020) but the evidence on the nutrient composition is inconclusive. The effects of the diverse treatment options on the final composition of the sludge, which will help determine the most suitable treatment option for reuse as crop fertilizer, is also debated.

Attempts to study the context of sludge and its usage have taken a descriptive approach to profiling existing practices of sludge management. Others have delved into the policies that regulate the management of sludge. A couple of studies

examined the diverse treatment options for sludge, but were limited in evidence on specific chemical composition of the product and how they conform to acceptable levels for crop production. This study therefore is intended to contribute towards establishing evidence on the nutrient suitability of sludge for crop fertilizer after thermal drying and assess its suitability for use as fertilizer for crop production. The evidence generated will have significant implications on agricultural practice

### 1.3 Significance of the Research

A study that seeks to better understand the conversion of faecal sludge into organic fertilizer will contribute towards enhancing the treatment of waste that has plagued most African countries, and the development of food production systems in an environmentally friendly way. With the Sustainable Development Goal (SDG) 6 focusing on clean water and sanitation, this study will contribute towards empirical evidence for engineering and policy to advance this goal. Its output will further be a significant contributing factor to enhancing food production and productivity, and ultimately the attainment of SDG 2 and Zero hunger (Willis, 2018).

The findings of this study will further serve as a contribution to science in the field of sludge management, water treatment and crop production. It has the potential of stimulating further intellectual debate into other aspects of the management of sludge and other inter-sectoral linkages.

The contribution of this study to the local development can be significant. It can have positive effects on the local waste management and crop production, which is expected to rise general living standards of the people.

### 1.4 Study Objectives

The main aim objective of this study was to study further the investigation of Septien et al. (2020). There was a need to understand how faecal sludge macronutrients (NPK) behave after been oven dried at different temperatures to obtain different moisture contents.



The specific objectives of the study including the following processes to dry and test for nutrients:

1. Conduct thermal drying of faecal sludge obtained from VIP latrines under different drying temperatures of 200°C, 100°C and 50°C.
2. Dry the faecal sludge to different moisture contents of 50%, 25% and 0%.
3. Tests the nutrients concentrations of FS at different moisture contents under different drying temperatures.

## 1.5 Work Scope

The study was to test macronutrients concentrations of faecal sludge after being dried at different moisture contents under different drying temperatures. The faecal sludge was collected from VIP latrines in the boundaries of eThekweni municipality.

## 2. Literature Review

### 2.1 Onsite and off-site sanitation system

According to the world health organization, sanitation regards all conditions that affect health. On-site sanitation system refers to a system which treats its waste on site, and as such, waste does not have to be transported in pipes over long distance to ensure treatment. On-site sanitation systems are usually adopted in areas where there is an absence of piped sewer system. Such solutions are regarded as temporary measures in the place of a fully decentralized system. In on-site systems, sewage can be treated either: on-site, or offsite, and in both cases, the sewage is treated near the source. On-site treatment of FS employs a septic tank, and a soak pit to treat sludge using the process of anaerobic treatment, and sewage penetration through the ground. Such a process is effective provided with a good soil permeability. In off-site sanitation systems, excreta and waste water are collected and conveyed to be treated away from their site of generation (FU Berlin, 2021).

#### 2.1.1 Ventilated Improved Pits (VIP) Latrine

VIP latrine technology is based on improvements made from the unventilated traditional pit latrines that are associated with the disadvantages of producing bad smell, as well as pose significant risks to human wellbeing and safety (UNICEF, 2016). VIP latrines minimize the disadvantages of a traditional pit latrine by offering a technology which caters for better ventilation through the use of a vent pipe, prevents movements of flies and insects in and out of the pit, as well as accounting for improved structural capabilities. VIP latrines can be single pit or double pit. In a double pit system, a temporary superstructure over a part of the pit is demolished whenever FS collection is required. FS characteristics have been observed to vary widely across individual VIP latrines. A study by authors (Bakare et al, 2012; Zuma et al., 2015; Getahun et al., 2020), investigated FS characteristics amongst VIP latrines and observed no two FS have the same characteristics.

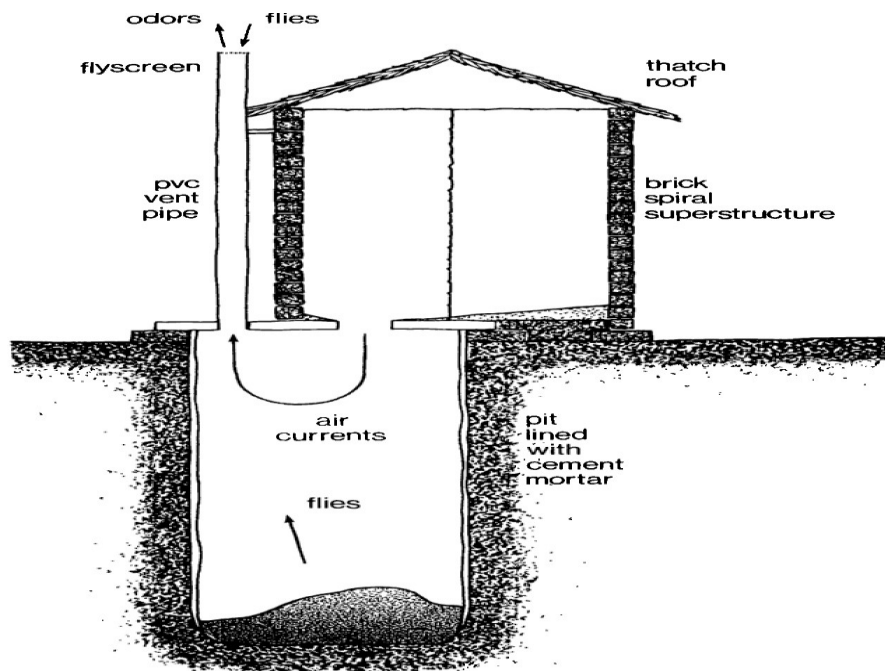


Figure 1: Cross section of a VIP latrine, Source: semanticscholar.org

## 2.1.2 Collection

The process of collecting, storing and treating a mixture of excreta and blackwater results in the formation of a substance in the slurry or semi-solid state, usually raw and undigested, referred to a faecal sludge

### 2.1.2.1 Manual collection

Manual collection of faecal sludge can be carried out using a cartridge containment device, or by a direct lifting technique. A cartridge container device, commonly called the uniloo, uses a replaceable storage tank into which urine is diverted. In the direct lifting method, a longhandled shovel is used to fill bucket which is hoisted vertically to the ground and manually empties into a tank fitted on to a cart or any other type of mechanical carrier.

### 2.1.2.2 Mechanical pumping

Mechanical pumping means of collection of FS using one of the four following devices; the sludge gulper, the diaphragm pumps, the nibbler and the manual pit emptying technology .

The sludge gulper is a simple design technology built using locally available PVC materials as riser, and two non-return butterfly valves made of stainless steel. The operational mechanism of the valves is such that one valve, called the foot valve is located at the bottom of riser and remains fixed in place, while the plunger valve pulls up and down, a T-handle, to open and close the two valves connected in series in order to lift FS through the riser pipe. In such a system, a strainer fitted at the bottom of the pipe prevents non-biodegradable waste from entering the riser that can lead to a blockage of the pump.

A manually operated diaphragm pump for FS extraction is a simple low-cost pump capable of extracting FS with low viscosity and little amount of non-biodegradable materials. In its configuration, a disc, shaped body is clamped to a rubber membrane, which is called the diaphragm.

A nibbler uses a continuous rotary action of a displacement pump to extract medium viscosity FS. Such a system has the capability of collecting medium viscosity sludge. This system has a configuration with a roller chain loop enclosed in a PVC pipe which can be inserted into an access hole of the containment structure, eliminating the need for breakage of any part in order to access the FS.

Manual Pit Emptying Technology (MAPET) was introduced in Tanzania in 1992. The system is made of a high-powered vacuum pump and a system a 200liter tank mounted on a pushcart. MAPET system is regarded as most technically advanced amongst the manual systems available, capable of pumping FS from a depth of 3metres, at a rate of between 10 to 40L/min subject to the viscosity of FS and the depth of the pit.

### 2.1.2.3 Fully mechanized collection

Fully mechanized FS collection systems can be electrically powered, or powered using a fuel or a pneumatic system. A number of fully mechanized systems exist for the extraction and collection of FS, some of the available systems employed include; motorized diaphragm pumps, pit screw auger and e.t.c

A motorized diaphragm pumps (MDP) suitably handles liquid FS with and solid particles with sizes ranging between 40mm to 60mm. such a system is capable of producing a flow speed between 300 to 330L/min. An MPD is suitable in applications with net head of pumping up to 15mm, and in cases with a variable depth across the pit.

A trash pump technology is employed in applications where FS is very liquid, and the solid particles have mass of between 30 to 30mm. This technology is capable of achieving maximum flow rate of 1200L/min, while pumping FS up to a height between 25 to 35m.

### 2.1.3 Transportation

Transportation modes of collected FS can be classified into Manual and motorized methods. Manual transportation methods include the use of carts general transportation of materials or a custom-made cart designed for the transportation of FS. In a motorized system, a large capacity of FS can be hauled and transported at a relatively faster speed. the two main recognised means of motorised transportation systems include: the use of a motorized tricycle, and the use of a motorised transport equipment such as a pick-up truck. In motorized tricycles, FS is transported in either a drum or a tank fitted at the back of the tricycle (O’Riordan, 2009). Researchers have identified load carrying capacities of up to 1000kg associated with certain models of this technology. Pick-up trucks can carry loads with capacities ranging from 2000kg to 5000kg. The cost-affordability of pick-up trucks limits its use to mostly large-scale applications.

#### 2.1.4 Treatment of FS

Conversion of FS into useful product can require several processes depending on the final goal of the treatment process. Typical treatment goals for FS treatment technologies include; dewatering, liquid-solid separation, pathogen reduction, composition stabilization, to obtain an end product (nutrients recovery).

Dewatering involves reducing liquid content in a FS. Dewatering of FS is affected by the age of the sludge as well as its on-site storage duration, it has been observed that raw (fresh) sludge is relatively more difficult to dewater than an older FS. The common methods of dewatering FS include evaporation (evapotranspiration), gravity settling, and the use of filter drying beds.

The large number of microorganisms contained in an untreated FS pose a health risk leading from an unsafe exposure. FS are treated to reduce the number of pathogens contained to ensure a safe end use or disposal into the environment. A stabilized FS contains a high proportion of carbon – based molecules that are not readily degradable. Stabilization of FS is significant to reduce the amount of oxygen demand from the organic matter contained during aerobic respiration. Disposal of an unstabilized FS depletes the amount of oxygen on surface waters. Some of the common treatment technologies include: co-composting of faecal sludge ,co-treatment in stabilization ponds and deep row entrenchment.

Co-composting involves the decomposition of organic matter piled into heaps or in wall enclosures by biological micro-organism, under a controlled aerobic condition (Cofie et al., 2009). Such a process produces as an end product, stabilized organic matter which can be used as a soil conditioner, and in some cases, organic fertilizer is produced as an end product. Composting methods can be grouped into: open, and closed composting with open composting requiring a relatively larger space to accomplish. In such a treatment process, the ratio of Carbon to Nitrogen, the Oxygen concentration, moisture content, and the size of particles, are factors usually controlled to ensure best practice. Composting process takes a minimum of six to eight weeks, with dewatered FS most suitable to undergo this process.

Stabilization ponds are used in mainly low-income countries to treat municipal wastewater (Strauss et al., 2000). Treating wastewater in such a system involves the

use of a series combination of three separate kinds of ponds varying in depth and retention time: anaerobic, facultative and aerobic maturation ponds.

Deep row entrenchment (DRE) is effective treatment technology for wastewater in areas with adequate land availability. This technology simultaneously treats the FS at the same time of providing an important end use option. DRE system fills and covers FS in deep trenches dug, while planted trees on the cover soil benefit directly from the decomposed organic nutrients.

## 2.2 Faecal Sludge characteristics

### 2.2.1 pH

The pH characteristics of a FS represent the hydrogen-ion composition of the sludge. Most biological organisms in FS have been identified to suitably exist within a typical pH range of 6 – 9, with FS containing extreme proportions of Hydrogen ion have been cited to undergo biological treatment process with greater levels of difficulties, as concentrations outside this range significantly inhibit the process of anaerobic digestion as well as the production of methane.

### 2.2.2 Nutrients

Excreta has been studied to contain nutrients originating mainly from food consumption. Faeces have been identified to contain between 80 to 90%, 20 to 50%, and 10 to 20% of the Nitrogen (N), Phosphorus (P) and potassium (K) respectively. Excreted urine contains 80 to 90% of N, 50 to 65% P, and 50 to 80% K. Raw FS contains ammonia ( $\text{NH}_3$ ) produced from the urine by the chemical deamination of organic nitrogen (Guyton, 1992, Berger 1960, Schouw et al., 2002). Nitrogen present in FS can be found in combination with hydrogen molecules in the form of ammonia ( $\text{NH}_3$ ) or ammonium ( $\text{NH}_4^+$ ) or with oxygen molecules as, nitrite ( $\text{NO}_2^-$ )

or nitrate ( $\text{NO}_3^-$ ). Nitrogen in FS is also found in the forms of amino acids and amines as organic nitrogen. The total concentration of Nitrogen in FS depends on the type of FS, the storage duration, the pH content, and the presence of oxygen. Phosphorous is present in a FS in the form of: Phosphate, orthophosphoric acid or as organic phosphates such as nucleic acid.

### 2.2.3 Total solids

Total solids concentration in a FS represents the cumulative amount of organic and inorganic matter contained. The fraction of the total solid matter ignited and burned at a temperature of  $500^\circ\text{C}$  are identified as and inorganic solid. Fixed solids represent remaining fraction of the amount of solid after an ignition and burning process. This characteristic of a FS quantifies the amount of substance remaining after treatment of the FS by a drying process, within a typical temperature range of  $103^\circ\text{C}$  to  $105^\circ\text{C}$ . In a FS treatment process, dissolved solids are particles that pass through a filter, whereas suspended particles are those that cannot pass through (Forster-Carneiro et al., 2008).

### 2.2.4 Oxygen demand

The oxygen demand is classified in two: biochemical, and chemical oxygen demand. Discharged FS can significantly deplete the oxygen content on the surface of water bodies. Bio chemical Oxygen demand measures the oxygen utilization of micro-organisms in the degradation of organic matter. When a dichromate is used in the chemical oxidation of an organic matter, the oxygen equivalent of the organic matter oxidized give a representation of the chemical oxygen demand (Heinss et al., 1999).



### 2.2.5 Grit and sand

The presence of grit and sand provides a major influence in determining the required size and the rate of filling of a treatment or storage tanks. Grit and sand is present in FS is common in unlined pit latrines. Flooding, utensils and vegetables washing and house cleansing are common sources of grit and sand in FS. The sand and grit contents can be reduced in FS with the use of sand traps or mesh entry points of pipes and sewer (Getahun et al., 2020a).

### 2.2.5 Oil and grease

Faecal sludge contains fats, oil and grease obtained from a variety of sources. The common sources of fats contained in FS include: meats, nuts and seeds. Sources of oil and grease include lubricating oil and kerosene. The individual and relative concentrations of oil and gas contained in a FS are determined using solvent extraction methods (Krueger et al., 2021).

## 2.3 Thermal drying of faecal sludge

Drying is a technological operation in which thermal energy is provided to sludge to evaporate water. The most common sludge drying technology is thermal drying (Colón et al., 2017). The process of drying sludge reduces volume of the product, making its storage, transportation, packaging and retail easier. Sludge drying also inactivates pathogens and leads to a sanitized final product in pellets in relatively short time, with low odours and good handling characteristics. The thermal drying technologies used in FSM can be classified into direct (convection), and Indirect (contact).

A direct thermal drying involves passing hot air or gases through a dewatered FS, or transporting FS through a chamber filled with hot air and gases. Drying in this case is achieved by mixing of the gas with the FS.

Indirect thermal drying is achieved with the use of a heat exchanger as means of transferring heat through convection carried in the form of a steam or oil. In this case, there is no physical contact between the FS and the heat carrying medium. (Rontelntap et al., 2014)

## Benefits of thermal drying

Thermal drying significantly reduces the volume as well as the pathogen content of FS. The Dried FS obtained has limited handling requirements and can be marketed easily. Dried FS can be used in agriculture as soil nutrients or as crop fertilizer. (Strande et al., 2014)

### 2.3.2 Limitations

There are several limitations to thermal drying of faecal sludge. Huge financial cost required to set up a thermal drying plant, drying process that require significant amount of energy supply are some of the limitations. Operation is also threatened by the risk of fire and explosion since there is presence of hot gases and dust particles in the drying system and also high maintenance requirement (Strande et al., 2014).

## 2.4 Effects of thermal drying on faecal sludge

Septien et al. (2020) observed that the total nitrogen content remained constant during drying, but the ammonium, nitrates and nitrites concentration decreased. The authors assumed that this could be result of changes of the chemical form of nitrogen

during drying. Drying does not affect nutrient concentration as concentration between raw sludge and dried sludge does not significantly change but only ammonium and nitrates are affected by thermal drying due to volatilization by increased temperatures ( Makununika, 2016). However, Afolabi et al. (2015) state that during drying, the form of nitrogen as ammonia is released as gas. The organic nitrogen partly mineralizes during drying, thus becoming more available for plant intake. Afolabi et al. (2015) continued to say at 160°C nitrogen compounds are hydrolysed and decomposed to amino acids, organic-N and ammonium compounds beyond this temperature. At 180°C there is deamination and hydrolysis of amino acids into short-chain volatile fatty acids, ammonia and carbon IV oxide occur. At 200°C, there is increasing concentration of ammonia recovered from both Sewage Sludge and Faecal Sludge as temperature increases.

## 2.5 Use of faecal sludge in agriculture

Sludge's potential as an agricultural supplement has been proven, this being because the addition of sludge to soil provides a better balance of nutrients while reducing their loss. Indeed, sludge is capable of immobilizing large quantities of nutrients and desirable heavy metals (Lima et al., 2012). Nutrients for plants are contained in the bio-solids resulting from sludge treatment. The major nutrients are nitrogen (N), phosphorus (P) and potassium (K). Important chemical characteristics to reuse the sludge in agriculture are the pH, soluble salts, plant macro and micro nutrients, trace elements and organic compounds (Colón et al., 2017). The agricultural applications of faecal sludge include soil structure improvement, soil buffer and soil amendment (Lamastra et al., 2018). Despite the recommendations to use faecal sludge in agriculture as fertilizer, some farmers are still reluctant to use FS as some international standard-setting bodies are still lacking behind to set standard for FS. This hinders most farmers as they are not comfortable with using sludge because they want their product to be recognised internationally for exports (Moya et al., 2019).

### 3. Materials and Methods

This chapter describes experiments and equipment used on this study. Feedstock used for the experiment is described in the first subtopic, then the experimental procedure and lastly different equipment used.

#### 3.1 Feedstock

Feedstock used for the experiments was faecal sludge collected from ventilated improved pit (VIP) latrine toilets. The sludge was collected from two different VIP toilets in eThekweni municipality boundaries, South Africa. Before use, domestic waste material was removed from the sludge such as sanitary pads, condoms, baby pampers and polythene bags and then mixed to achieve one homogenous sample of sludge. To preserve the sludge, it was stored in a cold room at 4°C when not in use and before been dried. Table 1 shows the characteristics of the two VIP sludges that were mixed to carry out the experiments. Figure 2 shows mixed sludges after trash removal.

Table 1 : faecal sludge characteristics

<b>Characteristics</b>	<b>Name</b>	
Untreated Sludge Characteristics	VIP1	VIP2
Sampling date	24/02/2021	24/02/2021
Moisture Content (% mean)	80.285	73.141
Total Solids (% mean)	20.813	26.772
Sludge Colour	Black/ Brown	Black/ Brown
Sludge Odour	Strong	Strong
Trash quantification	Large quantities	Large quantities



Figure 2: mixed sample of VIP1 and VIP2

## 3.2 Equipment Used

This chapter will give brief explanation of equipment used to carry out the experimental procedure.

### 3.2.1 Oven

Laboratory oven is a standard equipment found at WASH R&D Centre at the University of KwaZulu-Natal. Laboratory oven provide controlled uniform temperature for sterilising, evaporating and baking samples and equipment in the lab. The oven is connected to an extractor to control smoke pollution in the lab when heating samples. Figure 3 is a picture of the laboratory oven used for thermal drying.



Figure 3: laboratory oven used for drying under different temperatures

### 3.2.2 Moisture balance

Thermal balance is an equipment used to determine the moisture content in small samples of various substances, by measuring the change of weight due to water evaporation during radiative drying. This method is applicable for all types of sludge: liquid, slurry, semi-solid and solid. Before operation the equipment should be levelled on the surface placed on and should be placed where there are no environmental interferences such as air draught, vibration and direct sunlight. For moisture balance testing method, the equipment should be set to a temperature of 105°C. The equipment must be switched on for about 30 minutes before any tests can be done to allow self-calibration. Figure 4 is a picture of the laboratory thermal balance.



Figure 4: thermal balance machine used for measuring moisture content

### 3.2.3 Spectroquant

Spectroquant is an equipment used to measure concentration of solutes by measuring the amount of light absorbed by the sample solution. Before measuring a given chemical compound, a method of what is been measured must be selected by reading the barcode from the barcode cell that for comes with testing kits for preparing samples or the reactor cell or method chosen from the spectroquant screen. Before conducting any test, it is important to switch the equipment for sufficient time to allow self-calibration. Figure 5 shows a picture of a spectroquant with a barcode cell.



Figure 5: spectroquant machine used to measure nutrient concentration in prepared solutions.

### 3.3 Experimental Procedure

This chapter explains experiments carried out for this project. It includes experiments from oven drying of sludge to chemical analysis of sludge by the spectroquant.

#### 3.3.1 Oven drying

Samples were dried in the oven at different temperatures with varying residence time to achieve different moisture content. To determine varying times for different moisture content at different temperatures, drying curves had to be determined



### 3.3.1.1 Drying curves

First, the oven was set at specific temperatures of 50°C, 100°C and 200°C, One drying temperature was used at a time. Wet faecal sludge was taken out of the cold room to achieve ambient temperature. The sludge was then stirred using a stirring rod to get a homogenous sample then dished an evenly spread in an oven tray and placed into the oven, time of starting the process was recorded. In every fifteen minutes, the sludge was stirred to avoid forming surface crust that will hinder uniform drying, then every thirty minutes a sample was taken from the drying sample to the thermal balance to measure the moisture content and then recorded; this process was repeated until close to zero moisture content was recorded. The sludge was then left for absolute drying and taken out the following day at the same time it was placed in the Oven. The sample taken out after twenty-four hours were kept as completely dried sample (moisture content at zero percent). After the different moisture content were recorded, the results were then plotted into graphs using Microsoft excel to determine different times needed for 50% and 25% moisture content. The same process was repeated for temperatures of 100°C and 200°C. Figure 6 shows the sample been stirred during drying.



Figure 6: sample stirring in the Oven during drying

### 3.3.1.2 Drying at different temperatures

After determining drying curves, the same process was repeated by taking out the sample from the oven at certain determined times to get the desired moisture content.

### 3.3.2 Moisture Balance

For wet faecal sludge which was used for initial moisture content, the samples were taken out of the cold room for about 30 minutes to an hour to reach room temperature before the experiment can commence. The samples were then stirred using a stirring rod to get a homogenous sample, after the machine was well set and finished self-calibration a sample of about 1 to 3 grams was placed and evenly spread on the weighing boat then heated in the thermal balance to get the moisture content. The same process was done on the samples dried in the oven. Standard Operating Procedure (SOP) for thermal balance was adopted from (Ferré et al., 2021).

### 3.3.3 Spectroquant tests

Spectroquant test was used for primary macronutrients experiments. The primary macronutrients (N, P, K) were all tested using the spectroquant. Before the experiments can be done, a solution of sample had to be prepared. The solution was prepared by blending two grams of sample in one litre of distilled water; the sample solution was stored in the cold room if not in use. Before use, the sample was taken out of the cold room for about 30 minutes to an hour to achieve ambient temperature. The spectroquant equipment was switched on 30 minutes before any experiment was conducted to achieve self-calibration. The steps for primary macronutrients test were followed as stipulated in the SOPs (Ferré et al., 2021) and the tests were done in triplicates. A picture of diluted samples is shown in figure 7.



Figure 7: diluted samples stored in 1-litre bottles

### 3.3 Data Treatment

After getting spectroquant results in mg/l, equations 1 and 2 were used to convert and analyse the results

$$\text{wet sample concentration } \left( \frac{\text{g}}{\text{kg}} \right) = \left( \frac{A}{1000} \cdot \frac{v}{m} \right) \cdot 1000 \quad (1)$$

$$\text{dry sample concentration } \left( \frac{\text{g}}{\text{kg}} \right) = \frac{\text{wet sample concentration}}{1 - \text{moisture content}} \quad (2)$$

A	Spectroquant reading concentration
v	Volume of dilution (L)
m	Mass of sludge used in sample preparation
1-moisture content	Dry solids

For each test done in this study, samples were tested in triplicates and the mean value calculated for data analysis using equation 3.

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} \quad (3)$$

$\bar{x}$  Mean value

n Replicates

$x_i$  Individual values recorded

Standard deviation was calculated to see how dispersed values are from the calculated mean using equation 4.

$$\sigma_n = \sqrt{\frac{1}{n} \sum_{i=1}^n (x - \bar{x})^2} \quad (4)$$

$\sigma_n$  Standard deviation

The student T distribution was used to calculate the margin of error for the results using 80% confidence interval using equation 5.

$$EBM = t_{v, \frac{\alpha}{2}} \frac{\sigma}{\sqrt{n}} \quad (5)$$

EBM	margin of error
v	n - 1 Degrees of freedom
$\alpha$	1 - confidence interval
t	t value provided in the tab

## 4. Results and Discussion

This chapter discuss the results done for this project. The first section discusses the moisture content calibration curves. The section after that will discuss primary macro-nutrients at different moisture contents and different heating temperatures of faecal sludge (FS).

### 4.1 Drying tests

The calibration curves were obtained by drying the VIP sludge at different temperatures in the oven and plotting moisture content as a function of time. The sample moisture content was recorded every 30 minutes. Figure 8 shows that sludge at a lower temperatures loses moisture at lower rate. At a temperature of 50°C to obtain moisture content of 50% and 25% it took 225 and 285 min respectively. When dried at a temperature of 100°C, it took the FS 65 and 95 min to achieve 50% and 25% moisture content respectively. With a temperature of 200°C to obtain 50% and 25% moisture content, it took 70 and 45 minutes respectively. Absolute drying to attain 0% moisture content was achieved by leaving the FS sample to dry for 24 hours in the oven for all the temperatures, but for a temperature of 200°C it was impossible to attain absolute drying for a moisture content of 0% as FS start burning and turning into char after 90 minutes of heating. The burning of sludge at a temperature of 200°C is attributed to thermal degradation (Getahun et al., 2020b). For drying temperatures like 50°C, absolute drying of the samples did not test 0% moisture content and according to Getahun et al. (2020) the moisture content is assumed to be bonded and requires higher temperature to be bonded. Figure 8 is a graph of plotted drying tests and figure 9 is an image of burnt FS after 90 minutes of drying at 200°C.

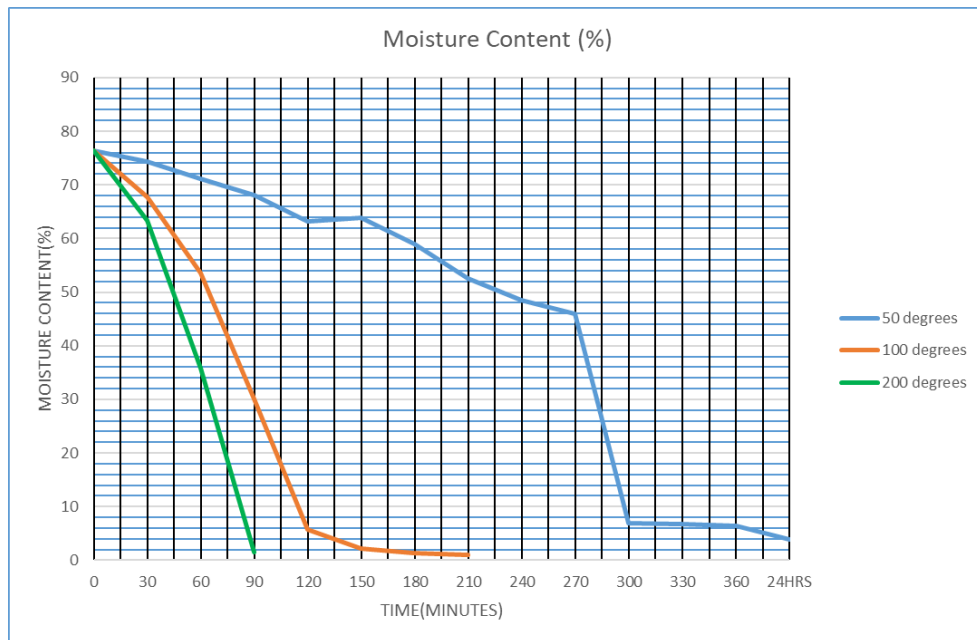


Figure 8: moisture content drying test curves used to estimate different moisture contents drying times



Figure 9 an image of FS after 90 minutes of oven drying at a temperature of 200°C

## 4.2 Nutrients Analysis

This section discusses nutrients evolution of FS under different moisture contents and temperatures. The importance of this analysis is to evaluate the nutrient content of faecal sludge to support plant growing as a form of organic fertilizer. Nutrients

analysed are primary macro-nutrients (N, P, and K) and their fractions (ammonium, nitrates, nitrites, phosphates, ortho-phosphates).

#### 4.2.1 Initial moisture content

The initial moisture content is the moisture content of FS recorded before any treatment was done to the sludge. The initial moisture content was recorded to be  $76.4 \pm 1.1\%$ . The moisture content was within range of initial moisture content as reported by (Zuma et al., 2015). Figure 10 shows different moisture contents of initial moisture content and moisture content after different drying temperatures. Table 2 shows nutrients concentrations of untreated sludge.

Table 2: Nutrient concentration (g/kg dry basis) of raw sludge before thermal drying

Nutrients	Concentration (g/kg dry sample)
Total N	$29.16 \pm 1.28$
NH <sub>4</sub> <sup>+</sup>	$9.63 \pm 0.46$
NO <sub>2</sub> <sup>-</sup>	$0.20 \pm 0.02$
NO <sub>3</sub> <sup>-</sup>	-
PO <sub>34</sub> <sup>-</sup>	$16.34 \pm 0.26$
Orthophosphate PO <sub>34</sub> <sup>-</sup>	$45.30 \pm 1.10$
K <sup>+</sup>	$9.43 \pm 0.03$

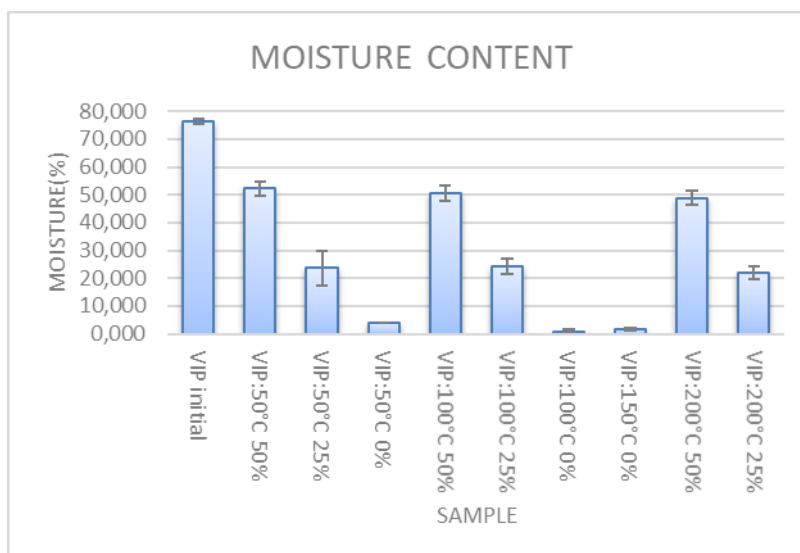


Figure 10: Moisture content of raw and dried faecal sludge at different drying temperatures

#### 4.2.1.1 Nitrogen (N)

Nitrogen is an essential primary macro-nutrient for plant metabolism and development transported by cells within a plant (Rentsch et al., 2007). Nitrogen can be up taken by plants as inorganic nitrogen (nitrates) or organic nitrogen (amino acids), but the most preferable form by plants is organic nitrogen because its carbon content is easily assimilated into proteins (Franklin et al., 2017). The amount of nitrogen for initial moisture content is suitable for plant growth like maize as it is estimated that maize needs 8.7g of nitrogen at full maturity, while a tone of maize will remove 15kg of nitrogen from the soil (Plessis, 2003); Us et al., 2020).

#### 4.2.1.2 Ammonium (NH<sub>4</sub><sup>+</sup>)

Ammonium is an inorganic fraction of nitrogen that is important to plant growth when applied at recommended levels but can very toxic when applied at high levels to plants that can inhibit root growth and plant shooting above the ground (Liu & Von Wirén, 2017). The ammonium concentration measured from untreated sludge was found to be 9.63 g/kg dry basis as reported in table 1.



#### 4.2.1.3 Nitrites ( $\text{NO}_2^-$ )

Nitrites are inorganic form of nitrogen that mostly plants do not utilise in their form and needs bacteria to act upon to be converted to nitrates for plants intake, but nitrite again can be harmful to plants with high concentration of nitrites. Plants such as rice and wheat are said to use nitrite for plant growth (Yoneyama et al., 1980). Table 1 report nitrite concentration to be 0.20 g/kg in raw faecal sludge.

#### 4.2.1.4 Nitrates ( $\text{NO}_3^-$ )

Nitrates are taken by plants as a form of nitrogen and be converted to amino acids to be used for plants growth and development. Plants also uptake nitrates to adopt ecosystem changes and environment (Boudsocq et al., 2012). The untreated sludge had low concentrations of nitrate that cannot be read by the spectroquant testing kit.

#### 4.2.1.5 Phosphates ( $\text{PO}_4^{3-}$ )

Phosphates as a fraction of phosphorus are a major plant nutrient needed for plant growth and reproduction, phosphorus is also important in the genetic information of plants and is responsible for formation of seeds. Phosphate are required as energy for chemical reactions in plants (IPNI (International Plant Nutrition Institute, 1999). The initial concentration of phosphate is more than values recorded by other studies as shown in table 3.

#### 4.2.1.6 Orthophosphates ( $\text{PO}_4^{3-}$ )

Orthophosphates are reactive form of phosphorus that are readily available for plant intake. From the table 1 it shows that the orthophosphate concentration in untreated sludge is  $45.30 \pm 1.1$ .

#### 4.2.1.7 Potassium (K)

Potassium is one of the plant major nutrient, K is important for enzyme activation, nutrient transport in the stomata, and also needed for energy transfer by plants (Wang et al., 2013). Potassium is also needed for nitrogen metabolism which plays a vital role in plant growth (Xu et al., 2020). The potassium concentration for this is not far from concentrations found by other authors as shown in table 3.

Table 3 shows results found in this study compared to literature data for raw sludge

Nutrient	Raw sludge concentration g/kg dry basis		
	Raw sludge	(Mirara, 2017)	( Makununika, 2016)
N	$29.16 \pm 1.28$	-	-
$\text{NH}_4^+$	$9.63 \pm 0.46$	18.7 - 26.7	-
$\text{NO}_2^-$	$0.20 \pm 0.02$	13.1 - 15.9	-
$\text{NO}_3^-$	-	1.1 - 1.3	0.5
$\text{PO}_4^{3-}$	$16.34 \pm 0.26$	11.0 - 12.0	2.4
Orthophosphates ( $\text{PO}_4^{3-}$ )	$45.30 \pm 1.10$	-	-
K	$9.43 \pm 0.03$	7.4 - 8.4	8.7

## 4.2.2 Nutrients at fifty degrees Celsius (50°C)

This section discusses nutrient behaviour when heated at 50°C to achieve different moisture content at different drying times.

### 4.2.2.1 N

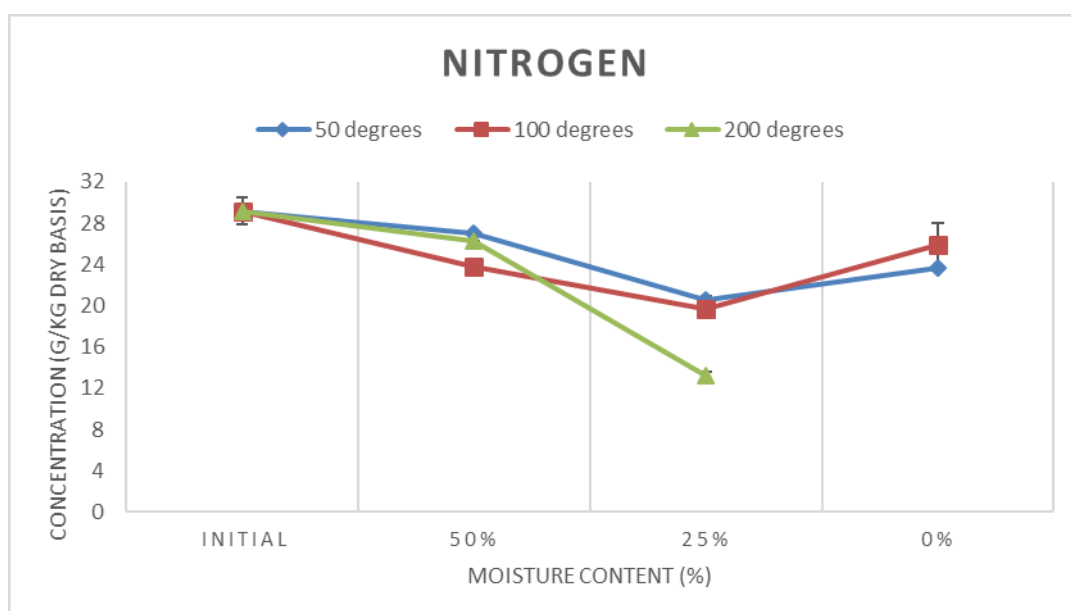


Figure 11 shows a graph of nitrogen concentrations at different moisture content dried at different temperature

Figure 11 shows that nitrogen concentration when dried at 50°C reduces with drying time in the oven until it reaches moisture content of 25%, after that it increase slightly. As a function of moisture content when dried at 50°C, the nitrogen concentrations reduced from 29.16 g/kg of initial concentration to 27.03 g/kg at 50% moisture content until it reached 20.55 g/kg and 23.08 g/kg at 25% and 0% moisture content respectively. This is because during drying the chemical form of nitrogen is modified as it mineralises and changes to fractions of total nitrogen, but not all nitrogen mineralized because the remaining organic nitrogen was bonded to the dry matter (Septien et al., 2020).

#### 4.2.2.2 NH<sub>4</sub><sup>+</sup>

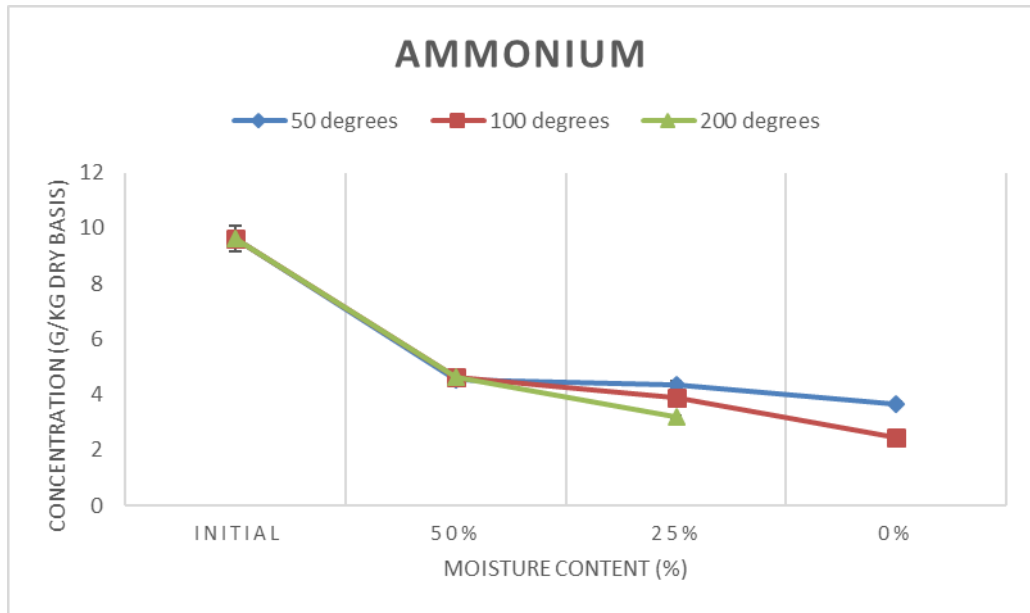


Figure 12 shows a graph of ammonium concentrations at different moisture content dried at different temperatures

The change in ammonium concentration during drying at 50°C is significant with over 50% difference, from initial concentration of 9.63 g/kg to 4.54 at 50% moisture content as shown by figure 12. Ammonium concentration start to stabilise at moisture content of 25% and 0% with concentrations of 4.35 g/kg and 3.68g/kg respectively. The change between initial concentration and 50% moisture content concentration is because drying might have volatilised ammonium and changed it to gas which can escape as ammonia gas (Afolabi & Sohail, 2017; Rigby et al., 2016). Most of the ammonium that was recorded at 50% to 0% is assumed to be bonded to the dry matter.

#### 4.2.2.3 NO<sub>2</sub><sup>-</sup>

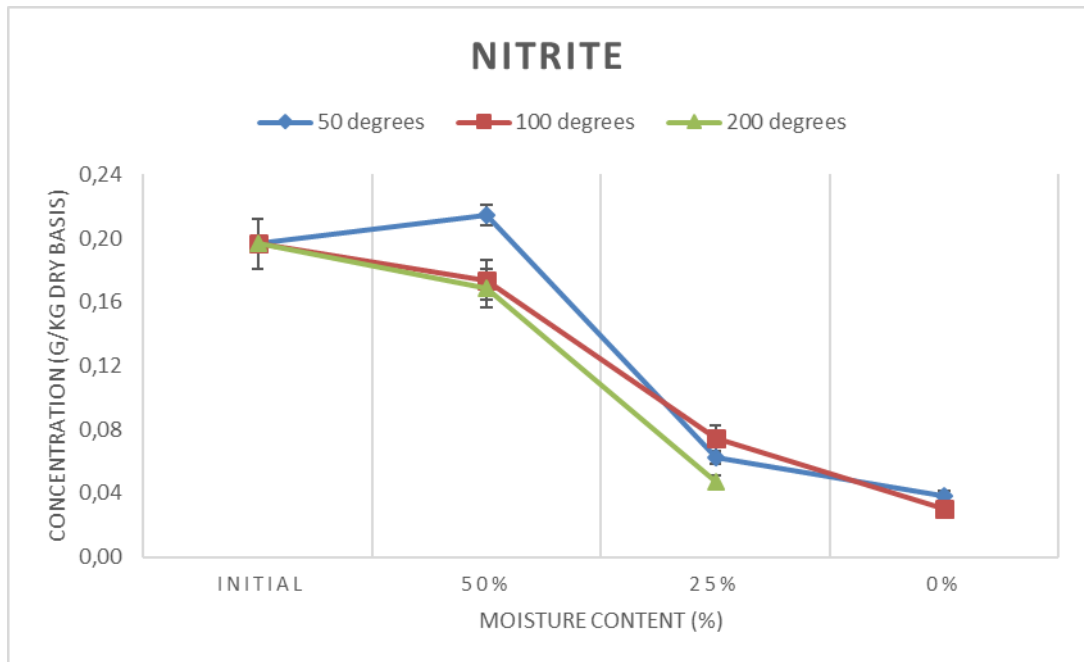


Figure 13 shows a graph of ammonium concentrations at different moisture content dried at different temperature

Figure 13 shows that nitrite changes concentration with drying time when dried at 50°C drying temperature, there was insignificant change of concentration between initial moisture content and 50% moisture content as 0.20 g/kg and 0.21 g/kg were recorded respectively. Concentration then drops by 70% respective to ininitial concentration to 0.06 g/kg at 25% moisture content followed by an insignificant change to 0.04 g/kg at 0% moisture content. Heat volatilizes the nitrites (Mirara, 2017) that will be transformed into nitrates then ammonia or gaseous state like carbon IV oxide and escape (Afolabi & Sohail, 2017).

#### 4.2.2.4 NO<sub>3</sub><sup>-</sup>

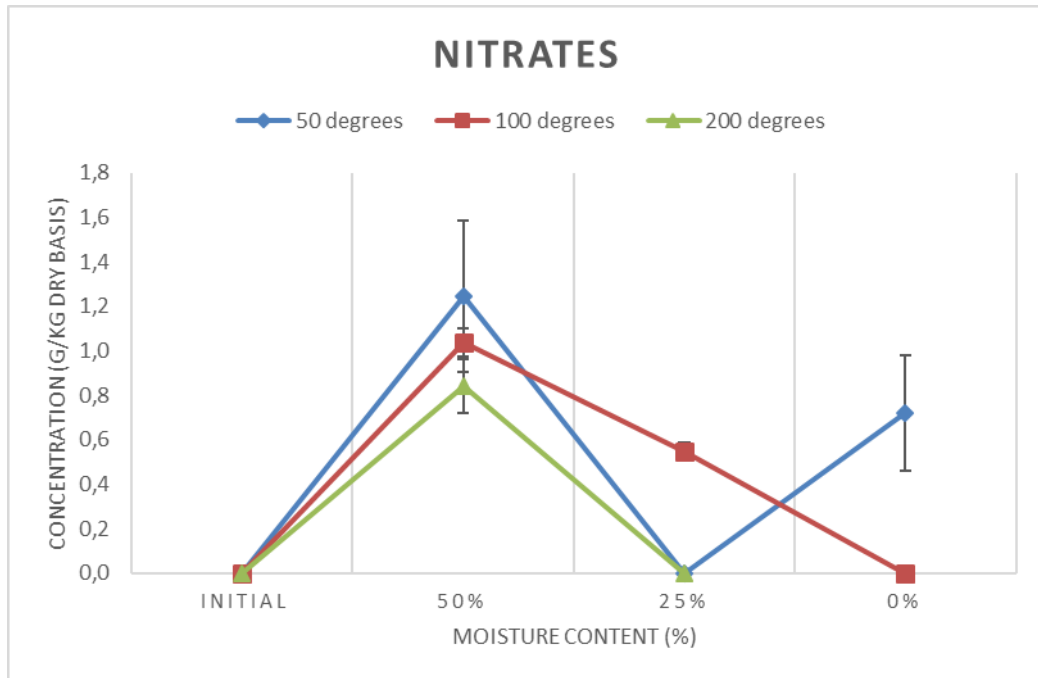


Figure 14 shows a graph of nitrates concentrations at different moisture content dried at different temperatures

The untreated sludge had low concentration of nitrates that could not be read by the spectroquant kit. When heated the nitrate concentration increased at 50% moisture content to a value of 1.25g/kg and then decreased with the drying time to reach low concentrations again at 25% moisture content, it then picked up again to a concentration of 0.72 g/kg as shown in figure 14. Due to heat and loss in moisture content, nitrate ions could have changed form to ammonium ions (Mirara, 2017).

#### 4.2.2.5 $\text{PO}_4^{3-}$

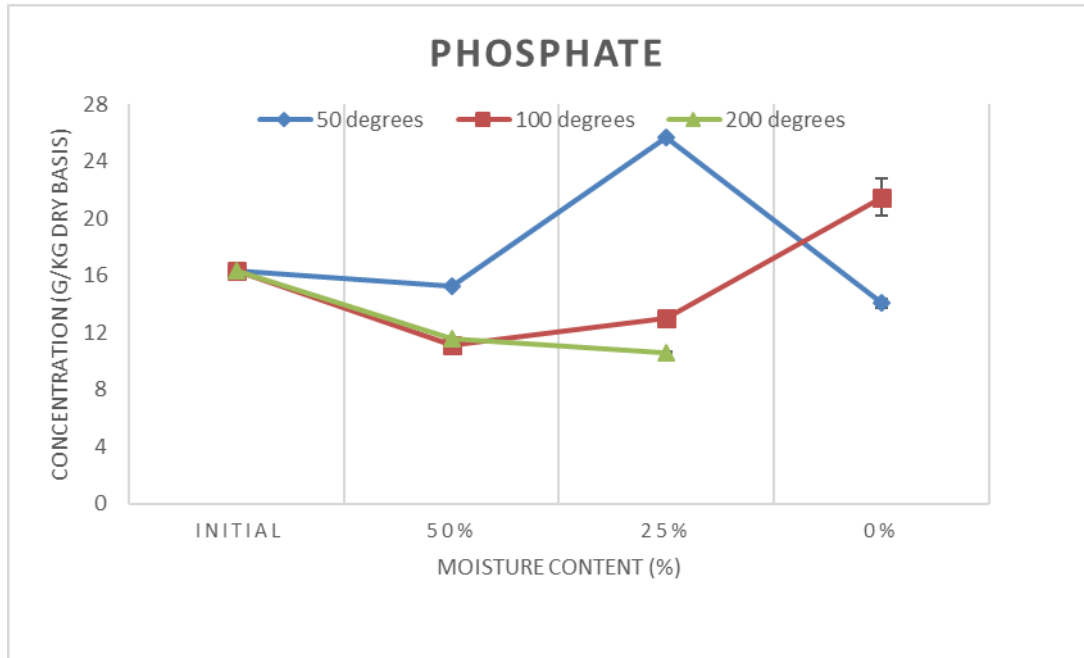


Figure 15 is a graph of phosphate concentrations at different moisture content dried at different temperature.

The phosphates concentrations change was insignificant at first when dried from initial moisture content to 50% moisture as shown in figure 15, it recorded 16.34 g/kg and 15.21 g/kg respectively. The concentration increases at moisture content of 25% with a value of 25.66 g/kg which is more than 50% increase relative to initial concentration, it then decreased at 0% moisture content to value of 14.03 g/kg. Drying activates speciation of phosphate to react with other elements to form different salts hence the value change in concentrations (He et al., 2004; Meng et al., 2019). The trend behaviour of phosphate in this sludge is different from what Mirara (2017) who reported that temperature and residence time did not affect the concentrations of phosphate on faecal sludge, this could be the heterogeneous behaviour of faecal sludge.

#### 4.2.2.6 Orthophosphates ( $\text{PO}_4^{3-}$ )

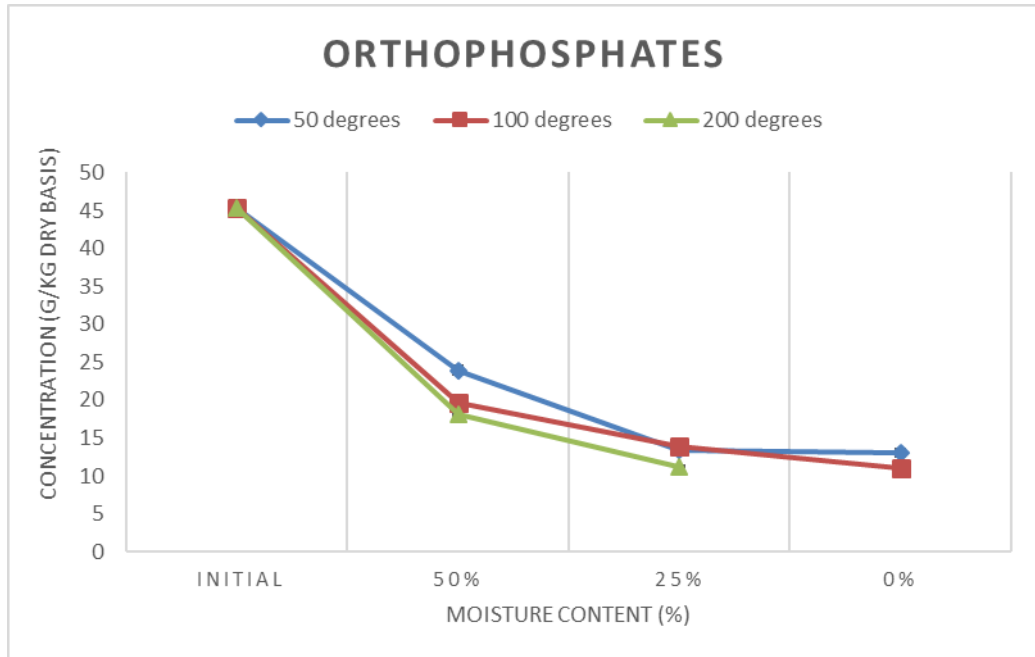


Figure 16 is a graph of orthophosphate concentrations at different moisture content dried at different temperatures.

Orthophosphate concentration as a function of drying temperature decreases with the drying time as shown in figure 16. The phosphate concentrations decreased from 45.30 g/kg to 23.94 g/kg which is over 45% change of concentration at 50% moisture content, the change in concentration continues to drop with drying time until the sludge reached moisture content of 0% with 13.01 g/kg. Change in concentrations is caused by loss of moisture by sludge and causes phosphorus speciation (He et al., 2004). The trend behaviour that was shown by orthophosphates in this study is different from what Makununika (2016) reported for orthophosphates not to be affected by drying temperature.



#### 4.2.2.7 K

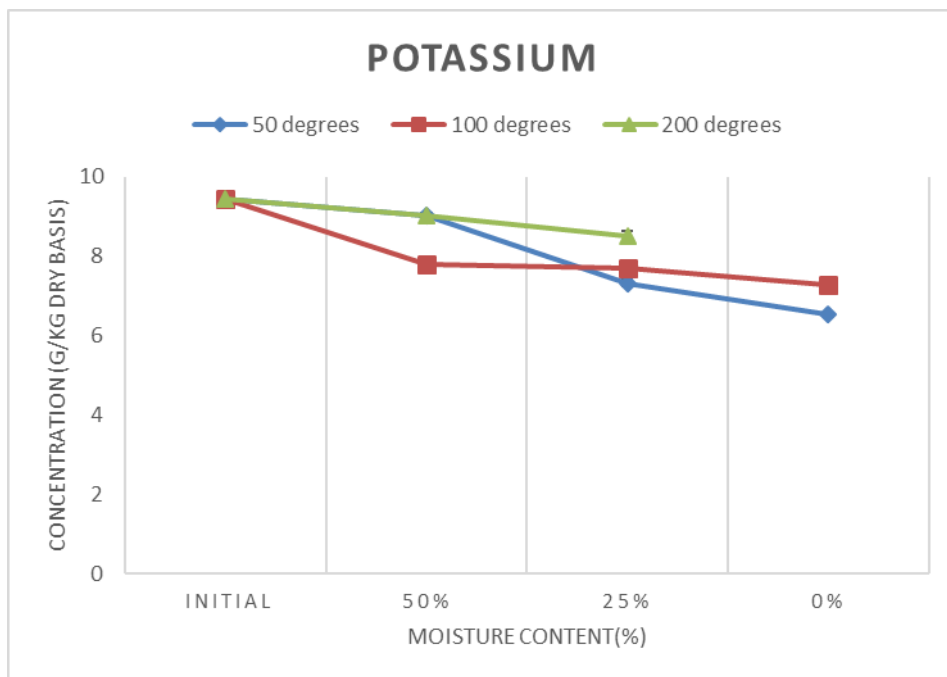


Figure 17 is a graph of potassium concentrations at different moisture content dried at different temperatures.

Figure 17 shows that the change in potassium concentration from untreated sludge to when dried was insignificant. The change in value was about 3g/kg which is less than 10% of concentration change when the sludge was dried until it reached 0% moisture content. This is because the potassium might be heavily bonded to the dry matter of sludge (Mirara, 2017; Makununika, 2016).

### 4.2.3 Nutrients at one hundred degrees Celsius (100°C)

This section discusses nutrient behaviour when heated at 100°C to achieve different moisture content at different drying times.

#### 4.2.3.1 N

The Nitrogen behaviour pattern when dried at 100°C as shown by figure 11 was determined to be the same when dried at 50°C where it drops until it reaches 25% moisture content and then picks up again. The difference is the value it changes with, when dried at 100°C, at 50% moisture content the concentration dropped to 23.72 g/kg then 19.66 g/kg at 25% moisture content then picked to 25.91 g/kg at 0% moisture content. The nitrogen as reported by Septien et al. (2020) changes the forms it stored in the sludge but not completely lost as heat causes it mineralize (Rigby et al., 2016).

#### 4.2.3.2 NH<sub>4</sub><sup>+</sup>

Ammonium concentration behaviour pattern when dried at 100°C and 50°C were the same as shown in figure 12. The concentration drops by half to 4.64 g/kg at 50% moisture content from initial concentration then to 3.89 g/kg at 25% moisture content at finally changes to 2.46 g/kg at 0% moisture content. According to Waweru (2017) it is because ammonium is very volatile at ambient conditions and heat catalyses the volatility of ammonium.

#### 4.2.3.3 NO<sub>2</sub><sup>-</sup>

Figure 13 shows that the pattern behaviour of nitrites at drying temperature of 100°C was slightly different from the pattern of 50°C, at 50% moisture content. The nitrites immediately drop value of concentration at 50% moisture content to 0.17 g/kg, according to Waweru (2017) it is because heat volatilizes nitrites. This becomes more evident as the nitrogen concentration continued to drop until it reached 0% moisture content with a concentration of 0.03 g/kg.

#### 4.2.3.4 NO<sub>3</sub><sup>-</sup>

From figure 14 shows that the behaviour pattern at 50°C and 100°C are the same, it shows that nitrate concentrations increases at 50% moisture 1.04 g/kg from low concentration recorded of untreated sludge and start to decrease at 25% moisture content to 0.55 g/kg. Makununika (2016) believes that drying time increases the volatility of nitrates and that supports the behaviour of nitrates showed in this study.

#### 4.2.3.5 PO<sub>4</sub><sup>3-</sup>

The behaviour pattern of phosphate displayed by figure 15 shows that drying temperature of 100°C and 50°C are the same. From initial concentration the value decreased at 50% moisture content where it recorded 11.13 g/kg, and then it increased at 50% moisture content where it recorded a concentration 13.02 g/kg. The only difference in behaviour is evident at 0% moisture content, the concentration continued to increase to 21.47 g/kg which is more than the initial concentration by 30%. He et al. (2004) explains this behaviour of phosphates as a result of phosphorus speciation.

#### 4.2.3.6 Orthophosphates ( $\text{PO}_4^{3-}$ )

Orthophosphate behaviour pattern is the same for drying temperature of 100°C and 50°C as shown by figure 16. Concentration decreases to 19.71 g/kg at 50% moisture content then continue to drop to 10.99 g/kg at 0% moisture content which is more than 75% change. Drying FS may cause mobility of phosphorus affecting orthophosphate concentrations (Meng et al., 2019). As figure 16 shows, wet conditions support the availability of orthophosphates, with decreasing moisture content phosphorus may react with other elements to form different salts (He et al., 2004).

#### 4.2.3.7 K

The behaviour pattern of potassium displayed by figure 17 is the same for 50°C and 100°C drying temperatures. Potassium in FS is believed to be strongly bonded to dry matter and is not lost or affected by drying according to Waweru (2017); Makununika (2016). From initial potassium concentration there was only a decrease of less than 2 g/kg and after that it stabilised and did not record any significant change until 0% moisture content.

#### 4.2.4 Nutrients at one hundred and fifty degrees Celsius (200°C)

This section discusses nutrient behaviour when heated at 200°C to achieve different moisture content at different residence time, but under this condition 0% moisture was not achieved as the FS sample started burning and turning into char after 90 minutes of drying. A drying temperature of 150°C was introduced to achieve absolute drying.

#### 4.2.4.1 N

At drying temperature of 200°C, nitrogen concentration behaviour as shown in figure 11 was the same as for drying temperature of 50°C and 100°C. The concentration reduces from initial concentration to 26.24 g/kg to achieve 50% moisture content, at 25% it reduced by more than 50% to 13.19 g/kg from the initial concentration which is the highest reduction compared to other drying temperatures but when dried at 150°C to achieve 0% moisture content it records a concentration of 19.67 g/kg. Loss of moisture affects how nitrogen changes forms as discussed by Makununika (2016).

#### 4.2.4.2 NH<sub>4</sub><sup>+</sup>

Ammonium shows the same behaviour pattern as shown in figure 12 when dried with temperatures of 50°C, 100°C and 200°C. The concentration decreased with closely similar values from initial concentration throughout the moisture contents. Temperature increase did not have a big impact more than other drying temperatures but maintained the same pattern. This might be that the remaining ammonium was strongly bonded to the dry matter (Mirara, 2017).

#### 4.2.4.3 NO<sub>2</sub><sup>-</sup>

Nitrites exhibited the same behaviour pattern as shown in figure 13 for temperatures of 200°C, 100°C and 50°C. NO<sub>2</sub><sup>-</sup> experienced reduction in concentration respective to drying time to achieve moisture content of 50% and 25%. The change in concentrations were closely the same with other drying temperatures even when dried at 150°C to achieve 0% moisture content. Most of the nitrites might be strongly bonded to the dry matter (Mirara, 2017).

#### 4.2.4.4 NO<sub>3</sub><sup>-</sup>

Figure 14 shows that nitrates exhibited same behaviour pattern for drying temperature of 200°C, 100°C and 50°C where from low initial concentration it increases at 50% moisture content then decreases again to low concentration at 25% moisture content as it proves Makununika (2016) discussion that temperature increases the volatility of nitrates.

#### 4.2.4.5 PO<sub>4</sub><sup>3-</sup>

Under 200°C drying temperature, phosphate had closely the same behaviour pattern as compared to drying temperature of 100°C and 50°C as shown in figure 15. The concentration decreased to 11.60 g/kg from initial moisture content to 50% moisture content but did not increase at moisture content of 25% as compared to other drying temperatures, the concentration dropped to 10.62 g/kg. Meng et al. (2019) discussed that heat increases the mobility of phosphorus and the behaviour pattern displaced by phosphates under different temperatures and moisture contents proves that.

#### 4.2.4.6 Orthophosphates (PO<sub>4</sub><sup>3-</sup>)

Figure 16 shows that at drying temperature of 200°C the orthophosphates still maintained the same behaviour pattern of 100°C and 50°C. The concentration decreased with drying time to achieve moisture contents of 50%, 25% and 0% moisture content at drying temperature of 150°C. This pattern supports He et al. (2004) theory that phosphorus is more available under wet conditions.

#### 4.2.4.7 K

Under drying temperature of 200°C, potassium maintained the same behaviour pattern as under drying temperatures of 100°C and 50°C. Increase in temperature and drying time did not affect potassium concentrations or its form as insignificant change in concentration levels was observed in figure 17. Mirara (2017); Makununika (2016) observed the same behaviour of potassium.

### **5. Conclusion and Recommendations**

The study was focused on thermal drying of faecal sludge using the laboratory oven with different temperatures of 50°C, 100°C and 200°C to achieve different moisture contents of 50%, 25% and 0% under each drying temperature and study the behavioural change of macronutrients (N,P,K) under each condition. Total nitrogen was studied with its inorganic fraction ( $\text{NH}_4^+$ ,  $\text{NO}_2^-$ , and  $\text{NO}_3^-$ ) and phosphorus was studied as  $\text{PO}_4^{3-}$  and Orthophosphates ( $\text{PO}_4^{3-}$ ) and potassium.

Drying with different temperatures and change in moisture content proved to have an impact on the nutrient concentrations of faecal sludge, so it can be concluded that there is evolution of nutrients due to thermal drying. Total nitrogen was changing concentrations loss of moisture of FS. The total nitrogen concentrations changed more with increase to loss of moisture content, this was believed to be a resultant of mineralisation of total nitrogen. At the end of drying all the total nitrogen was not lost as the remaining nitrogen was believed to be organic nitrogen bonded to the dry matter which can be easily assimilated to proteins to be used by plants.

Ammonium was more volatile with loss of moisture content as the change in ammonium concentration were following the same behavioural pattern irrespective

of temperature. The concentration reduces by more than 50% at 50% moisture content with a value of 4.54 g/kg, 4.64 g/kg and 4.63 g/kg for drying temperatures of 50°C, 100°C and 200°C respectively, the concentration is so close irrespective of increase in drying temperature. Continued dried to other moisture contents of 25% and 0% moisture content the ammonium concentrations still followed the same pattern. It can be concluded that moisture content has more impact in volatilization of ammonium than temperature and little ammonium is left bonded to the dry matter at 0% moisture content compared to the initial concentration.

Nitrites also showed the same behavioural pattern as ammonium of reacting to loss of moisture content more than the varying of drying temperatures. The concentrations kept on dropping from the initial concentration respective to moisture loss and following the same pattern. Nitrates also exhibited the same pattern to reacting towards loss of moisture content. At 50% moisture content, all drying temperatures recorded a concentration for nitrates but was low at initial conditions of FS. The concentrations then reduced to low values at 25% moisture content except with a drying temperature of 100°C but still records a concentration lower than at 50°C. For nitrates, it can be concluded that moisture content had impact in how nitrates behave due to their volatility. When looking at total nitrogen and its fractions it can be concluded 50% moisture content conditions is the one with high concentration values irrespective of drying temperature and that the condition that plants get both inorganic and organic nitrogen at fair concentrations.

Phosphate reacted differently as a function of moisture content. With a drying temperature of 50°C phosphates recorded the highest concentration of 25.66 g/kg at 25% moisture content. For all the drying temperatures the concentration decreased at 50% moisture content from the initial concentration. With a drying temperature of 100°C the concentration increased at 0% moisture content but for a drying temperature of 50°C it decreased this proves phosphates mobility is influenced by moisture content.

From this study, it proved that orthophosphates follows the same pattern behaviour irrespective of different drying temperatures but moisture content. For all the drying temperatures used in this study, the concentrations decreases at 50% moisture



content and kept on decreasing until 0% moisture content was reached. The value change under different drying temperatures at different moisture content was not that big but for higher temperature of 200°C it recorded low concentrations compared to 50°C drying temperature under different moisture contents.

The change in potassium concentrations at different moisture content and different drying temperatures was insignificant and the potassium was strong believed to be bonded to the dry matter. The changes were less than 2g/kg and did not show any particular pattern respective to moisture content and drying temperature. As potassium is an important nutrients for plants development and growth this could be good for plants.

It can be recommended that the following be done in studying faecal sludge:

Testing of the dried sludge at different temperatures and moisture content on plants to determine how nutrients are released to be used by plants. The study could not be carried out due to time constraints.

Testing of total phosphorus to understand how it behaves not only its fractions. The tests could not be done, as there were no testing kits for total phosphorus.

Study the community perception on faecal sludge use as a substitute for synthetic fertilizer in agricultural production.

Study the comparison of costs between production and use of synthetic fertilizer and faecal sludge as crop fertilizers.

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## 7. Appendix

### Appendix 7.1. Standard Operating Procedure

#### Appendix 7. 1.1. Standard operation procedure – Ammonium Test (Cat. No. 1.00683)

##### Scope and Field of Application

Test measures both ammonium ions and dissolved ammonia in a concentration range of 2 – 150 mg/l NH<sub>4</sub>-N

##### Principle

Ammonium nitrogen (NH<sub>4</sub>-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<sub>4</sub>-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.

##### Interferences

Concentrations of foreign substances in mg/l or %					
Al <sup>3+</sup>	1000	Mn <sup>2+</sup>	100	EDTA	1000
Ca <sup>2+</sup>	1000	Ni <sup>2+</sup>	250	Primary Amines	0
Cd <sup>2+</sup>	1000	NO <sub>2</sub> <sup>-</sup>	1000	Secondary Amines	250
CN <sup>-</sup>	100	Pb <sup>2+</sup>	1000	Aminophenols	10
Cr <sup>3+</sup>	100	PO <sub>4</sub> <sup>2-</sup>	1000	Aniline	50
Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	1000	S <sub>2</sub> <sup>-</sup>	50	Triethanolamine	1000
Cu <sup>2+</sup>	1000	SiO <sub>3</sub> <sup>2-</sup>	1000	Surfactants	1000
F <sup>-</sup>	1000	Zn <sup>2+</sup>	500	Na-acetate	10%
Fe <sup>3+</sup>	25			NaCl	20%
Hg <sup>2+</sup>	500			NaNO <sub>3</sub>	20%
Mg <sup>2+</sup>	500			Na <sub>2</sub> SO <sub>4</sub>	20%

### 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 NH<sub>4</sub>-N must be diluted with distilled water.

### 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.

### Apparatus

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

### Reagents

- Reagent NH<sub>4</sub>-1
- Reagent NH<sub>4</sub>-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

### Calibration

To calibrate test solutions of 5.0, 10, 50 and 100 mg/l NH<sub>4</sub>-N.

## Procedure

### **Measuring range of 2.0 – 75.0 mg/l NH<sub>4</sub>-N (2.6 – 96.9 mg/l NH<sub>4</sub><sup>+</sup>):**

- Pipette 5.0 ml of reagent NH<sub>4</sub>-1, stored between 20 – 30 °C, into a test tube
- Pipette 0.2 ml of pretreated sample into the test tube and mix.
- Add 1 level blue microspoon of reagent NH<sub>4</sub>-2 and shake vigorously until the reagent is completely dissolved.
- 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 NH<sub>4</sub>-N (6 – 193 mg/l NH<sub>4</sub><sup>+</sup>):**

- Pipette 5.0 ml of reagent NH<sub>4</sub>-1, stored between 20 – 30 °C, into a test tube
- Pipette 0.1 ml of pretreated sample into the test tube and mix.
- Add 1 level blue microspoon of reagent NH<sub>4</sub>-2 and shake vigorously until the reagent is completely dissolved.
- 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<sub>4</sub>-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)

## Data Quality

Measurement	2 – 75 mg/l NH <sub>4</sub> -N	5 – 150 mg/l NH <sub>4</sub> -N
StandardDeviation (mg/lNH <sub>4</sub> N)	± 0.49	± 1.0



Confidence Interval (mg/l NH <sub>4</sub> -N)	± 1.2	± 2
Sensitivity (mg/l NH <sub>4</sub> -N)	0.3	1
Accuracy (mg/l NH <sub>4</sub> -N)	± 1.8	± 4.0

#### Chemical Waste Disposal

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

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

### Appendix 7.1.2. Standard Operation Procedure – Nitrates Test (Cat. No. 1.09713)

#### Scope and Field of Application

Test measures the nitrate concentration, in the range of 0.10 – 25.0 mg/l NO<sub>3</sub><sup>-</sup>-N, of solutions with a maximum of 0.2% sodium chloride and 50 mg/l NO<sub>2</sub><sup>-</sup>.

#### Principle

In sulphuric and phosphoric solution nitrate ions react with 2,6-dimethylphenol(DMP) to form 4-nitro-2,6-dimethylphenol that is determined photometrically.

#### Interferences

Concentrations of foreign substances in mg/l or %					
Al <sup>3+</sup>	1000	Hg <sup>2+</sup>	100	Surfactants	1000
Ca <sup>2+</sup>	500	Mg <sup>2+</sup>	1000	COD (K-Hydrogen phthalate)	500
Cd <sup>2+</sup>	250	Mn <sup>2+</sup>	1000		
Cl <sup>-</sup>	1000	NH <sub>4</sub> <sup>+</sup>	1000	Organic substances (glucose)	500
CN <sup>-</sup>	100	Ni <sup>2+</sup>	500		
Cr <sup>3+</sup>	500	NO <sub>2</sub> <sup>-</sup>	5	Na-acetate	25%
Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	50	Pb <sup>2+</sup>	100	NaCl	0.2%
Cu <sup>2+</sup>	500	PO <sub>4</sub> <sup>3-</sup>	1000	Na <sub>2</sub> SO <sub>4</sub>	25%
F <sup>-</sup>	1000	SiO <sub>3</sub> <sup>2-</sup>	500		
Fe <sup>3+</sup>	100	Zn <sup>2+</sup>	1000		

## Sampling

Analyze immediately after sampling.

Check the chloride content, with Merckoquant Chloride Test, if concentration range is unknown. Samples containing more than 1000mg/l  $\text{Cl}^-$  must be diluted with distilled water.

Check the nitrite content, if necessary, eliminate interfering nitrite ions (stated amounts apply for nitrate contents of up to 50 mg/l). To 10ml of sample add approximately 50mg of amidosulphuric acid and dissolve. The pH of this solution must be within the range of 1-3. Adjust, if necessary with sulphuric acid.

Check the nitrate content with the Merckoquant® Nitrate Test. Samples containing more than 25.0 mg/l  $\text{NO}_3\text{-N}$  (110.7 mg/l  $\text{NO}_3^-$ ) must be diluted with distilled water.

Filter turbid samples

## Safety Precautions

Handle concentrated acid with care.

Always use safety goggles, gloves and laboratory coat while working in laboratory.

After the analysis clean bottles and beakers with distilled water keep.

Dispose 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 use copious washings with cold water and call for medical attention.

## Apparatus

Pipettes

Rectangular cells

Spectrophotometer

Reagents

Reagent  $\text{NO}_3\text{-1}$

Reagent  $\text{NO}_3\text{-2}$

Amidosulphuric acid

Sulphuric acid

Universal indicator strips pH 0 – 14

### Calibration

To check the photometric measurement system (test reagents, measurement device, and handling) and the mode of working, the nitrate standard solutions CRM, 0.500 mg/l NO<sub>3</sub>-N, CAT No. 125036, 2.50 mg/l NO<sub>3</sub>-N Cat No 125037, and 15.0 mg/l NO<sub>3</sub>-N Cat No 125038 can be used. Alternatively make up equivalent solutions.

### Procedure

Pipette 4.0ml of reagent NO<sub>3</sub>-1 into a dry test tube, which can be sealed.

Add with pipette 0.50ml of pretreated sample (5-25°C), do not mix.

Add with pipette 0.5ml of reagent NO<sub>3</sub>-2 (Wear eye protection as the mixture becomes hot) and mix, holding only the upper part of the tube.

Leave the hot reaction to stand for 10 min (reaction time). Do not cool with water.

Fill the sample into the rectangular cell and 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 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 %.)

### Data Quality

Measurement	0.1 – 5.0 mg/l NO <sub>3</sub> -N	1.0 – 25.0 mg/l NO <sub>3</sub> -N
Standard Deviation (mg/l NO <sub>3</sub> -N)	± 0.11	± 0.11
Confidence Interval (mg/l NO <sub>3</sub> -N)	± 0.3	± 0.3
Sensitivity (mg/l NO <sub>3</sub> -N)	0.04	0.2
Accuracy (mg/l NO <sub>3</sub> -N)	± 0.10	± 0.5

### Chemical Waste Disposal

Dilute 10 ml into 1000ml.

Slowly add NaCO<sub>3</sub> until pH 6-8 is reached.

Flush down the sink with excess water.

### Appendix 7.1.3. Standard operation procedure – Nitrites Cell Test (Cat. No. 1.00609)

#### Scope and Field of Application

Test measures the nitrite concentration in the range 1.0 – 90 mg/l NO<sub>2</sub>-N.

#### Principle

In acidic solution nitrite ions react with iron(II) ethylenediammonium sulphate to form a yellow to green-brown iron(II) compound that is determined photometrically.

#### Interferences

Concentrations of foreign substances in mg/l or %				
Ag <sup>+</sup>	1	Hg <sup>2+</sup>	100	EDTA 1000
Ca <sup>2+</sup>	1000	Mg <sup>2+</sup>	1000	Reducing agents 10 (ascorbic acid, sulfite)
Cd <sup>2+</sup>	1000	Mn <sup>2+</sup>	1000	
BO <sub>3</sub> <sup>2-</sup>	1000	Mo <sup>6+</sup>	500	NaCl 20 %
CN <sup>-</sup>	1000	NH <sub>4</sub> <sup>+</sup>	1000	NaNO <sub>3</sub> 20 %
Cr <sup>3+</sup>	100	Pb <sup>2+</sup>	1000	Na <sub>2</sub> SO <sub>4</sub> 15 %
CrO <sub>4</sub> <sup>2-</sup>	100	PO <sub>4</sub> <sup>3-</sup>	1000	
Cu <sup>2+</sup>	100	S <sub>2</sub> <sup>-</sup>	10	
F <sup>-</sup>	100	SiO <sub>3</sub> <sup>2-</sup>	1000	
Fe <sup>3+</sup>	1	Zn <sup>2+</sup>	1000	
CO <sub>3</sub> <sup>2-</sup>	100	Sn <sup>2+</sup>	10	

#### Sampling

Preferably collect samples in glass bottles.

Analyze immediately after sampling.

Check the Nitrite content with the Merckoquant Nitrite Test. Samples containing more than 90.0 mg/l NO<sub>2</sub>-N must be diluted with distilled water.

The pH must be within the range 1 - 12. Adjust, if necessary, with sulfuric acid.

Filter turbid samples.

### Safety Precautions

Handle concentrated acid with care

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.

### Apparatus

Spectrophotometer

Reaction cells

Pipette

### Reagents

Reagent  $\text{NO}_2\text{-1K}$

Universal indicator strips pH 0-14.

Acidic indicator strips, pH 0-6.

Sulphuric acid 0.5 mol/l

### Calibration

To check the photometric measurement system (test reagents, measurement device, handling) and the mode of working, the nitrite solution 40 mg/l  $\text{NO}_2\text{-N}$  can be used.

### Procedure

Place 2 level blue microspoons of reagent  $\text{NO}_2\text{-1K}$  in a reaction cell.

Pipette 8.0 ml of pretreated sample into the reaction cell, close the cell and shake until the reagent is completely dissolved.

Leave to stand for exactly 20 minutes, then measure the sample in the photometer.

Do not shake or swirl the cell before the measurement.

### Notes on the measurement:

For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry paper towel.

Measurement of turbid solutions yields false-high readings.

The pH of the measurement solution must be within the range 1.2 - 1.6.

The colour of the measurement solution remains stable for only a short time after the end of the reaction time stated above.

#### Data Quality

Measurement	1.0 – 90.0 mg/l NO <sub>2</sub> -N
Standard Deviation (mg/l NO <sub>2</sub> -N)	0.47
Confidence Interval (mg/l NO <sub>2</sub> -N)	± 1.1
Sensitivity (mg/l NO <sub>2</sub> -N)	±0.8
Accuracy (mg/l NO <sub>2</sub> -N)	± 2.0

#### Chemical Waste Disposal

### Appendix 7. 1.4. Standard Operation Procedure – Nitrogen (Total) Cell Test (Cat. No. 1.14763)

#### 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.

#### 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.

#### Interferences

Concentrations of foreign substances in mg/l or %

Al <sup>3+</sup>	1000	Hg <sup>2+</sup>	1000	Surfactants	500
Ca <sup>2+</sup>	1000	Mg <sup>2+</sup>	1000	CSB (K-Hydrogen 3500 phthalate)	
Cd <sup>2+</sup>	1000	Mn <sup>2+</sup>	1000		

Cl <sup>-</sup>	10000	Ni <sup>2+</sup>	1000	Na-acetate	10 %
Cr <sup>3+</sup>	100	Pb <sup>2+</sup>	1000	NaCl	2 %
Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	100	PO <sub>4</sub> <sup>3-</sup>	1000	Na <sub>2</sub> SO <sub>4</sub>	10 %
Cu <sup>2+</sup>	1000	SiO <sub>3</sub> <sup>2-</sup>	1000		
F <sup>-</sup>	1000	Sn <sup>2+</sup>	1000		
Fe <sup>3+</sup>	1000	Zn <sup>2+</sup>	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.

### 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.

### Safety Precautions

Handle concentrated acid with care

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.

### Apparatus

Spectroquant

Reaction cells

Thermoreactor

Pipettes

### Reagents

Reagent N-1K

Reagent N-2K

Reagent N-3K

#### 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.

#### Procedure

Pipette 1 ml of pretreated sample into an empty cell.

Add 9 ml of distilled water into cell and mix.

Add 1 level blue microspoon of reagent N-1K and mix.

Add 6 drops of reagent N-2K, close cell and mix.

Heat the cell at 120 °C in the preheated thermoreactor for 1 hour. Shake the cell briefly after 10 minutes.

Pipette 1 ml of the digested solution into a reaction cell. Do not mix.

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.

Leave the hot reaction to stand for 10 min (reaction time). Do not cool with water.

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 %.)

#### 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



### Appendix 7.1.5. Standard operation procedure – Phosphorous (total) test (Cat. No. 1.14543)

#### Scope and Field of Application

Test measures the total phosphorous, in a concentration range of 0.05 – 5.0 mg/l PO<sub>4</sub>-P.

#### Principle

In sulfuric solution orthophosphate ions react with molybdate ions to form molybdophosphoric acid. Ascorbic acid reduces this to phosphomolybdenum blue (PMB) that is determined photometrically.

#### Interferences

Concentrations of foreign substances in mg/l or %					
Ag <sup>+</sup>	1000	Fe <sup>3+</sup>	1000	EDTA	1000
AsO <sub>4</sub> <sup>3-</sup>	0.2	Hg <sup>2+</sup>	10	Surfactants	100
Ca <sup>2+</sup>	1000	Mg <sup>2+</sup>	1000	COD (K-hydrogen phthalate)	150
Cd <sup>2+</sup>	1000	Mn <sup>2+</sup>	1000		
CN <sup>-</sup>	1000	NH <sub>4</sub> <sup>+</sup>	1000	Na-acetate	1 %
Cr <sup>3+</sup>	1000	Ni <sup>2+</sup>	500	NaCl	5 %
Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	5	NO <sub>2</sub> <sup>-</sup>	1000	NaNO <sub>3</sub>	10 %
Cu <sup>2+</sup>	250	Pb <sup>2+</sup>	25	Na <sub>2</sub> SO <sub>4</sub>	10 %
F <sup>-</sup>	50	S <sub>2</sub> <sup>-</sup>	2.5		
SiO <sub>3</sub> <sup>2-</sup>	1000	Zn <sup>2+</sup>	1000		
SO <sub>3</sub> <sup>2-</sup>	1000				

Sample for phosphate analysis must be pretreated by filtration (0.45µm) to remove most of turbidity (interferes with photometric measurement)

In case of total P sample mustn't be filtrated! The filtration step would remove already precipitated struvite during urine storage and thus false the analysis

In any case urine should be diluted at least 1:100 to avoid matrix effects

#### Sampling

Preferably collect samples in glass bottles.

Analyze immediately after sampling.

## Safety Precautions

Handle concentrated acid with care

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 used gloves after completion of analysis

Clean hands using antiseptic soap and disinfect with ethanol

Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

## Apparatus

Heating Block for Total P measurement

Spectrophotometer

Glass ware: Use acid washed glassware for determining low concentrations of orthophosphates. Phosphate contamination is common because of its absorption on glass surfaces. Avoid using commercial detergents containing phosphate. Clean all glassware with hot dilute HCL and rinse well with distilled water. Preferably reserve the glassware only for phosphate determination and after use, wash and keep filled with water until needed. If this is done, acid treatment is required only occasionally.

## Reagents

Sulphuric acid: 10-15% concentration (phosphate test)

Sodium nitrate: 50 – 100 % concentration (total P test)

Potassium persulfate: 25-100% concentration (total P test)

## Calibration

To check the photometric measurement system (test reagents, measurement device, handling) and the mode of working, Spectroquant® CombiCheck 10 can be used. Besides a standard solution with 0.80 mg/l PO<sub>4</sub>-P, the CombiCheck 10 also contains an addition solution for determining sample-dependent interferences (matrix effects).

## Procedure

Note: Procedures according to Merck operational Manual for test kits (Phosphate 1.14848.0001 and total P 1.14543.0001)

Ortho-Phosphate measurement:

Pipette 5.0 ml pretreated sample into a test tube.

Reagent PO<sub>4</sub>-1 5 drops Add and mix.

Reagent PO<sub>4</sub>-2 1 level blue microspoon, add and shake vigorously until the reagent is completely dissolved

Leave to stand for 5 min (reaction time), then fill the sample into the cell, and measure in the photometer.

Total P measurement:

Digestion for the determination of total phosphorus (Wear eye protection!):

Pipette 5.0 ml pretreated sample into a reaction cell

Add 1 dose Reagent P-1K, close the cell tightly, and mix.

Heat the cell at 120 °C in the preheated thermoreactor for 30 min.

Allow the closed cell to cool to room temperature in a test-tube rack.

Do not cool with cold water!

Shake the tightly closed cell vigorously after cooling.

Add 5 drops reagent P-2K, close the cell tightly, and mix.

Add 1 dose reagent P-3K, close the cell tightly, and shake vigorously until the reagent is completely dissolved.

Leave to stand for 5 min (reaction time), then measure the sample in the photometer

Notes on the measurement:

For photometric measurement the cells must be clean. Wipe, if necessary, with a clean, dry paper towel.

Measurement of turbid solutions yields false-high readings.

The pH of the measurement solution must be within the range 0.80 - 0.95.

The colour of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.

#### Data Quality

Measurement	0.05 – 5.0 mg/l PO <sub>4</sub> -P
Standard Deviation (mg/l PO <sub>4</sub> P)	± 0.024
Confidence Interval (mg/l PO <sub>4</sub> P)	± 0.04
Sensitivity (mg/l PO <sub>4</sub> -P)	0.02

Accuracy (mg/l PO<sub>4</sub>-P) ± 0.06

#### Chemical Waste Disposal

Dilute 10 ml into 1000ml.

Slowly add NaCO<sub>3</sub> until ph 6-8 is reached.

Flush down the sink with excess water

### Appendix 7.1.6. Standard operation procedure – Potassium cell test (cat. no. 1.14562)

#### Scope and Field of Application

Measuring range 5.0 – 50 mg/l K.

#### Principle

In alkaline solution, potassium ions react with Kalignost (sodium tetraphenyl-borate) to form a slightly soluble precipitate. The resulting turbidity is measured in the photometer (turbidimetric method).

#### Interferences

Concentrations of foreign substances in mg/l or %					
Al <sup>3+</sup>	1000	Hg <sup>2+</sup>	100	EDTA	10 %
Ca <sup>2+</sup>	1000	Mg <sup>2+</sup>	1000	Surfactants	250
Cd <sup>2+</sup>	1000	Mn <sup>2+</sup>	1000	Oxidising Agents	1000
S <sub>2</sub> <sup>-</sup>	1000	NH <sub>4</sub> <sup>+</sup>	50	Na-acetate	20 %
CN <sup>-</sup>	100	Ni <sup>2+</sup>	1000	NaCl	20 %
Cr <sup>3+</sup>	10	NO <sub>2</sub> <sup>-</sup>	1000	NaNO <sub>3</sub>	20 %
Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	1000	Pb <sup>2+</sup>	1000	Na <sub>2</sub> SO <sub>4</sub>	20 %
Cu <sup>2+</sup>	100	PO <sub>4</sub> <sup>3-</sup>	1000		
Zn <sup>2+</sup>	1000	SiO <sub>3</sub> <sup>2-</sup>	1000		
Fe <sup>3+</sup>	1000	SO <sub>3</sub> <sup>2-</sup>	1000		

#### Sampling

Analyze immediately after sampling.

Use the Merckoquant Ammonium Test to check ammonium content. Samples containing more than 50mg/l  $\text{NH}_4^+$  (Cat. No. 114562) must be diluted with distilled water.

Check the potassium content with a Merckoquant Potassium Test. Samples containing more than 50 mg/l K (Cat. No. 114562) must be diluted with distilled water.

The pH must be within the range 3 – 12, adjust with NaOH (1 mol/l) or  $\text{H}_2\text{SO}_4$  (0.5 mol/l)

Turbid sampled must be filtered

Safety Precautions

Handle concentrated sulphuric acid with care.

Always use safety goggles, gloves and laboratory coat while working in laboratory

Wear face shield and protect hands from heat produced when contents of the vessels are mixed.

After the analysis clean bottles and beakers with water then dry

Dispose the used gloves after completion of analysis

Clean the hands using antiseptic soap

Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

Reclose the reagent bottles immediately after use

The test reagents must not be run off with the wastewater

Reagents

Reagent K-1K

Reagent K-2K

Universal indicator strips pH 0 - 14, Cat. No. 109535

Alkalit indicator strips pH 7.5 – 14 (Cat. No. 109532)

Sodium Hydroxide solution 1 mol/l

Sulphuric acid 0.5 mol/l

Standard potassium solution 1000 mg/l

Apparatus

Spectroquant

Reaction Cells

## Pipettes

### Calibration

To check the measurement system (test reagents, measurement device, and handling) and the mode of working, a dilute potassium standard solution containing 25.0 mg/l K can be used.

### Procedure

Pipette 2.0ml of the pretreated sample into a reaction cell, close the cell, and mix.

The pH must be within the range 10 – 11.5, adjust with NaOH solution if necessary

Add 6 drops of reagent K-1K and mix

Add 1 level microspoon of reagent K-2K and dissolve by closing and shaking the cell.

Leave to stand for exactly 5 minutes, then measure the sample in the photometer.

Notes on the measurement:

For turbidimetric measurement, the cells must be clean. Wipe if necessary with clean dry paper towel.

The turbidity of the measurement solution remains stable for only a short time (the measurement value increases by 5-7% per minute)

### Data Quality

Measurement	0.1 – 5.0 mg/l K
Standard Deviation (mg/l K)	± 0.71
Confidence Interval (mg/l K)	± 1.7
Sensitivity (mg/l K)	0.3
Accuracy (mg/l NO <sub>3</sub> -N)	± 2.4

### Chemical Waste Disposal

Collect waste in a labeled 2.5L bottle for collection from Waste Tech. Do not run off with waste water.

 UNIVERSITY OF KWAZULU-NATAL	<b>Standard Operating Procedure</b>  pollution research group	Effective Date: 17 Feb 2014	Version: <b>002</b>
		Reviewed: January 2021	

## Appendix 7.1.7. Standard Operation Procedure – Thermal Balance for Drying Tests

### Scope and Application

A moisture analyser is designed to determine relative moisture content in small samples of various substances, by measuring the change of weight due to water evaporation during convective drying. This method is applicable for all types of sludge – liquid, slurry, semi-solid and solid; however, samples with a higher moisture content will have a longer drying and measurement time.

Default password: 319039

### Summary

Before using a moisture analyser, make sure the instrument was left on power for a sufficient period of time (mentioned in the user manual).

Minimize external environmental influences such as air draft, vibrations, or direct sunlight.

Ensure the analyser is level. This is essential for testing liquid samples, which must be at uniform level in the sample pan

Do not place any flammable substances on or near the moisture analyser, because the area around the heating unit will heat up.

### Collection, Preservation and Storage

Collect samples in 1L plastic buckets.

Preferably, analyse samples immediately after sampling.

Store samples at 4 °C or freeze dry samples.

Preserve wastewater samples by acidifying with concentrated sulphuric acid to pH 2 and faecal samples by freeze drying or freezing.

Determine COD on well- homogenised samples.

#### Safety Precautions

General health and safety (H&S) procedures specific for conducting laboratory analysis of faecal sludge are to be followed. It is important to be familiar with these to ensure safety measures are properly carried out.

Appropriate personal protective equipment (PPE) should be used

Wear gloves suitable for withstanding high temperatures when removing crucibles from the oven.

Always conduct the total solids analysis in a room with sufficient airflow and an exhaust system.

Do not place any flammable substances on or near the moisture analyser.

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

#### Operation Procedure for Temp Change

Press “Test Menu”

Move arrow to the profile icon(top left)

Press the right arrow until the T blinks and use the top/bottom arrow to adjust to required temp.

Press enter to set T.

#### Quality control

General information on quality assurance and quality control (QA/QC) should be followed.

Before using a moisture analyser, make sure the instrument was left on for a sufficient period of time.

Minimise external environmental influences such as air draught, vibrations, or direct sunlight.



Ensure the analyser is levelled. This is essential for testing liquid samples, which must be at uniform level in the sample container.

Exclude larger, inconsistent or floating particles from the sample if it is determined that their inclusion could affect the final result (e.g. hair, stones, glass and maggots). Disperse visible floating oil and grease with a blender or stainless steel mixing rod before withdrawing a sample portion for analysis.

#### Sample preservation

Samples should be analysed as soon as possible. If samples cannot be analysed immediately, they should be stored in a refrigerator at 4 °C for no longer than 7 days. Before analysis, let the samples return to ambient temperature. Do not freeze the samples.

#### Sample preparation

Uniformly mix all the samples using a stainless steel rod (or other appropriate tool) in order to have well mixed representative samples. If desired, samples can also be blended.

#### Operating Procedure

##### Equipment preparation

Switch the instrument on. Wait until the analyser completes its self-examination and finishes heating up. To deliver accurate results and enable the moisture analyser to reach the required operating temperature, it must be switched on for at least 20-30 minutes every time before use. The program must be set to end when the sample mass changes less than 0.05% of mass per minute.

Check that the temperature is 105 °C for moisture analysis.

##### Procedure

Press 'Start Program' and follow prompts on the display screen; this can vary per model and brand.

Open the lid of the moisture analyser, place the clean and empty weighing boat on the weighing cradle.

Close the cover gently and tare the boat weight; the LCD screen should now show weight as '0' and a flashing icon to indicate that the machine is ready for loading the sample.

Lift the lid of the moisture analyser and then evenly spread 1-3 g of the wet sample on the weighing boat.

Close the cover gently.

The halogen light will start to heat the sample until it reaches a steady reading. Note: this process usually takes between 2-15 min, depending on the sample weight and its moisture content.

Record the moisture reading (before lifting the lid); this is the end of the drying procedure.

Press 'Stop' and lift the lid to end the current testing.

Clean the weigh boat for future use

#### Reference

ASTM (2015). Standard Test Method for Determination of Total Solids in Biomass (E1756–08), Method B.

## Appendix 7.2. Nutrient Results

### 7.2.1. Nutrient concentration summary (g/kg dry basis)

		Nutrients															
		g/kg dry sample										(%)					
temp	SAMPLE name	PHOSPHATE	confidence	ORTHO PHOSPHATE	confidence	NITROGEN	confidence	AMMONIUM	confidence	NITRITE	confidence	NITRATE	confidence	POTASSIUM	confidence	MOISTURE	confidence
		interah/-		interah/-		interah/-		interah/-		interah/-		interah/-		interah/-		interah/-	
0	initial	16,3433	0,2557	45,3002	1,0963	29,1603	1,2787	9,6297	0,4611	0,1967	0,0157	0,0000	0,0000	9,4262	0,0302	76,373	1,122
	50%	15,2131	0,0653	23,9459	0,5697	27,0301	0,0000	4,5397	0,1729	0,2149	0,0065	1,2475	0,3395	9,0100	0,0312	52,318	2,563
	25%	25,6590	0,0408	13,3487	0,6173	20,5532	0,4080	4,3486	0,1413	0,0627	0,0041	0,0000	0,0000	7,3126	0,0825	23,572	6,408
50°C	0%	14,0373	0,3213	13,0131	0,5602	23,6754	0,0000	3,6772	0,0548	0,0386	0,0032	0,7220	0,2592	6,5317	0,0304	3,963	0,139
	50%	11,1250	0,0000	19,7109	0,2746	23,7199	0,6300	4,6438	0,1260	0,1737	0,0126	1,0357	0,0630	7,7841	0,0312	50,540	2,870
	25%	13,0201	0,0412	13,8940	0,3973	19,6613	0,0000	3,8886	0,0824	0,0743	0,0082	0,5461	0,0412	7,6897	0,0623	24,418	2,767
100°C	0%	21,4718	1,3009	10,9883	0,0635	25,9142	2,0807	2,4568	0,0839	0,0303	0,0000	0,0000	0,0000	7,2863	0,0314	0,960	0,519
	50%	11,5967	0,0611	18,0429	0,1616	26,2383	0,0000	4,6322	0,0611	0,1684	0,0122	0,8422	0,1222	9,0052	0,0827	48,843	2,500
200°C	25%	10,6169	0,0401	11,2552	0,0802	13,1914	0,4012	3,2127	0,0401	0,0468	0,0040	0,0000	0,0000	8,5106	0,1129	21,951	2,370
50°C	0%	12,8926	0,3487	9,2114	0,1268	19,6667	0,0000	1,0422	0,2594	0,0151	0,0000	0,0000	0,0000	7,9843	0,0312	1,669	0,354

## 7.2.2 Nitrogen results (wet basis)

Name of Test	NITROGEN									
Sample Name		Conc. (mg/l)	Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg / mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval +/-	mg/kg wet sample	Std deviation	
VIP Initial	1-a	14,0	0,0005	0,0067	0,0067	0,0069	0,0003	6889,7007	0,0003	
	1-b	14,0	0,0005	0,0067	0,0067					
	1-c	15,0	0,0005	0,0072	0,0072					
VIP:50°C 50%	1-a	26,0	0,0005	0,0129	0,0129	0,0129	0,0000	12888,5144	0,0000	
	1-b	26,0	0,0005	0,0129	0,0129					
	1-c	26,0	0,0005	0,0129	0,0129					
VIP:50°C 25%	1-a	32,0	0,0005	0,0159	0,0159	0,0157	0,0003	15708,4511	0,0003	
	1-b	31,0	0,0005	0,0154	0,0154					
	1-c	32,0	0,0005	0,0159	0,0159					
VIP:50°C 0%	1-a	47,0	0,0005	0,0227	0,0227	0,0227	0,0000	22737,1680	0,0000	
	1-b	47,0	0,0005	0,0227	0,0227					
	1-c	47,0	0,0005	0,0227	0,0227					
VIP:100°C 50%	1-a	23,0	0,0005	0,0114	0,0114	0,0117	0,0003	11731,8528	0,0003	
	1-b	24,0	0,0005	0,0119	0,0119					
	1-c	24,0	0,0005	0,0119	0,0119					
VIP:100°C 25%	1-a	30,0	0,0005	0,0149	0,0149	0,0149	0,0000	14860,3131	0,0000	
	1-b	30,0	0,0005	0,0149	0,0149					
	1-c	30,0	0,0005	0,0149	0,0149					
VIP:100°C 0%	1-a	54,0	0,0005	0,0270	0,0270	0,0257	0,0021	25665,3834	0,0019	
	1-b	53,0	0,0005	0,0265	0,0265					
	1-c	47,0	0,0005	0,0235	0,0235					
VIP:150°C 0%	1-a	39,0	0,0005	0,0193	0,0193	0,0193	0,0000	19338,5233	0,0000	
	1-b	39,0	0,0005	0,0193	0,0193					
	1-c	39,0	0,0005	0,0193	0,0193					
VIP:200°C 50%	1-a	27,0	0,0005	0,0134	0,0134	0,0134	0,0000	13422,8188	0,0000	
	1-b	27,0	0,0005	0,0134	0,0134					
	1-c	27,0	0,0005	0,0134	0,0134					
VIP:200°C 25%	1-a	21,0	0,0005	0,0105	0,0105	0,0103	0,0003	10295,7538	0,0003	
	1-b	21,0	0,0005	0,0105	0,0105					
	1-c	20,0	0,0005	0,0100	0,0100					

### 7.2.3. Ammonium results (wet basis)

Name of Test	AMMONIUM																
Sample Name		Conc. (mg/l)	Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg/mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval +/-	mg/kg wet sample	Std deviation								
VIP Initial	1-a	4,9	0,0005	0,0024	0,0024	0,0023	0,0001	2275,2035	0,0001								
	1-b	4,5	0,0005	0,0022	0,0022												
	1-c	4,8	0,0005	0,0023	0,0023												
VIP:50°C 50%	1-a	4,5	0,0005	0,0022	0,0022	0,0022	0,0001	2164,6095	0,0001								
	1-b	4,4	0,0005	0,0022	0,0022												
	1-c	4,2	0,0005	0,0021	0,0021												
VIP:50°C 25%	1-a	6,5	0,0005	0,0032	0,0032	0,0033	0,0001	3323,5776	0,0001								
	1-b	6,7	0,0005	0,0033	0,0033												
	1-c	6,9	0,0005	0,0034	0,0034												
VIP:50°C 0%	1-a	7,2	0,0005	0,0035	0,0035	0,0035	0,0001	3531,5176	0,0000								
	1-b	7,4	0,0005	0,0036	0,0036												
	1-c	7,3	0,0005	0,0035	0,0035												
VIP:100°C 50%	1-a	4,7	0,0005	0,0023	0,0023	0,0023	0,0001	2296,7994	0,0001								
	1-b	4,7	0,0005	0,0023	0,0023												
	1-c	4,5	0,0005	0,0022	0,0022												
VIP:100°C 25%	1-a	5,8	0,0005	0,0029	0,0029	0,0029	0,0001	2939,0397	0,0001								
	1-b	6,0	0,0005	0,0030	0,0030												
	1-c	6,0	0,0005	0,0030	0,0030												
VIP:100°C 0%	1-a	4,7	0,0005	0,0023	0,0023	0,0024	0,0001	2433,2117	0,0001								
	1-b	4,9	0,0005	0,0024	0,0024												
	1-c	5,0	0,0005	0,0025	0,0025												
VIP:150°C 0%	1-a	1,7	0,0005	0,0008	0,0008	0,0010	0,0003	1024,7764	0,0002								
	1-b	1,9	0,0005	0,0009	0,0009												
	1-c	2,6	0,0005	0,0013	0,0013												
VIP:200°C 50%	1-a	4,8	0,0005	0,0024	0,0024	0,0024	0,0000	2369,7075	0,0000								
	1-b	4,7	0,0005	0,0023	0,0023												
	1-c	4,8	0,0005	0,0024	0,0024												
VIP:200°C 25%	1-a	5,0	0,0005	0,0025	0,0025	0,0025	0,0000	2507,5142	0,0000								
	1-b	5,0	0,0005	0,0025	0,0025												
	1-c	5,1	0,0005	0,0025	0,0025												

### 7.2.4. Nitrite results (wet basis)

Name of Test	NITRITE											
Sample Name		Conc. (mg/l)	Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg/mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval +/-	mg/kg wet sample	Std deviation			
VIP Initial	1-a	0.09	0.0005	0,000043	0,000043	0,000046	0,000004	46,4654	0,000003			
	1-b	0.10	0.0005	0,000048	0,000048							
	1-c	0.10	0.0005	0,000048	0,000048							
VIP:50°C 50%	1-a	0.20	0.0005	0,000099	0,000099	0,000102	0,000003	102,4472	0,000003			
	1-b	0.21	0.0005	0,000104	0,000104							
	1-c	0.21	0.0005	0,000104	0,000104							
VIP:50°C 25%	1-a	0.09	0.0005	0,000045	0,000045	0,000048	0,000003	47,9521	0,000003			
	1-b	0.10	0.0005	0,000050	0,000050							
	1-c	0.10	0.0005	0,000050	0,000050							
VIP:50°C 0%	1-a	0.07	0.0005	0,000034	0,000034	0,000037	0,000003	37,0890	0,000003			
	1-b	0.08	0.0005	0,000039	0,000039							
	1-c	0.08	0.0005	0,000039	0,000039							
VIP:100°C 50%	1-a	0.16	0.0005	0,000079	0,000079	0,000086	0,000006	85,9234	0,000006			
	1-b	0.18	0.0005	0,000089	0,000089							
	1-c	0.18	0.0005	0,000089	0,000089							
VIP:100°C 25%	1-a	0.10	0.0005	0,000050	0,000050	0,000056	0,000006	56,1390	0,000006			
	1-b	0.12	0.0005	0,000059	0,000059							
	1-c	0.12	0.0005	0,000059	0,000059							
VIP:100°C 0%	1-a	0.06	0.0005	0,000030	0,000030	0,000030	0,000000	29,9985	0,000000			
	1-b	0.06	0.0005	0,000030	0,000030							
	1-c	0.06	0.0005	0,000030	0,000030							
VIP:150°C 0%	1-a	0.03	0.0005	0,000015	0,000015	0,000015	0,000000	14,8758	0,000000			
	1-b	0.03	0.0005	0,000015	0,000015							
	1-c	0.03	0.0005	0,000015	0,000015							
VIP:200°C 50%	1-a	0.16	0.0005	0,000080	0,000080	0,000086	0,000006	86,1712	0,000006			
	1-b	0.18	0.0005	0,000089	0,000089							
	1-c	0.18	0.0005	0,000089	0,000089							
VIP:200°C 25%	1-a	0.07	0.0005	0,000035	0,000035	0,000037	0,000003	36,5333	0,000003			
	1-b	0.07	0.0005	0,000035	0,000035							
	1-c	0.08	0.0005	0,000040	0,000040							

### 7.2.5. Nitrate results (wet basis)

Name of Test	NITRATE										
Sample Name	Conc. (mg/l)		Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg / mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval+/-	mg/kg wet sample	Std deviation		
VIP Initial	1-a	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-b	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-c	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
VIP:50°C 50%	1-a	1,5	0,0005	0,0007	0,0007	0,0006	0,0002	594,8545	0,0001		
	1-b	1,2	0,0005	0,0006	0,0006	0,0006	0,0002	594,8545	0,0001		
	1-c	0,9	0,0005	0,0004	0,0004	0,0004	0,0002	594,8545	0,0001		
VIP:50°C 25%	1-a	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-b	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-c	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
VIP:100°C 0%	1-a	0,9	0,0005	0,0004	0,0004	0,0005	0,0000	512,2358	0,0000		
	1-b	0,8	0,0005	0,0004	0,0004	0,0004	0,0000	412,7865	0,0000		
	1-c	0,8	0,0005	0,0004	0,0004	0,0004	0,0000	412,7865	0,0000		
VIP:100°C 25%	1-a	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-b	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-c	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
VIP:150°C 0%	1-a	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-b	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-c	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
VIP:200°C 50%	1-a	1,0	0,0005	0,0005	0,0005	0,0005	0,0001	430,8559	0,0001		
	1-b	0,8	0,0005	0,0004	0,0004	0,0004	0,0001	430,8559	0,0001		
	1-c	0,8	0,0005	0,0004	0,0004	0,0004	0,0001	430,8559	0,0001		
VIP:200°C 25%	1-a	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-b	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		
	1-c	0,0	0,0005	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000		

## 7.2.6. Phosphate results (wet basis)

Name of Test		PHOSPHATE							
Sample Name		Conc. (mg/l)	Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg / mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval +/-	mg/kg wet sample	Std deviation
VIP initial	1-a		7,9	0,0038	0,0038				
	1-b		8,1	0,0039	0,0039				
	1-c		8,1	0,0039	0,0039	0,0039	0,0001	3861,4369	0,0001
VIP:50°C 50%	1-a		14,7	0,0073	0,0073				
	1-b		14,6	0,0072	0,0072				
	1-c		14,6	0,0072	0,0072	0,0073	0,0000	7253,9203	0,0000
VIP:50°C 25%	1-a		39,6	0,0196	0,0196				
	1-b		39,5	0,0196	0,0196				
	1-c		39,5	0,0196	0,0196	0,0196	0,0000	19610,7611	0,0000
VIP:50°C 0%	1-a		28,3	0,0137	0,0137				
	1-b		28,1	0,0136	0,0136				
	1-c		27,2	0,0132	0,0132	0,0135	0,0003	13481,0443	0,0003
VIP:100°C 50%	1-a		11,1	0,0055	0,0055				
	1-b		11,1	0,0055	0,0055				
	1-c		11,1	0,0055	0,0055	0,0055	0,0000	5502,4042	0,0000
VIP:100°C 25%	1-a		19,9	0,0099	0,0099				
	1-b		19,9	0,0099	0,0099				
	1-c		19,8	0,0098	0,0098	0,0098	0,0000	9840,8295	0,0000
VIP:100°C 0%	1-a		43,9	0,0219	0,0219				
	1-b		39,8	0,0199	0,0199				
	1-c		43,9	0,0219	0,0219	0,0213	0,0013	21265,6034	0,0012
VIP:150°C 0%	1-a		26,3	0,0130	0,0130				
	1-b		25,2	0,0125	0,0125				
	1-c		25,2	0,0125	0,0125	0,0127	0,0003	12677,4764	0,0003
VIP:200°C 50%	1-a		12,0	0,0060	0,0060				
	1-b		11,9	0,0059	0,0059				
	1-c		11,9	0,0059	0,0059	0,0059	0,0000	5932,5545	0,0000
VIP:200°C 25%	1-a		16,7	0,0083	0,0083				
	1-b		16,6	0,0083	0,0083				
	1-c		16,6	0,0083	0,0083	0,0083	0,0000	8286,4212	0,0000



### 7.2.7. Orthophospahte results (wet basis)

Name of Test	ORTHO-PHOSPHATE	Name of Test	ORTHO-PHOSPHATE						
Sample Name		Conc. (mg/l)	Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg / mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval +/-	mg/kg wet sample	Std deviation
VIP Initial	1-a	21,8	0,0005	0,0105	0,0105	0,0107	0,0003	10703,0699	0,0002
	1-b	22,5	0,0005	0,0108	0,0108				
	1-c	22,5	0,0005	0,0108	0,0108				
VIP:50°C 50%	1-a	23,5	0,0005	0,0116	0,0116	0,0114	0,0003	11417,9018	0,0002
	1-b	23,1	0,0005	0,0115	0,0115				
	1-c	22,5	0,0005	0,0112	0,0112				
VIP:50°C 25%	1-a	21,3	0,0005	0,0106	0,0106	0,0102	0,0005	10202,2256	0,0004
	1-b	19,6	0,0005	0,0097	0,0097				
	1-c	20,8	0,0005	0,0103	0,0103				
VIP:50°C 0%	1-a	27,0	0,0005	0,0131	0,0131	0,0125	0,0005	12497,3796	0,0005
	1-b	25,1	0,0005	0,0121	0,0121				
	1-c	25,4	0,0005	0,0123	0,0123				
VIP:100°C 50%	1-a	19,9	0,0005	0,0099	0,0099	0,0097	0,0001	9749,0044	0,0001
	1-b	19,7	0,0005	0,0098	0,0098				
	1-c	19,4	0,0005	0,0096	0,0096				
VIP:100°C 25%	1-a	21,8	0,0005	0,0108	0,0108	0,0105	0,0003	10501,2879	0,0003
	1-b	21,1	0,0005	0,0105	0,0105				
	1-c	20,7	0,0005	0,0103	0,0103				
VIP:100°C 0%	1-a	21,7	0,0005	0,0108	0,0108	0,0109	0,0001	10882,7892	0,0001
	1-b	21,7	0,0005	0,0108	0,0108				
	1-c	21,9	0,0005	0,0109	0,0109				
VIP:150°C 0%	1-a	18,4	0,0005	0,0091	0,0091	0,0091	0,0001	9057,7015	0,0001
	1-b	18,4	0,0005	0,0091	0,0091				
	1-c	18,0	0,0005	0,0089	0,0089				
VIP:200°C 50%	1-a	18,6	0,0005	0,0092	0,0092	0,0092	0,0001	9230,2593	0,0001
	1-b	18,7	0,0005	0,0093	0,0093				
	1-c	18,4	0,0005	0,0091	0,0091				
VIP:200°C 25%	1-a	17,7	0,0005	0,0088	0,0088	0,0088	0,0001	8784,6029	0,0001
	1-b	17,7	0,0005	0,0088	0,0088				
	1-c	17,5	0,0005	0,0087	0,0087				

## 7.2.8. Potassium

Name of Test	POTASSIUM	Name of Test	POTASSIUM						
Sample Name		Conc. (mg/l)	Dilution 1 (L/mg) for diluted wet samples	mg/mg wet sample	Final result (mg / mg wet sample) XDF	Average (mg/mg wet sample)	80% confidence interval +/-	mg/kg wet sample	Std deviation
VIP Initial	1-a	4,7	0,0005	0,0023	0,0023	0,0022	0,0000	2227,1358	0,0000
	1-b	4,6	0,0005	0,0022	0,0022	0,0022	0,0000	2227,1358	0,0000
	1-c	4,6	0,0005	0,0022	0,0022	0,0022	0,0000	2227,1358	0,0000
VIP:50°C 50%	1-a	8,6	0,0005	0,0043	0,0043	0,0043	0,0000	4296,1715	0,0000
	1-b	8,7	0,0005	0,0043	0,0043	0,0043	0,0000	4296,1715	0,0000
	1-c	8,7	0,0005	0,0043	0,0043	0,0043	0,0000	4296,1715	0,0000
VIP:50°C 25%	1-a	11,1	0,0005	0,0055	0,0055	0,0056	0,0001	5588,9016	0,0001
	1-b	11,3	0,0005	0,0056	0,0056	0,0056	0,0001	5588,9016	0,0001
	1-c	11,4	0,0005	0,0057	0,0057	0,0056	0,0001	5588,9016	0,0001
VIP:50°C 0%	1-a	13,0	0,0005	0,0063	0,0063	0,0063	0,0000	6272,8783	0,0000
	1-b	12,9	0,0005	0,0062	0,0062	0,0063	0,0000	6272,8783	0,0000
	1-c	13,0	0,0005	0,0063	0,0063	0,0063	0,0000	6272,8783	0,0000
VIP:100°C 50%	1-a	7,7	0,0005	0,0038	0,0038	0,0039	0,0000	3850,0306	0,0000
	1-b	7,8	0,0005	0,0039	0,0039	0,0039	0,0000	3850,0306	0,0000
	1-c	7,8	0,0005	0,0039	0,0039	0,0039	0,0000	3850,0306	0,0000
VIP:100°C 25%	1-a	11,8	0,0005	0,0058	0,0058	0,0058	0,0001	5812,0336	0,0001
	1-b	11,8	0,0005	0,0058	0,0058	0,0058	0,0001	5812,0336	0,0001
	1-c	11,6	0,0005	0,0057	0,0057	0,0058	0,0001	5812,0336	0,0001
VIP:100°C 0%	1-a	14,4	0,0005	0,0072	0,0072	0,0072	0,0000	7216,3059	0,0000
	1-b	14,4	0,0005	0,0072	0,0072	0,0072	0,0000	7216,3059	0,0000
	1-c	14,5	0,0005	0,0072	0,0072	0,0072	0,0000	7216,3059	0,0000
VIP:150°C 0%	1-a	15,8	0,0005	0,0078	0,0078	0,0079	0,0000	7851,1099	0,0000
	1-b	15,9	0,0005	0,0079	0,0079	0,0079	0,0000	7851,1099	0,0000
	1-c	15,8	0,0005	0,0078	0,0078	0,0079	0,0000	7851,1099	0,0000
VIP:200°C 50%	1-a	9,4	0,0005	0,0047	0,0047	0,0046	0,0001	4606,8440	0,0001
	1-b	9,3	0,0005	0,0046	0,0046	0,0046	0,0001	4606,8440	0,0001
	1-c	9,1	0,0005	0,0045	0,0045	0,0046	0,0001	4606,8440	0,0001
VIP:200°C 25%	1-a	13,5	0,0005	0,0067	0,0067	0,0066	0,0001	6642,4218	0,0001
	1-b	13,4	0,0005	0,0067	0,0067	0,0066	0,0001	6642,4218	0,0001
	1-c	13,1	0,0005	0,0065	0,0065	0,0066	0,0001	6642,4218	0,0001

### 7.3. Moisture Content Results

SAMPLE	REPLICATE	SAMPLE MASS FINAL (g)	MOISTURE CONTENT (%)	AVERAGE MOISTURE CONTENT (%)	80% confidence Interval +/-	Std deviation	DURATION (min)
VIP Initial	1	0.250	76.170	76.373	1.122	1.031	13:54
	2	0.361	75.459				14:29
	3	0.361	77.490				13:08
VIP:50°C 50%	1	0.223	49.663	52.318	2.563	2.355	12:07
	2	0.276	54.153				09:55
	3	0.223	53.138				13:20
VIP:50°C 25%	1	0.324	28.976	23.572	6.408	5.886	11:36
	2	0.198	17.300				07:40
	3	0.302	24.439				08:25
VIP:50°C 0%	1	0.604	3.968	3.963	0.139	0.128	02:20
	2	0.551	3.833				03:11
	3	0.610	4.088				03:15
VIP:100°C 50%	1	0.162	51.917	50.540	2.870	2.637	10:12
	2	0.141	52.203				08:45
	3	0.251	47.5				11:41
VIP:100°C 25%	1	0.236	22.112	24.418	2.767	2.541	07:18
	2	0.152	27.143				09:46
	3	0.189	24				07:22
VIP:100°C 0%	1	2.450	0.410	0.960	0.519	0.477	01:37
	2	0.474	1.253				01:55
	3	0.229	1.218				01:42
VIP:150°C 0%	1	0.546	1.444	1.669	0.354	0.325	01:37
	2	0.777	1.521				01:45
	3	0.672	2.041				01:38
VIP:200°C 50%	1	0.107	51.351	48.843	2.500	2.296	06:56
	2	0.124	48.333				05:34
	3	0.16	46.844				08:45
VIP:200°C 25%	1	0.225	20.567	21.951	2.370	2.177	07:32
	2	0.46	20.826				08:30
	3	0.205	24.46				08:50