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**Thermal Drying of Faecal Sludge from VIP latrines and
Characterisation of Dried Faecal Material**

By

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Examiner's Copy

December 2016

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ABSTRACT

Ensuring safe, adequate, effective and sustainable sanitation is a major global challenge, especially in developing countries. This project is part of the 'Reinvent the Toilet Challenge' (RTTC) which focuses on the development and implementation of new and sustainable technologies for processing human excreta from on-site sanitation systems. This project investigated the drying kinetics of pit latrine sludge by convective drying using different air properties and analysed the resulting thermal properties and nutrient content of the sludge.

The drying experiments were conducted at air temperatures of 40 °C, 60 °C and 80 °C; relative humidity of 5 %, 15 % and 25 %; effective air flow velocity of 0.03 cm/s, 0.06 cm/s and 0.12 cm/s. The drying curves obtained indicated two distinct stages during the drying process, a constant rate period and the falling rate period. It was observed that the drying kinetics were greatly affected by temperature, relative humidity and sample thickness but the effect of air flow rate seemed negligible in the range of study. The effective moisture diffusivity was found to range between 7.8×10^{-8} and 2.1×10^{-7} m²/s. The relationship between the effective moisture diffusivity and temperature was described well using the Arrhenius type equation. The concentration of nutrients was not affected by the conditions except for ammonia and nitrates which decreased greatly. The thermal conductivity of the wet sludge, of moisture content 79 % (wb), was 55 W/m.K and it showed a linear relationship with the moisture ratio of the sludge sample. The average thermal conductivity of the final dried product was 0.04 W/m.K. Heat capacity also showed great dependence on the moisture content of the sample but it was not a linear relationship. The calorific value of the dried samples was around 13 MJ/kg. The drying curves were regressed against common empirical drying models and the Page model described the experimental data well of the models analysed, yielding a R² greater than 0.996.

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LIST OF ABBREVIATIONS

BMGF	Bill & Merlinda Gates Foundation
RTTC	Reinvent the Toilet Challenge
LaDePa	Latrine Dehydration and Pasteurisation
MIR	Medium-infrared
PRG	Pollution Research Group
RH	Relative humidity
TKN	Total Kjeldahl Nitrogen
UD	Urine Diversion toilet
UKZN	University of KwaZulu-Natal
VIP	Ventilated Improved Pit
WWTW	Wastewater treatment works

NOMENCLATURE

A	Area	[m ²]
Ea	Activation energy	[kJ/ mol]
H	Absolute humidity	[kg/m ³]
H_s	Air saturation humidity	[kg/m ³]
h_t	Heat exchange coefficient	[W/m ² /K]
i	Sample number	[-]
M	Moisture content	[%]
N	Number of samples	[-]
R	Universal gas constant	[J/ (K.mol)]
T	Temperature	[K]
t	Time	[min]
TS	Total solids	[g]
VS	Volatile solids	[%]
w_f	Final weight of sample	[g]
w_{f1}	Mass after oven drying	[g]
w_{f2}	Mass after furnace combustion	[g]
w_i	Initial weight of sample	[g]
\bar{x}	Mean value	[-]
σ^2	Variance	[-]
p_A^S	Saturation partial pressure of substance A	[atm]
w	Mass	[g]

CHEMICAL NOMECLATURE

Ca Calcium

K Potassium

Mg Magnesium

NH_4^+ Ammonium

NO_2^- Nitrites

NO_3^- Nitrates

P Phosphorous

PO_4^{3-} Phosphates

1 INTRODUCTION

Ensuring safe, adequate, effective and sustainable sanitation is a major global challenge, especially in developing countries. The global sanitation community has made great efforts in trying to resolve the sanitation problem, hence the provision of basic sanitation was selected as one of the key issues in the United Nations Millennium Development Goals as well as the Sustainable Development Goals (SDGs) implemented in 2016 (Sachs, 2012). A report from the Joint Monitoring Programme for Water Supply and Sanitation, facilitated by the World Health Organisation (WHO), estimated that 2.1 billion people had gained access to an improved sanitation facility since 1990. However in the same report, in 2015, an estimated 2.4 billion people globally had no access to improved sanitation facilities, with over 946 million people practising open defecation, most of which reside in low and middle-income countries (WHO, 2015). The majority of the world's population, most of which are in Africa and Asia, do not rely on a piped sewer system but on on-site sanitation facilities such as septic tanks, pour-flush toilets, pit latrines and ventilated improved pit latrines. In most countries, ventilated improved pit latrines (VIP) have been deemed as the minimum acceptable level of sanitation and a fundamental human right (Langergraber and Muellegger, 2005, Bakare et al., 2012).

South Africa also faces the sanitation challenge, like the rest of the global community, as it is difficult to satisfy the sanitation needs for all residents using the conventional centralised sewerage system (Austin and Van Vuuren, 2001). In addition, increased urbanisation in major cities has resulted in the emergence of numerous informal settlements where sanitation facilities are few and overloaded or non-existent. Therefore, most municipalities within the country have adopted and implemented on-site sanitation systems. Over 3 million conventional pit latrines, septic tanks, Urine Diversion toilets (UD toilets) and, Ventilated Improved Pit latrines (VIP) have been built across most of the rural and the densely populated peri-urban areas of the country in order to address the sanitation problem in the country (Mnisi, 2011, Austin and Van Vuuren, 2001). Much attention has been placed on the building and provision of new toilets but not enough on their maintenance. Most of the toilets that were initially built in rural and peri-urban settlements are either full or are reaching their full capacity, which present a local health hazard leading to the need for them to be emptied (Still et al., 2005).

The major challenge facing most municipalities is finding an effective and sustainable faecal sludge management for the disposal of the sludge emptied from the full pits. Faecal sludge is normally dumped into the environment (landfilling/composting) or sent to a wastewater treatment plant for processing (Rose et al., 2015, O'Riordan, 2009, Arlabosse et al., 2004). These practices emanate from the general misperception that faecal sludge is waste without any value, whereas it has great valorisation potential. Most of the nutrients needed for crop production are found in excreta. Therefore, the nutrients in faecal sludge can be recycled for agricultural use, which could consequently reduce the use of chemical

fertilizers, usually produced from non-renewable resources (Rose et al., 2015, Malkki, 1999, Sharma Sunil, 2013). While faecal sludge use is primarily focused for agricultural applications, other products, such as biogas, represent potential revenue generating sources that have not yet been implemented (Langergraber and Muellegger, 2005, Muspratt et al., 2014a). Research has been conducted to assess the feasibility of faecal sludge as a fuel, a product that could offer an environmentally and financially beneficial solution for disposal-oriented faecal sludge management, while replacing the energy from fossil fuels which are in fast depletion (Fytili and Zabaniotou, 2008, Rose et al., 2015, Muspratt et al., 2014b).

Prior to faecal sludge reuse, drying is a critical and necessary treatment process. Traditionally, solar drying beds were employed in most treatment plants to dry sludge, mostly from wastewater treatment facilities. The major drawbacks facing this method is the large area of land required as well as the long drying time to attain the desired moisture content. As an alternative, thermal drying has gained popularity as it requires less surface area to process the same amount of sludge than drying beds technology, leading to more compact units, and additionally the process occurs faster. Thermal drying has been successfully implemented over the years in the drying of sludge from wastewater treatment facilities (Sapienza, 2005, Tao et al., 2005, Arlabosse et al., 2004).

1.1 Purpose of the study

The Pollution Research Group at the University of KwaZulu-Natal is among the grantees of the Bill & Melinda Gates Foundation (BMGF) initiative projects known as the ‘Reinvent the Toilet Challenge (RTTC), which focuses on the development and implementation of new sustainable technologies for the processing of human waste from on-site sanitation systems. The principle objective of the challenge is to develop a cost effective and self-sustaining sanitation process by developing a toilet that can integrate and utilize different excreta waste streams and recover valuable constituents such as energy, sterilised fertiliser and portable water. The toilet must cost less than 0.05 dollars per day per individual, should be out of the electric and sewage grid and it should be culturally accepted (Woolley et al., 2014, McCoy et al., 2009, Elledge and McClatchey, 2013).

Grantees from the RTTC, as well as other researchers in the sanitation field, have carried out research to evaluate resource recovery from innovative faecal sludge treatment processes to generate revenue that could make the sanitation system sustainable. Some of the proposed processes include gasification, combustion, pyrolysis, hydrothermal carbonization, anaerobic digestion and composting. Figure 1-1 shows a flowchart detailing the processing of various excreta streams and the possible end uses of the by-product. Some of grantees potentially interested in the outcomes from this study are tabulated in Table 1-1. In this light, drying of faecal sludge is a process of significant interest and relevance. It is on this basis that the objectives for the work were consolidated.

Table 1-1: Grantees of the RTTC project in need of data regarding the drying characteristics and thermal properties of faecal sludge

Grantee	Proposed technology
Research Triangle institute	Electrochemical disinfection, combustion, auger separation of liquid and solid heat conversion into electricity
Shijiazhuang University of Economics	Screw conveying and solar drying and disinfection
University of Toronto	Belt drying, sand filtering, smouldering and ultraviolet disinfection
Columbia University	Production of bio-diesel
Janicki Industries	Electricity producing facility that uses faecal sludge as a fuel source for steam generation

The understanding, characterisation and modelling of faecal sludge drying kinetics are essential for the design, operation, control and optimisation of the drying process. Nevertheless, the information pertaining to the drying of faecal sludge from on-site sanitation is not readily available in literature, thus forming the basis of this particular study.

1.2 Objectives of the study

This project investigated the drying characteristics of faecal sludge using a custom designed drying rig. The experimental rig enabled the determination of the drying kinetics at varying drying temperature, flow-rate and relative humidity. In addition, the chemical and physical properties of the dried faecal sludge were analysed to ascertain the effect of drying on the product.

The objectives of the present study include the investigation of the following parameters during experiments of convective drying of faecal sludge from VIP latrines:

1. Drying kinetics at varying air temperature, air flow-rate, air humidity and different sample geometry and size. This enables the calculation of the effective diffusivity and testing existing empirical models on faecal sludge convective drying.
2. Thermal properties of faecal sludge, namely thermal conductivity, caloric value and heat capacity, as a function of the drying conditions.
3. Nutrient content of the dried faecal sludge, namely potassium, total phosphorus, nitrates, magnesium, calcium, as a function of the drying conditions.

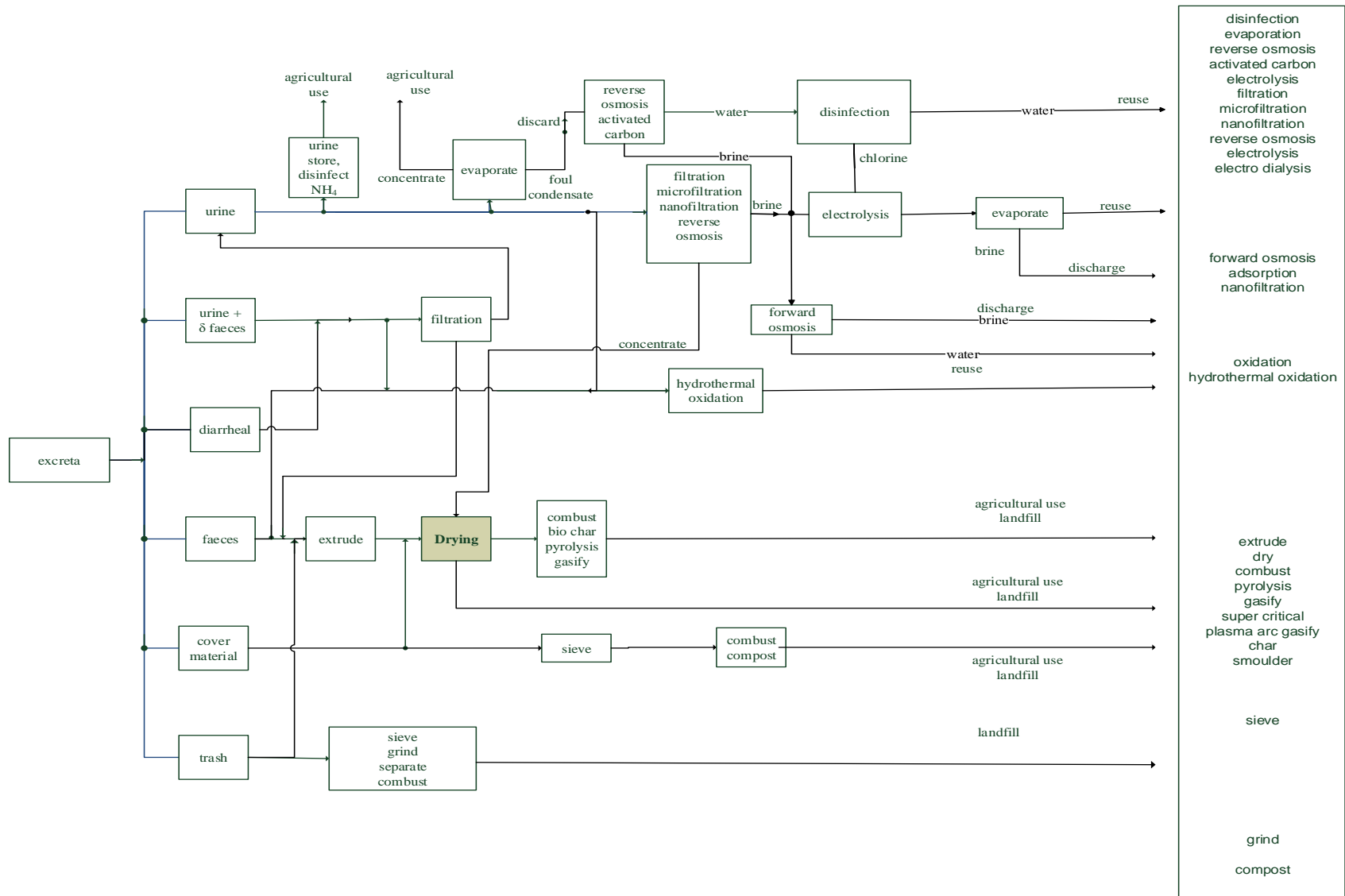


Figure 1-1: Flowchart indicating the possible uses of excreta (<http://prg.ukzn.ac.za>)

1.3 Dissertation outline

- **INTRODUCTION**

This section outlines the context and the motivation to perform the present study. The objectives of this work are indicated.

- **LITERATURE REVIEW**

This section is divided into 6 sub categories. Section 2.1 introduces the general sanitation concepts and the existing systems. Section 2.2 describes in detail the ventilated improved pit (VIP) latrines which is the sanitation system of concern in this project. The faecal sludge content of the VIP latrines is described, as well as the collection and disposal methods of the waste. Sections 2.3 and 2.4 focus on the drying theory, paying particular attention to the convective heat and mass transfer due to its relevance to the present project. The empirical modelling approaches are also discussed. Section 2.5 summarises the previous works conducted on drying of animal manure and the effects on the nutrient concentration.

- **MATERIALS AND METHODS**

This section outlines the experimental apparatus and methods employed in this project. It also gives a description of the parameters employed for data analysis.

- **RESULTS AND DISCUSSION**

The results obtained are presented in this section and discussed. The main trends deduced from the experimental results are highlighted and are compared to literature data.

- **CONCLUSION AND RECOMMENDATIONS**

The results obtained were summarised and their implication in the sanitation field, particularly in the context of the RTTC, were discussed in this section. The limitations of this study were presented and possible future researches on this topic were suggested.

2 LITERATURE REVIEW

The previous chapter highlighted the aims and objectives of this study. This chapter is aimed at outlining the relevant literature required to achieve the desired goals. The first section of this chapter briefly introduce sanitation systems and the management of faecal sludge from VIP latrines. Thereafter it focuses on drying theory which relates to the drying process and kinetics. Lastly, the effect of drying animal manure is reviewed.

2.1 Sanitation systems

There exist various types of sanitation facilities which are employed depending on the financial, social and geographical context. They can be broadly classified into two distinct categories, on-site and off-site sanitation facilities (Sharpe, 2010).

Off-site sanitation

This term is also referred to as centralized sanitation system because the waste is transported for processing from its generation point to a distant wastewater treatment facility through sewage network systems. This sanitation system is particularly common in urban areas of developed countries (Sharpe, 2010). Conventional off-site sanitation requires high amounts of water and energy used to transport excreta from the household toilet to the treatment plants, therefore it is neither an ecological nor economical solution in both industrialized and developing countries (Diaz and Barkdoll, 2006).

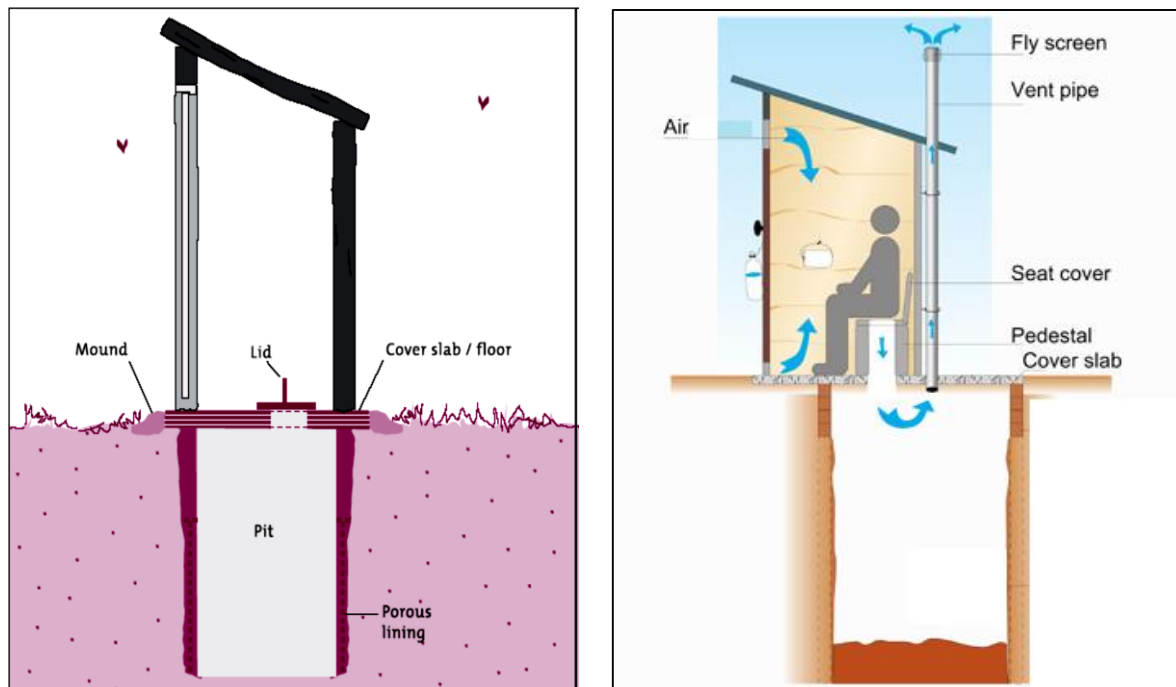
On-site sanitation

The majority of the world's population relies on on-site sanitation. This system is characterized by the containment of the human excreta at the proximity of the toilet. The various types of on-site sanitation methods that are available include: pit latrine, ventilated improved pit (VIP) latrine also known as a Blair toilet, septic tanks, unsewered public ablution blocks and urine diversion toilet (UD). The VIP latrines are considered as the basic minimum level of sanitation that a person should have access to by the South African government (Bakare, 2014). This is the main reason this project focuses particularly on the waste from the VIP latrines.

2.2 Ventilated improved pit (VIP) latrines

VIP latrines were designed in order to improve conventional pit latrines by reducing foul odours and the number of flies within the toilets. VIP latrines consist of an enclosed brick superstructure with a door, a pit with a concrete slab cover and a ventilation pipe, as shown in Figure 2-1b. The ventilation pipe serves to eliminate the malodorous air in the pit and prevent flies due to a fly screen that is located

at the end of the pipe (Bester and Austin, 2000). Human excreta are collected inside the pit and the liquid fraction leaches away into the underground whilst the bulk solid decomposes (Nwaneri, 2009).



(a) Traditional pit latrine

(b) Ventilated Improved Pit latrine (VIP)

Figure 2-1: Diagrams of a conventional pit latrine and a VIP toilet (Brikké and Bredero, 2003)

2.2.1 Description and characterisation of the contents of VIPs

The contents of VIP latrines comprise mainly of raw or semi degraded human excreta (faeces and urine), anal cleansing material and a wide variety of trash introduced in the pits by the toilet users, such as plastics, paper and clothes. Human faeces contain a large quantity of organic matter in comparison to urine. Nwaneri (2009) characterised and analysed the biodegradability of the organic material in human faeces and it was observed that 80 % of faeces contained biodegradable organic matter whilst the remainder was inert material.

Human excreta is rich in nutrients that are not assimilated during body metabolism. These nutrients can be utilised to supplement the synthetic fertilisers that are manufactured from fossil fuels and phosphorus. However, mismanagement or careless disposal of faecal sludge can have a detrimental effect on the environment as excess nutrients can lead to eutrophication, contamination of underground water and algal blooms in surface waters. Faecal sludge comprises of water-soluble and insoluble nutrients, most of which are essential for the proper growth of plants. These nutrients comprise of the main macronutrients which are nitrogen (N), phosphorus (P), potassium (K), calcium (Ca) and magnesium (Mg). Vinnerås et al. (2006) during their study on the composting of human excreta observed that urine contained the largest proportion of the total nutrients. They reported that urine

contains nearly 80% of the nitrogen (N), 50% of the phosphorus (P) and approximately 60% of the potassium (K). The amount of plant nutrients excreted in urine per person per year has been reported by Chaggu (2004) as 2.5 - 4.3 kg nitrogen, 0.4 - 1.0 kg phosphorus and 0.9 - 1.0 kg potassium. Concerning the nutrients in faeces, approximately 50% of nitrogen and most of the potassium in fresh faeces are soluble in water, while phosphorus is primarily found as calcium phosphate particles that have low solubility in water (Malkki, 1999, Niwagaba, 2007). Nutrients are released slowly from faeces than from urine. Niwagaba (2007) and Jönsson et al. (2004) explained this behaviour by highlighting that a large proportion of the nitrogen and phosphorus has to be degraded before the nutrients become water soluble and available to plants. The composition and characteristics of faecal sludge from VIP toilets are presented in Table 2-1.

Table 2-1: Characteristics of faecal sludge from VIP latrines

Parameter	Units	References		
		Gaillard (2002)	Lopez Zavala et al (2002)	Almeida et al (1999)
Moisture content	%	-	81.1	79.2
Volatile solids %	%	-	84.4	-
Total COD	mg/mg	0.57	1.45	1.38
Dissolved COD	mg/mg	0.09	-	-
Suspended COD	mg/mg	0.46	-	-
Volatile fatty Acids	g COD/ l	8.46	-	1.5
Total Nitrogen	mg/g	17.82	60.1	-
NH ₃ -	mg/g	-	3.4	7.2
NO ₃ ⁻	mg/g	-	0.03	0.14
pH		-	7.5	-
SO ₄ ²⁻	mg/g	-	1.1	-
Cl ⁻	mg/g	-	4.2	-
Ascaris eggs	(no./l)	2000-6000		
Helminthes eggs	(no./l)	20 000 – 60 000		

The composition of fresh excreta (urine and faeces) and faecal sludge collected from VIPs latrines differ greatly. Fresh excreta show higher values than the contents of VIPs for most parameters, as seen in Table 2-2. Research conducted by Nwaneri (2009) revealed that the organic matter in faecal sludge undergoes a certain degree of biological decomposition within the pit latrine which reduces the chemical oxygen demand (COD). This stabilisation process produces carbon-based molecules that are not readily degradable and consists of more stable, complex molecules such as cellulose and lignin thus lowering the oxygen demand.

In addition, another key factor that causes this marked difference is the diet, health and age of individuals as well as the employment mode of the toilets. Additional material, such as food residues, wash water and wiping instruments, are often disposed into the pits thereby changing the sludge properties. (Nwaneri, 2009).

Table 2-2: Comparison between the contents of faecal sludge from VIP latrines and fresh excreta (Nwaneri, 2009)

Characteristics	Units	Fresh excreta	Faecal sludge
BOD	[g/cap day]	45	8
TS	[g/cap day]	110	90
TKN	[g/cap day]	10	5
COD	[mg/l]	-	20 000 – 50 000

2.2.2 Collection and Disposal of Faecal Sludge

Management of faecal sludge in an economically and environmentally acceptable manner is a challenge for practitioners. The proper understanding of the characteristics of the waste is essential to identify the most suitable disposal routes (Bakare, 2014, O’Riordan, 2009, Rose et al., 2015). Depending on the conditions of temperature, moisture content and pH, the excreta could have undergone considerable decomposition and the pathogen activity could have significantly been reduced after 2 years of storage so that the excreta can be manually handled.

Generally, two pit emptying methods are employed in removing excreta from pit latrine, i.e. by mechanical operating devices or by manual emptying. In the former option, a suction or pumping device is employed into the pit. Manual emptying involves people removing the excreta with shovels rakes and bins. Both methods of pit emptying are shown in Figure 2-2



(a) Mechanical emptying



(b): Manual emptying

Figure 2-2: Photographs showing the manual and mechanical emptying

Once the sludge from the pits has been removed, it needs to be transported and treated. It must be ensured that the faecal sludge disposal route does not contaminate the environment and affect public health. Part of the difficulty in implementing proper faecal sludge management is related to the misconception of considering faecal sludge as waste and not as a resource. Therefore, finding innovative approaches for resource recovery is a major step towards solving the sanitation problem. Diener (2014) stated that if faecal sludge management systems are designed with the goal of resource recovery, viable business models could emerge. Different valorization routes proposed by various authors include use as a fuel for burning (Muspratt et al., 2014a, Werther and Ogada, 1999), production of biogas from anaerobic digestion, protein derivation use for animal feed (Čičková et al., 2015, Vinnerås et al., 2006), and use as an organic fertilizer or soil conditioner (Moe and Rheingans, 2006, Mihelcic et al., 2011, Niwagaba, 2007, Malkki, 1999).

2.2.2.1 Use as a biofuel

Currently, the use of faecal sludge as a biofuel is in a premature phase and it is much less utilised compared to sewage sludge from waste water treatment plants (Muspratt et al., 2014a). Stasta et al. (2006) stated that a third of the fuel required to power a cement kiln can be supplemented using dried sludge. Considerable strides have been made to show the faecal sludge fuel potential. Muspratt et al. (2014a) reported that the average calorific value of faecal sludge obtained from the cities of Kumasi (Ghana), Dakar (Senegal) and Kampala (Uganda) was 17.3 MJ/kg. Zuma et al. (2015) found the average calorific value of the sludge from VIP toilets located in the community of Bester, in Durban, South Africa, to be 14.3 MJ/kg. These values are comparable to the energy content of some common fuels as illustrated in Table 2-3.

Table 2-3: Calorific values of common industrial fuel

Fuel	Calorific value MJ/kg
Coffee husks	16
Firewood	16
Sawdust	20
Charcoal	28
Used engine oil	33
Diesel	42

2.2.2.2 Production of animal protein

Faecal sludge can be used as a medium to grow insect larvae that can be a source of protein to animals. Faecal matter is used to feed black soldier fly larvae, *Hermetia illucens* L, to transform the organic waste into valuable products. This process reduces the dry matter by up to 80 % and produces a nutrient-rich by-product in the form of the last larval stage, the pupae. Their protein content, approximately about 40 %, makes them a valuable alternative to aquaculture, chicken farms, and frog farms with respect to other forms of municipal organic waste (Čičková et al., 2015). Black soldier flies are currently being bred commercially in Accra, Ghana and Kampala, Uganda for aquaculture (Diener et al., 2014).

2.2.2.3 Biogas production

Biogas is produced from the decomposition of biomass in a biological process. Bio-digesters are commonly used to generate biogas from biodegradable waste and sewage sludge. There exist small-scale bio-digesters using faecal matter as feedstock and the gas produced is used for cooking. California University of Technology (Caltech) are currently researching into another innovative hydrogen gas production method from black water (faeces, urine and water). Their toilet prototype oxidizes black water via an electrochemical process to produce hydrogen. This process is powered by photovoltaic solar energy (Goodier, 2012). Diener et al. (2014) observed that the faecal matter from public latrines have a greater potential for biogas production as it would have undergone less stabilization due to shorter sludge retention times as compared to household pit latrines.

2.2.2.4 Agriculture

Research on excreta-based fertilizers has gained attention as a viable alternative to the current methods, which are expensive or have negative impact on the environment. It is estimated that around 60-70 % of nutrients discharged from the fields ends up in human excreta (Malkki, 1999). This gives a reason to use human excreta, as an alternative to artificial fertilizers, to provide the nutrients necessary for plant

growth. As observed in Table 2-4, human waste is rich in the three essential nutrients nitrogen, phosphorus and potassium. Faecal matter can be applied in agriculture for land spreading or composting.

Studies have shown that organic soil conditioners such as faecal sludge, possess the following advantages in comparison to synthetic substances (Henley et al., 2011, Sapienza, 2005):

- slow release of nitrogen;
- increases the water retention capacity of sandy soils;
- increases aeration and drainage of loamy and clay soil;
- increases the nutrient retention capacity of soils.

Human excreta is richer in major nutrients in comparison to other animal manure that are often used as fertilizers, as seen in Table 2-4.

Table 2-4: Comparison of nutrients from different excreta sources and inorganic fertilizers (Montangero and Strauss, 2002)

Source	Elemental composition				
	N	P	K	Ca	Mg
Fresh manure composition (% wet mass)					
Poultry ^a		0.5 - 1.1	0.8 - 1.2	0.4	0.2
Cattle ^b	2.5	0.3	0.5	0.3	0.1
Sheep ^b		0.5	0.8	0.2	0.3
Horse ^b		0.3	0.6	0.3	0.1
Swine ^b		0.5	0.4	0.2	0.0
Goat ^c		0.4	4.6	0.7	1.2
Inorganic fertilizer composition (%)					
Rock phosphate ^d		11 - 17	-	25.0	-
Single superphosphate ^d		7 - 10	-	20.0	-
Triple superphosphate ^{d,e}		19 - 23	-	13.0	-
Potassium chloride ^{d,e}		-	26 - 27		
Dolomite ^d				22.00	19.0

data source: ^a (Ghosh et al., 2004), ^b (Ecochem.), ^c (Mnkeni and Austin, 2009), ^d (Silva, 2000), ^e (Koenig and Rupp, 1999), ^f (Montangero and Strauss, 2002)

2.3 Concept of drying

Drying is a chemical engineering unit operation which is commonly used for various applications and it is generally defined as the removal of water or any other solute from the solid. Drying is an intricate transient process involving coupled momentum, heat and mass transfer. Physical, chemical or biochemical transformations may occur during the drying process, resulting in changes in texture, colour, odour and other properties of the solid product (Perry et al., 2008, Mujumdar, 2006).

2.3.1 Drying technologies for sludge treatment

Drying reduces the costs associated with sludge storage, transportation, packaging and retail as the process reduces the mass and volume (Arlabosse and Chitu, 2007). It also increases the lower calorific value through the decrease of the moisture content of the sludge, consequently turning the material into a suitable combustible. In addition to the aforementioned, drying at high enough temperatures (above 80 °C), makes sludge hygienic by killing pathogenic organisms (Henley et al., 2011). In their studies on *Ascaris* eggs, Aitken et al. (2005) and Popat et al. (2010) reported the inactivation of the organisms was greater than two log reductions achieved within 2 h at a temperature of 50°C. Thomas et al. (2015) reported the inactivation of *Ascaris* using a conical-augur device using temperatures of 70 °C and a residence time of 6 seconds.

2.3.1.1 Solar drying

Solar drying is usually performed in a greenhouse with the structures constructed on a concrete basin and the walls in a transparent material to allow solar radiation to enter. The influencing parameters on the process are the solar thermal energy flux received, the air temperature and the ventilation rate, as well as the initial dry moisture content of the sludge. Bennamoun et al. (2013) reported drying times as long as 20 days, but solar drying is an economic method when compared to the other process as it utilises a renewable source of energy. However, the downside to this drying method, apart from the long drying times, is the inefficiency to eliminate pathogens (Chen et al., 2002). The radiation of short electromagnetic wave lengths, such as the ultraviolet (UV) light, is blocked by the cover, consequently reducing the degree of pasteurisation, especially for coliforms sensitive to UV light. In addition, other disadvantages associated with solar drying are the high surface area required to process large amounts of sludge and the need to mechanically turn the sludge during the process.

2.3.1.2 Radiative drying (Microwave and Infrared)

Radiative drying can be distinguished into two sections, microwave and infrared drying. Microwaves have a wavelength between 0.025 to 0.75 microns that fall between radio and infrared waves. It has been reported that when microwave heating is used, approximately 75 % less energy is required in comparison to thermal heating (Thiagarajan, 2008). During microwave drying, the microwave

electromagnetic radiation penetrates within the solid and elevates the solid temperature by exciting the dielectric molecules (such as water). This provides the energy necessary to remove the moisture in the solid. Microwave drying has been commonly used to dry food as it preserves the texture and taste (Thiagarajan, 2008).

In the case of infrared (IR) drying, the surface of the solid is heated by exposure to the IR radiation. IR can penetrate a certain depth in the solid, but not entirely. After the surface is heated, there is the classical mechanism for internal heat transfer occurring within the solid. An example of IR drying for faecal sludge drying is the Latrine Dehydration and Pasteurisation (LaDePa) machine in Durban, South Africa. Prior to drying, the LaDePa machine mechanically separates detritus from the waste and pelletises the sludge. In the drying chamber, the pelletized sludge is initially dried by hot air, then by medium-wave infrared radiators. The LaDePa machine is shown in Figure 2-3.

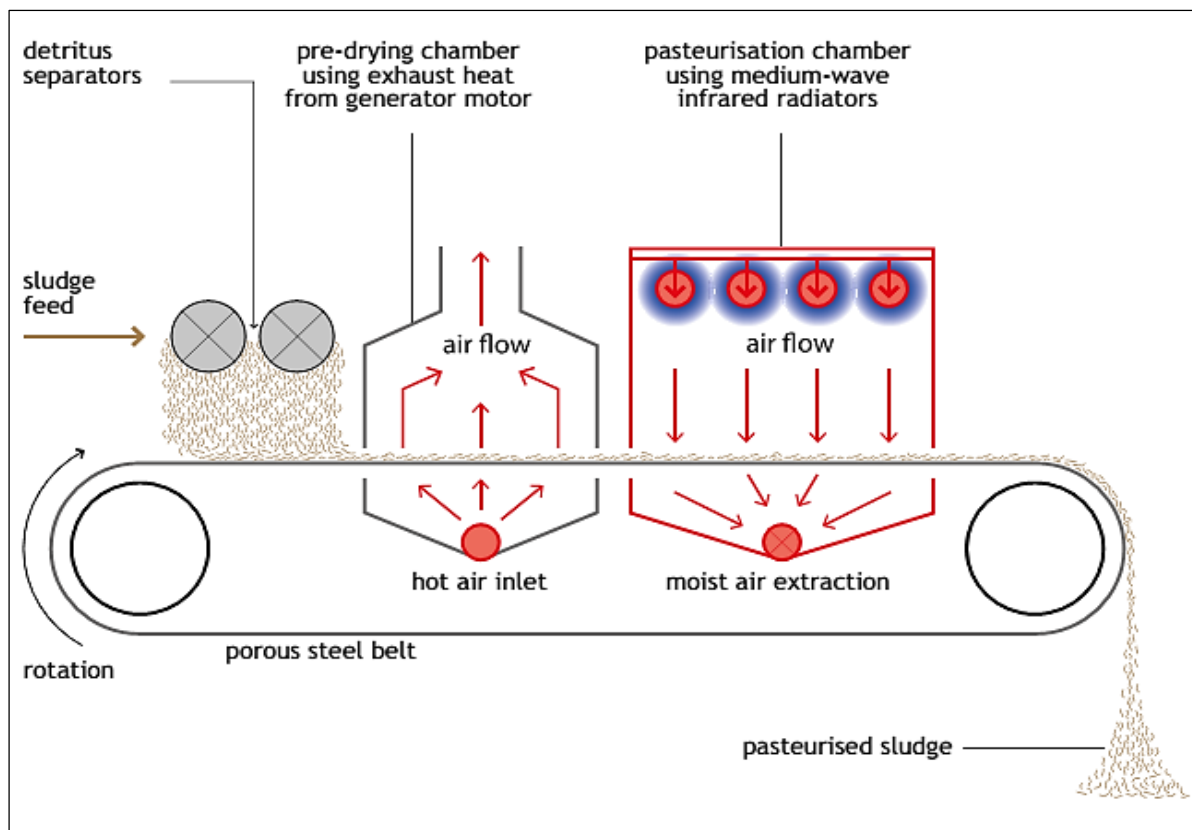


Figure 2-3 Schematic of the LaDePa machine

2.3.1.3 Freeze drying / Lyophilisation

Freeze drying or lyophilisation employs the phenomenon of sublimation (primary drying) in conjunction with desorption. Sublimation is the direct transition of a substance in its solid form into a gaseous form. In the case of water, sublimation occurs when the temperature and vapour pressure of the solid are below the triple point (611.2 Pa and 0°C).

This drying method is commonly applied in the manufacture of pharmaceutical and biological substances that are denatured or deactivated by heat (thermolabile material) or materials that are unstable in aqueous solutions for prolonged storage periods, but stable in the dry state. Freeze drying is also implemented in the food industry to produce dehydrated coffee, soups and meals for consumers in the supermarket. This method has also been tested for poultry manure drying (Sistani et al., 2001, Dail et al., 2007).

2.3.1.4 Hot air convective drying

This method is a form of thermal drying in which the sensible heat required to elevate the temperature of the wet solid is supplied by means of hot air convection. The heated air flows through the wet material and acts as a carrier or sweep gas to remove the moisture evaporated from the material. The operating parameters that influence the drying rate in this method are the air temperature, humidity and velocity. Heating up air to the desired temperature requires a significant amount of energy and therefore to ensure the economic sustainability of the processing technology, this method often implements exhaust gas where no further heating is required. This method of drying is mainly utilised in the thermal drying of wastewater sewage sludge in which the commonly used driers are the belt dryer, drum dryer and fluidized bed. However, there is a gap in literature detailing the convective drying of sludge from onsite facilities, hence the focus of this study.

2.3.2 Moisture in solids

Wet solids can be split into two basic groups in accordance to their drying behaviour (Henley et al., 2011):

1. Granular / crystalline solids – these tend to hold moisture in open pores between particles and are mainly inorganic materials. During drying, the solid is generally unaffected by moisture removal, therefore the drying conditions do not influence the properties and appearance of the dried product.
2. Fibrous, amorphous and gel-like materials – these are mainly organic materials and tend to dissolve moisture or trap moisture in fibres or very fine pores. These materials are affected by the removal of moisture and often reduce in volume upon drying and swell when wetted. Drying in the later stages tend to be slow. If the surface is dried rapidly, high temperature and moisture gradients may exist, which will result in warping, case hardening or cracking.

2.3.2.1 Types of moisture

Wet materials differ in their structural, physical, chemical, and biochemical properties which may significantly affect the drying process. Despite this variability, Strumillo (1986) states that, in practice,

the important parameters to consider are the types of moisture in the solid, defined as a function of the type of bonding with the material. The types of moisture present in a solid are bound moisture and unbound moisture. These are diagrammatically presented in Figure 2-4.

Bound moisture is that which is held to the solid matrix and exerts a vapour pressure which is less than that of the pure liquid at the same temperature. Moisture can be bounded biologically, chemically or physically

Unbound moisture is that which exerts an equilibrium vapour pressure equal to that of the pure liquid at the same temperature. This type of moisture can be removed relatively easier in a solid in comparison to bound moisture.

Free moisture is the moisture contained in a substance in excess of the equilibrium moisture content at particular air temperature and humidity (Treybal, 1980). It can be either bounded and / or unbounded

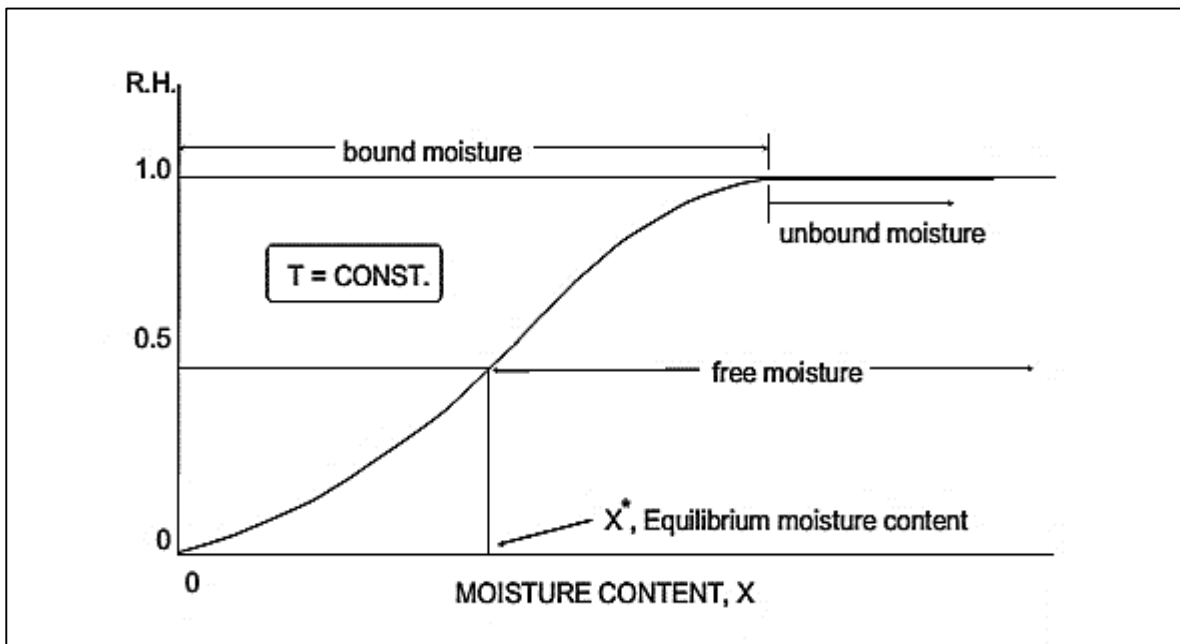


Figure 2-4: Different type of moisture contents (Mujumdar and Devahastin, 2000)

2.3.2.2 Psychometrics

The drying of solids leads to the humidification of the surrounding air. It is therefore necessary to understand psychometrics, which is the study of the properties of air and water vapour mixtures. For moisture to evaporate from the solid, its temperature should be such that its vapour pressure exceeds the partial pressure of the moisture in the gas in contact with the wet solid (Perry et al., 2008). The moisture removal capacity of the air increases by increasing the temperature and decreasing the air humidity (Mujumdar, 2006). Common definitions encountered in psychometry are presented in Table 2-5

Table 2-5: Common terms encountered in drying

Parameter	Definition	Relationship
Absolute humidity	Mass weight of water vapour per mass of moisture-free gas	$H = \frac{w_A P_A}{w_B (P - P_A)} \quad (2-1)$
Dew point temperature	Temperature at which moisture begins to condense when mixture is cooled at constant pressure	
Dry-bulb temperature	Temperature of the vapour gas mixture	
Relative humidity	Ratio of partial pressure of moisture to partial pressure of moisture at saturation	$H_R = 100\% \times \frac{P_A}{P_A^s} \quad (2-2)$
Absolute saturation humidity	Absolute Humidity at saturation	$H_s = \frac{w_A P_A^s}{w_B (P - P_A^s)} \quad (2-3)$
Wet-bulb temperature	Equilibrium temperature reached by a small amount of gas – vapour mixture after cooling it so as to reach saturation (100% relative humidity)	

A - Moisture; B - Moisture free gas

2.3.2.3 Equilibrium moisture content

When dry air flows over the surface of a moist solid, moisture is lost by surface evaporation until an equilibrium condition is achieved (Richardson et al., 2002). Similarly, if a dry solid is in contact with moist air, the solid will absorb moisture from the air until equilibrium is achieved. From a thermodynamics perspective, the drying rate is directly proportional to the difference of the chemical potentials of the moisture in the material and in the drying agent, thus the two are equal at equilibrium.

$$\text{Drying rate} \propto (p_{\text{pure}}^{\text{sat}} - p_{\text{air}}) \quad (2-4)$$

Perry et al. (2008) defines the activity of water in the gas phase as the ratio of the partial pressure of water to the vapour pressure of pure water. This has the same form as the definition of relative humidity. The activity of water in a mixture or solid is defined as the ratio of the vapour pressure of water in the mixture to that of a reference, usually the vapour pressure of pure water.

$$a_{mix} = \frac{P_{mix}^{sat}}{P_{pure}^{sat}} \quad (2-5)$$

At equilibrium, it therefore means that the moisture activity in the material equals the relative humidity of the air (Coumans, 2000).

$$a_{mix} = a_{air} = H_R \quad (2-6)$$

Therefore, in either cases of absorption or desorption of moisture to the material, the rate of moisture transfer depends on the difference between the moisture concentration on the surface of the solid and the relative humidity of the air. The moisture content of the solid material which is in equilibrium with the vapour present in the drying air, at a given temperature and humidity known as the equilibrium moisture content (EMC) or sometimes referred to as the minimum hygroscopic moisture content (Strumillo, 1986). Theoretically, a solid cannot be dried to a moisture content below the EMC.

While the EMC of a material varies with both humidity and temperature, it is strongly dependent on the air humidity (Lewis, 1921). For this reason, it is more convenient to study the relationship of equilibrium moisture at different air relative humidity than temperature.

2.3.3 Convective heat and mass transfer

When a wet material is subjected to thermal convective drying, heat and mass transfer processes occur simultaneously within the material being dried and in the boundary layer of the solid. These processes are depicted in Figure 2-5. The rate at which drying occurs is governed by the rate at which these transfer processes proceed (Mujumdar, 2006).

1. Energy transfer (mostly as heat) from the surrounding environment is used to heat the solid. The removal of moisture from the surface of the material by convection depends on the external conditions of temperature, air humidity, air flow, external surface area of the solid, and pressure.
2. Mass transfer of internal moisture to the surface of the solid. This is achieved by moisture migration from the interior of the solid out to the surface, from where its evaporation by the process previously explained. The internal movement of moisture within the solid to the surface is a function of the morphological characteristics of the solid (porosity, tortuosity, pore size), temperature and moisture concentration.

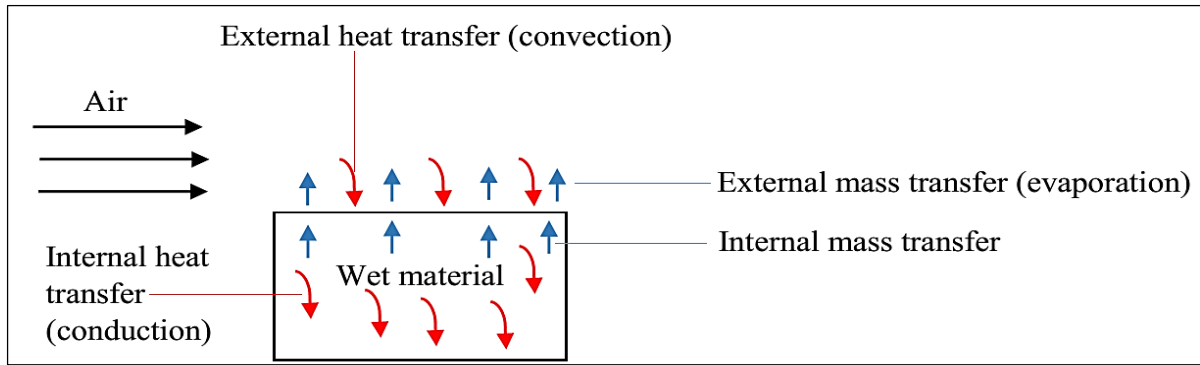


Figure 2-5: Heat and mass transfer during convective drying

Mechanism of moisture migration

Movement of moisture within the solid may be effected by one or several of the following mass transfer mechanisms (Gavrila et al., 2008, Mujumdar, 2006):

- Liquid diffusion - if the wet solid is at a temperature below the boiling point of the liquid. It is regarded that the rate at which moisture is transferred is proportional to the change in moisture concentration of the material subjected to drying.
- Vapor diffusion - if the liquid vaporizes within material. This is perceived as the main mechanism by which moisture is transferred in its vapor state. It occurs in materials which have pores with size greater than 10^{-7} m.
- Capillary moisture movement - when a number of capillaries of different radii exist within a material forming interconnected channels. Capillary pressure gradient causes the redistribution of moisture by capillary suction from the large capillaries to the small ones.
- Hydrostatic pressure differences - when internal vaporization rates exceed the rate at which the vapour moves through the solid matrix to the peripheral and to the surrounding.

Various authors agree that the moisture movement mechanism within a solid could be represented by diffusion phenomena in accordance to Fick's second law (Aguerre et al., 1985, Léonard et al., 2005, Crank, 1975, Qian et al., 2011, Chemkhi and Zagrouba, 2005). The unsteady state decrease in the moisture concentration during drying is described by equation (2-7)

$$\frac{\partial M}{\partial t} = \frac{\partial}{\partial x} \left[D \frac{\partial M}{\partial x} \right] \quad (2-7)$$

Where, M is the moisture concentration (g.water/g.dry solid)

D is the constant of diffusion known as the moisture diffusivity (m^2/s)

x is the distance of moisture migration (m),

t is the drying time (s)

Apart from being used to describe the drying behaviour of material, Fick's second law of the unsteady state diffusion can also be used to determine the of moisture diffusivities of the material being dried.

The moisture diffusivity in drying is essentially a lumped parameter as it describes a combination of moisture migration due to capillary forces, liquid diffusion and vapour diffusion hence the name 'effective' moisture diffusivity. A constant effective diffusivity during convective drying of materials has been assumed by various authors (Doymaz, 2007a, Reyes et al., 2004, Hassini et al., 2007, Vega et al., 2007). As reported in previous works on convective drying, temperature significantly affects the effective diffusivity of moisture during the falling rate period (Celma et al., 2012).

Analytical solutions for Fick's second law are available in literature and are dependent upon the geometry of the solid (Crank, 1975). The analytical solution of the flat slab geometry is commonly used in the determination of the effective diffusivity due to its simplicity in relation to the other geometries (Reyes et al., 2004, Yu et al., 2009, Stasta et al., 2006). For slab of half thickness, z , the solution is given by equation (2-8)

$$MR = \frac{M - M_e}{M_0 - M_e} = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_{eff} t}{4z^2}\right] \quad (2-8)$$

Where M_e is the equilibrium moisture content (g.water/g.dry solid)

M_0 is the initial moisture content (g.water/g.dry solid)

z is the half thickness of slab (m)

D_{eff} is the effective moisture diffusivity (m²/s)

t is the drying time (s)

The effective moisture diffusivity is evaluated on the falling rate phase of the drying process using Fick's second law of unsteady state diffusion. At large drying times, only the leading term in the series expansion, $n = 0$, is significant, therefore the diffusion coefficients are determined by plotting experimental drying data in terms of $\ln(MR)$ versus time. This manipulation is made on the assumption that the effective diffusivity is not significantly affected by the moisture content. This assumption is formulated by various authors for the analysis of convective drying processes (Celma et al., 2012, Hassini et al., 2007, Vasić et al., 2012).

$$\ln(MR) = \ln\left(\frac{8}{\pi}\right) - \frac{\pi^2 D_{eff} t}{4z^2} \quad (2-9)$$

The effective moisture diffusivity of materials studied under convective drying are presented in Table 2-6

Table 2-6: Approximate effective moisture diffusivity of various materials (Celma et al., 2012)

Material	Moisture content (dry basis)	Temperature (°C)	Diffusivity (m ² /s)
Asbestos cement	0.10 - 0.60	20	$2.0 \times 10^{-9} - 5.0 \times 10^{-9}$
Clay brick	0.20	25	$1.3 \times 10^{-8} - 1.4 \times 10^{-8}$
Kaolin clay	< 0.50	45	$1.5 \times 10^{-8} - 1.5 \times 10^{-7}$
Silica gel		25	$3.0 \times 10^{-6} - 5.6 \times 10^{-6}$
Tobacco leaf		30 - 50	$3.2 \times 10^{-11} - 8.1 \times 10^{-11}$
Wastewater sludge		80 - 112	$1.6 \times 10^{-8} - 3.7 \times 10^{-8}$
Tomato wastewater sludge		30 - 50	$6.1 \times 10^{-10} - 2.5 \times 10^{-9}$
Wood, yellow poplar	1.00	100 - 150	$1.0 \times 10^{-8} - 2.5 \times 10^{-8}$

2.3.4 Drying kinetics

Every material has representative drying characteristics at a given set of conditions. Drying kinetics are determined experimentally by measuring the change in mass of a sample per given period of time. There are three experimental methods to determine drying kinetics (Kemp et al., 2001):

Periodic sampling or weighing – The entire or part of the sample is extracted at regular intervals during the drying process and its moisture content is measured. This method is time consuming and usually gives a few points on a moisture – time graph.

Continuous weighing - The sample is put on a thermo-balance and its weight is recorded continuously. This gives a much larger number of points on the moisture – time graph. However, slight variations in weight during drying can be concealed by random noise, e.g., by vibrations of the sample caused by the airstream.

Intermittent weighing - The sample is put on a balance. At intervals, the air flowing around the sample is cut off or diverted, so that an accurate weight reading can be obtained when the system has stabilised (which takes a few seconds). It has been discovered that this method does not affect the overall drying kinetics unless the drying times are very short.

As stated by Saeed et al. (2008), drying data can be represented in various different ways.

- i. **Drying curves** – These are obtained directly from data of weight loss as a function of time data. Moisture content is plotted versus time.
- ii. **Drying rate curves** - These graphs are the derivative of the drying curves and they show the drying rate versus time.
- iii. **Krischer curves** - These curves are derived from the combination of the first two types of graphs and show the drying rate versus the moisture content.

The typical drying curves of a material being dried at constant conditions are shown in Figure 2-6.

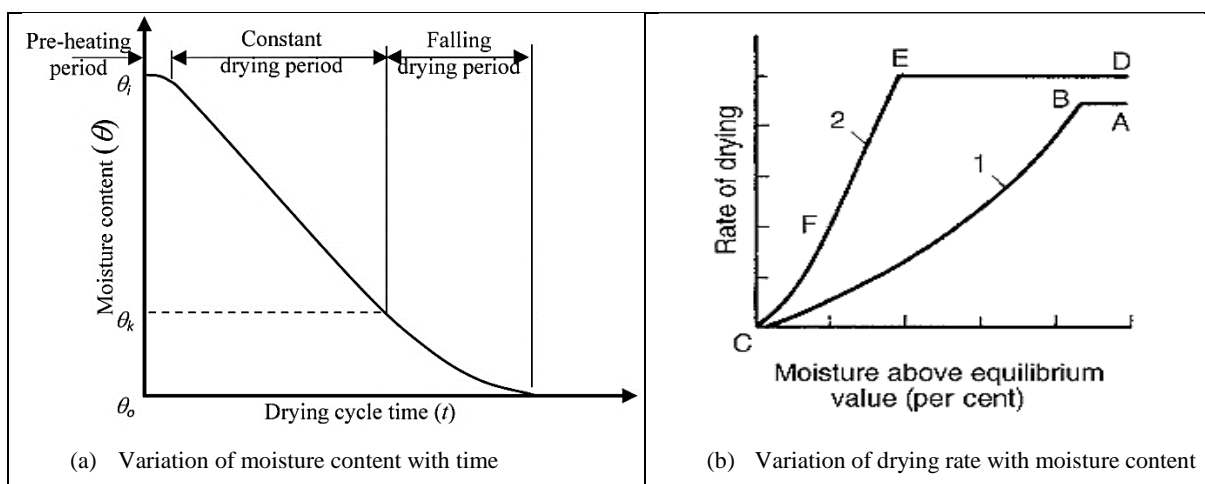


Figure 2-6: Typical drying curves obtained during the drying of a material (Rokey, 2006).

Figure 2-6(a) depicts the typical evolution of the moisture content with time of material undergoing drying. Figure 2-6(b) is obtained by mathematically differentiating Figure 2-6(a). It is from this figure that one can clearly see the various phases of drying that the material undergoes. The first drying curve depicts two clearly-defined segments. Segment AB represents a constant drying rate period and BC represents the region where there is a gradual decline fall of the drying rate as the moisture content decreases. The second curve illustrates three different stages that may occur during drying. Segment DE represents the constant rate period, the same period represented by AB. Segments EF and FC represent falling rate periods in which EF is known as the first falling rate period and subsequently FC is referred to as the second falling rate period. Generally, the two can be distinguished apart because the first falling rate period is a straight line whereas the second falling rate period is curved. Points B and E represent the transition from the constant rate period to the falling rate period. The moisture content at which this transition occurs is known as the critical moisture content.

2.3.4.1 Constant rate period

During the constant rate period, the entire surface of the material is saturated in moisture which continuously evaporates and is replaced by moisture from inside the particle. Basically, the internal moisture transfer to the surface and the evaporation at the surface are in equilibrium, therefore the free moisture on the surface will be evaporated in a steady continuous mode. The temperature of material during this drying phase is constant, and it approximates to the wet-bulb temperature value. The length of the constant rate period depends on the difference between the moistness on the surface of the sludge and the amount of unbounded water inside the material (Flaga, 2005). This period is completely controlled by the rates of external heat and mass transfer since a film of free water is always available at the evaporating surface. Thus, heat transfer to the material is utilised as the latent heat necessary to evaporate the water at the surface.

2.3.4.2 First falling-rate period

After the critical moisture content, the surface is no longer completely saturated in moisture. The temperature of the sludge will begin to increase from the wet bulb temperature to the temperature of heating. Moisture from the surface of the material evaporates at a faster rate than it can be replaced from inside the particle. The speed of drying will decrease until balanced hydration is accomplished. When these conditions are reached, the drying rate greatly depends on the internal mass transfer (Richardson et al., 2002).

2.3.4.3 Second falling-rate period

At the conclusion of the first falling rate period, it may be assumed that the surface is completely dry and that the evaporation front within the solid has been created and moves towards the centre. Drying in this phase is not influenced by the external drying conditions and moisture migration may be as a result of any of the mechanisms of moisture migration detailed in section 2.3.3.

NB. Not all material exhibit all the drying phases mentioned

2.3.5 Process and material related factors that influence convective drying

It is generally accepted by various researchers that the drying rate is significantly affected by either the rate at which moisture migrates from within the material to the surface or the rate at which moisture leaves the surface by evaporation (Léonard et al., 2005, Vega et al., 2007, Lewis, 1921, Doymaz, 2007, Bacelos and Almeida, 2011). This is mainly dependent upon the drying conditions employed. The key parameters that influence external mass transfer are air temperature, relative humidity and velocity as well as the available contact surface area. However, the internal mass transfer is affected by the physical composition of the material, material temperature and moisture content.

Considerable research has been conducted on the hot air convective drying of different types of materials. Several factors that have major influence on drying rate have been identified and can be lumped into two distinct categories: process conditions and material characteristics.

2.3.5.1 Temperature

For different applications, such as drying of food, pharmaceuticals, wood or wastewater sludge, temperature is a parameter of importance, as it significantly influences the drying rate. From a heat transfer perspective, it is commonly known that the greater the difference in temperature between the drying air and the moist material, the faster heat transfer occurs between the two. Higher temperatures also means higher input of heat for moisture vaporization (Bergman et al., 2011, Serth, 2007). However, high temperatures may result in loss of product quality, change of chemical structure as well as charring (Ruiz-López et al., 2004). Vast literature is available detailing the effect of temperature on the drying rate of various materials, with the most work conducted in the drying of agricultural material. Banga and Singh (1994) came to the conclusion that most properties that are important in drying such as thermal conductivity, mass diffusivity, and latent heat of evaporation greatly depend on temperature.

2.3.5.2 Relative humidity

Another relevant operating parameter is the amount of moisture present in the drying air as compared to the total amount of moisture that the air can hold at a particular temperature. This parameter is known as the relative humidity. The relative humidity influences particularly the external mass transfer, and consequently the drying rate particularly during the constant rate period. In other words, the increase of air relative humidity decreases the moisture concentration transfer potential between the surface of the material and the drying air, resulting in a reduced external mass transfer rate.

2.3.5.3 Air velocity

Air velocity during drying affects drying rate through its influence on the external mass transfer. Indeed, the increase of air velocity allows for the removal of more moisture from the material surface at given instance. Its effect on the falling rate period is much lower. A number of researchers have investigated the effects of air velocity on the drying rate of different food samples and have come to the conclusion that the effect of air velocity is not as pronounced as the contribution of drying temperature and relative humidity on moisture removal (Thiagarajan, 2008). Iguaz et al. (2003) reported that the effect of air velocity is more pronounced in the case of low temperature drying.

2.3.5.4 Size and surface area of the material

The surface area of the material available for heat and mass transfer is important, more particularly during the constant rate period. Higher surface area leads to higher moisture removal rate from the total surface area and consequently to lower drying times. Material size is also an influencing parameter. During the falling rate period, the decrease of the material size decreases for the moisture the distance to diffuse to the surface of the material, leading to faster a falling rate period and thus shorter drying times.

2.3.6 Drying Models

There are basically three methods of modelling drying processes, which are: (a) a theoretical approach (b) a semi-theoretical approach and (c) an empirical approach. The theoretical approach gives a better understanding of the transport processes as it is based on the basic physical principles of drying. The drying process can be completely described using heat and mass transfer equations. Thus, it is essential to know the material and transport properties so as to implement the equations. Some of the required properties include the heat and mass transfer coefficients, thermal conductivity, moisture diffusivity, density and specific heat of the material (Karathanos, 1999). However, some of these physical properties are difficult to ascertain, therefore the relevance and accuracy of the model are limited by the formulated assumptions. Theoretical models are usually mathematically complex and their resolution is computationally challenging.

The semi-theoretical approach gives understanding of the transport process in a simplified way but it is not as comprehensive as a theoretical approach (Omid et al., 2006). These kind of models typically predict the variation of moisture content with time and space whilst also describing the general moisture transport process. On the other hand, empirical equations, also referred to as experimental based models, are based on the mathematical fitting of the experimental data with poor or no understanding of the phenomena involved. Empirical models are restricted to the cases from where they were developed (Wang and Brennan, 1995). These empirical models consist of lumped parameters and in most cases, they generally predict only the average moisture content of the material as a function of time.

Empirical models are mathematically easier to formulate and compute in comparison to theoretical based models. For design purpose and industrial applications, simple empirical expressions that adequately describe the drying kinetics are preferable (Midilli et al., 2002). In an extensive review on thin layer models and their applicability conducted by Kucuk et al (2014), it was noted that there are no theoretical models developed so far that are practical and can also unify the calculations. The marked discrepancy that exists between the results formulated from theoretical models and industrial drying practice has led manufacturers to rely more on empirical models based on pilot plant tests (Kemp and Oakley, 2002).

An equation with the general form of Newton's law in heat transfer is often used to describe the moisture loss during thin-layer drying.

$$\frac{dM}{dt} = -k(M - M_e) \quad (2-10)$$

The Newton's equation yields a general exponential solution in terms of moisture ratio, MR as shown by equation (2-11).

$$MR = \exp(-kt) \quad (2-11)$$

Where k is a constant

t is the drying time

Due to the model's inability to provide kinetic data to the desired level of accuracy, numerous modifications to the Newton's equation have been proposed to provide better fits (Akgun and Doymaz, 2005, Kucuk et al., 2014). The variation to the Newton model are presented in Table 2-7. Empirical models are extensively used for drying and about 67 models have been reported up to date (Kucuk et al., 2014, Jain and Pathare, 2004). The models from Table 2-7 are the most widely used.

Table 2-7: Common thin-layer empirical models used in air drying (Doymaz, 2007)

Model name	Equation
1. Newton	$MR = \exp(-kt)$ (2-12)
2. Henderson and Pabis	$MR = a \exp(-kt)$ (2-13)
3. Logarithmic	$MR = a \exp(-kt) + c$ (2-14)
4. Page	$MR = \exp(-kt^n)$ (2-15)
5. Modified page	$MR = \exp(-(kt)^n)$ (2-16)
6. Verma	$MR = a \exp(-kt) + (1-a)\exp(-gt)$ (2-17)
7. Two-term exponential	$MR = a \exp(-kt) + (1-a)\exp(-kat)$ (2-18)
8. Midilli	$MR = A_0 \exp(-k_0t) + bt$ (2-19)
9. Two-term	$MR = a \exp(-k_0t) + b \exp(-k_1t)$ (2-20)

The Page model has been regarded as accurate for food drying (Guan et al., 2013). Montazer-Rahmati and Amini-Horri (2005) investigated the convective air drying of picrite on a conveyor belt dryer and the results obtained showed that of all the proposed models, the Page, modified Page and the Logarithmic models fit drying curves better than the rest with an R^2 value greater than 0.975. Of relevance to this project, the Page model was found the best model in describing the drying of sludge from municipal wastewater treatment plant as well as sludge from a tomato processing plant (Celma et al., 2012, Qian et al., 2011).

2.4 Effect of drying on nutrient content and thermal properties of excreta

Most of the research regarding the effect of drying on excreta properties has been conducted mostly for animal manure that have been traditionally known to be rich in nutrients and therefore can be used as a valuable fertilizer resource. The first study on the effect of drying on manure nutrient content was conducted by Cuthbertson and Turnbull (1934), who came to the conclusion that excreta drying in a water bath may lead to a significant loss in nitrogen and sulphur. Manoukas et al. (1964), whilst researching on the drying of fresh excreta from hens using freeze drying and convective oven drying, observed that the nutrient and calorific value are affected by the drying method and temperature. They reported reduction in the calorific value of up to 20 % and nitrogen losses of 15 % whilst using convective drying. However, some of the methods employed to ascertain these values were questioned by Shannon and Brown (1969) and a similar experiment using hen excreta was conducted to verify the results. They reported similar results, although the energy and nutrient losses were significantly lower than previously reported by Manoukas et al. (1964), i.e. 4.6 % and 10 % respectively. Both researchers admitted facing great difficulty in evaluating the energy content of wet excreta.

Sibbald (1979) investigated the effect of convective drying on the calorific value of manure for various farm animals (chicken, cows, pigs, horses, and sheep) by varying the drying temperature. Temperature did not have an effect on the calorific value of the excreta. In a study conducted by Sistani et al. (2001) about the drying effects on the phosphorus content of excreta from broiler chickens, it was noted that freeze drying and oven drying at 60°C significantly reduced total phosphorus concentrations than convective air drying at 35 °C. In a follow up study on dairy cattle manure, Chapuis-Lardy et al. (2004) reported higher water-soluble inorganic phosphorus concentration in fresh wet excreta than in dried manure. On the contrary, Dail et al. (2007) reported that oven drying at 65°C increased the water-extractable phosphorus in both swine and dairy manures.

The most important parameters required to describe heat transfer in a solid material are the thermal conductivity and heat capacity. Thermal conductivity is defined as the rate at which heat passes through a medium. Heat capacity, also referred to as thermal capacity, is the ratio of the heat added to or removed from an object to the corresponding temperature change. When a material is said to have a low heat

capacity, a large temperature change will result for a relatively small heat input. The ratio of thermal conductivity and heat capacity yields an important property called thermal diffusivity. Thermal diffusivity provides an indication of the rate that the temperature of a material changes when subjected to a temperature gradient (Bergman et al., 2011). Thermal properties are greatly influenced by the composition of the material, water content and temperature. Numerous researchers in diverse fields have investigated the effects of these parameters on the thermal properties of soil (Hanson et al., 2000, Hall and Allinson, 2009, O'Donnell et al., 2009), organic matter (Read and Lloyd, 1948, Huet et al., 2012, Dewil et al., 2007) and food (Rao et al., 2014, Lewicki, 2004, Wang and Brennan, 1995). All the authors came to the same conclusion that thermal conductivity and heat capacity increases as the moisture content of the material increases. The same effect on thermal conductivity is observed as the temperature of the material is increased.

It is evident that drying can affect the nutrient concentration as well as the thermal properties of excreta and this differs with the type of excreta employed. The operational sustainability of the drying process hinges on the ability to harness both energy and nutrient value, so the operating parameters during drying for this propose should be determined.

It should be noted, however, that the use of excreta derived products should be done with extreme caution as they may contain pathogens (bacteria, viruses, protozoa and helminths) which may pose as a health hazard. Health problems attributed to excreta reuse are very common in developing countries, especially where crop fertilisation using untreated excreta (Mara and Cairncross, 1989). Therefore treatment of the dried sludge is essential before it can be used.

3 MATERIAL AND METHODS

This chapter details the type of sludge used for this study as well as the custom designed rig used for the determination of the drying kinetics. This section also describes the experimental protocol for the characterization of the chemical and thermal properties of the sludge before and after drying.

3.1 Sludge source and processing

The sample employed in this study was faecal sludge from ventilated improved pit (VIP) latrine in the eThekwinini municipality (Durban, South Africa). The sample was obtained during pit emptying campaigns. An example of pit emptying is shown in Figure 3-1.

In the laboratory, the sludge was sieved using a 5 mm grid in order to remove detritus such as plastics and textiles. The sieved sludge was then stored in a closed bucket and placed in the cold room at 4°C until used for experiments.



Figure 3-1: Photograph showing the collection of samples during a pit emptying exercise

3.2 Description of the Drying rig

A convective dryer with a thermobalance was used for the drying experiments. The dryer can be classified as a direct-heat dryer, which provides the energy to heat the material with the hot air and sweeps away the moisture. The apparatus can be categorised into 3 distinct sections, which are: 1) humidification section, 2) heating section, and 3) the drying section.

Dehumidified air, supplied by a compressor, was employed for the experiments. A 23 mm diameter orifice plate alongside with a Pt 100 temperature sensor were placed before the entrance of the humidifier section. Pressure transducers were placed on either side of the orifice plate to determine the differential pressure as the air flowed through. The temperature and differential pressure readings were then used to evaluate the volumetric flow rate of the air entering the humidification section. The flow rate was regulated by a globe valve.

The humidifier was a packed column, in which air entering from the bottom section came into counter-current contact with water droplets from a shower rose entering from the top. The water went out the column by the bottom, passed into a water bath where it was maintained at the desired temperature, and was recirculated into the column. The relative humidity of the air used was adjusted by controlling the temperature of the water in the bath. Increasing the temperature of the water increases the amount of water that evaporates into the gas stream thus effectively increasing the humidity of the air. After absorbing moisture in the column, the air passed into the heating section, where electric heating coils elevated its temperature to the set value.

After the heating zone, the heated air is sent to the drying chamber where the faecal samples were dried. A probe to measure relative humidity was installed between the heating and drying chamber to monitor the humidity of the air entering the chamber. The drying chamber was constituted of a grid disc to place the sample, which was suspended on a precision weighing strain gauge load cell, with an accuracy of 0.01 g, connected to a computer. The sample mass was measured on-line which enabled to track the change in mass with time. A probe situated in the drying chamber was used to monitor the temperature and negative feedback was applied to regulate the heat supplied by the heating coils.

Air temperature, flow velocity, relative humidity, and sample mass were constantly monitored and the values from the measurements were continually logged on the computer. The flowchart and photograph of the entire set-up is presented in Figure 3-2 and Figure 3-3.

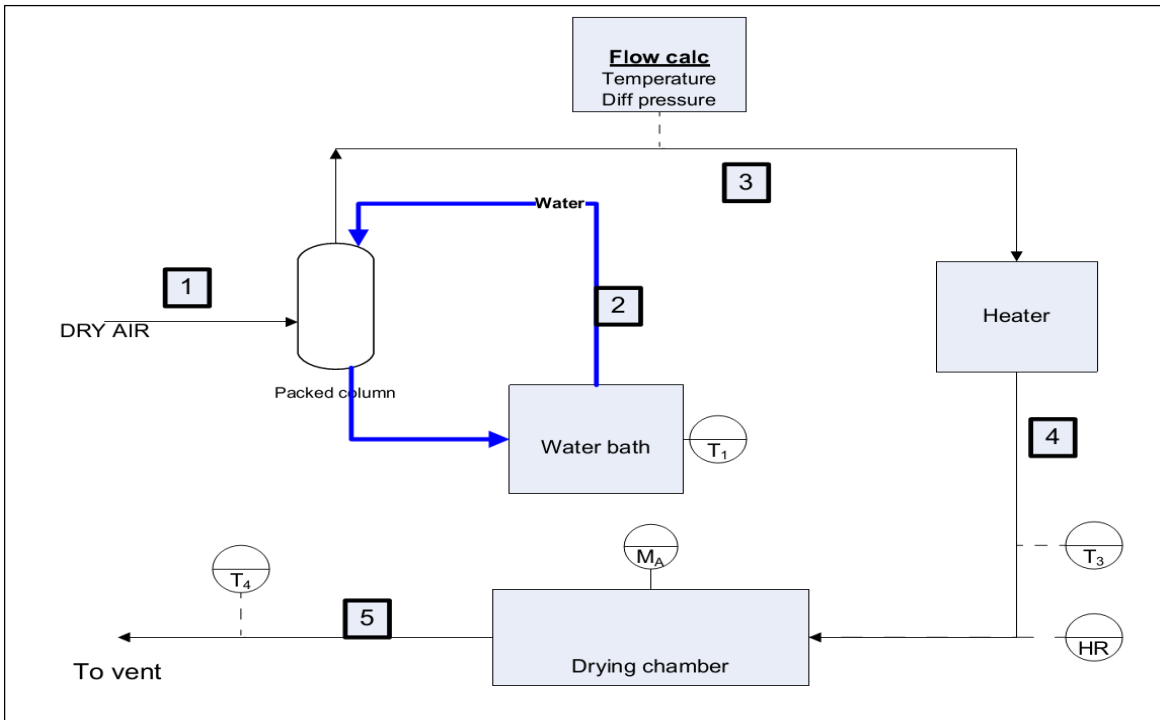


Figure 3-2: Flow chart of the drying rig (1. Dry air from the compressor, 2 – heated water from water bath, 3- humidified air to heater, 4 – heated air to the drying chamber, 5 exhaust air.)



Figure 3-3: Photograph of the drying rig

3.3 Experimental procedure

Unless stated otherwise, the procedures were performed in accordance to the standard operating procedures detailed in the operations manual which can be accessed of the website for the Pollution Research Group (<http://prg.ukzn.ac.za/laboratory-facilities/standard-operating-procedures>). The relevant Standard Operating Procedures are reproduced in Appendix A

3.3.1 Sample preparation

The effect of the operating conditions was tested on two different geometries, i.e. flat slab and cylindrical geometry in the form of pellets. Prior to use of the sludge, it was mixed in the bucket to ensure that it was homogeneous. Cylindrical shaped pellets were extruded by the use of a hand-held extruder. The diameter of the pellets was varied by using different sized extruder nozzles.

For the flat slab configuration, faecal sludge was placed on a circular holder measuring 70 mm diameter and 4 mm height. The average mass of sludge before the beginning of the experiment was $45 \text{ g} \pm 2 \text{ g}$.

3.3.2 Operating conditions

Prior to the start of a drying experiment, the conditions to investigate were set and the dryer was run for approximately 30 minutes so as to ensure that steady operating conditions were achieved. During the experiment, the sample mass was recorded at 5 minute intervals and the experiment was stopped when there was no mass decrease after 30 minutes.

The following drying conditions were investigated:

Temperature - Temperatures of 40 °C, 60 °C and 80 °C were investigated while keeping the relative humidity at 5 % and the air velocity at 0.06 cm/s.

Relative humidity - Relative humidity of 5 %, 15 % and 25 % were investigated while maintaining the air temperature at 60 °C and air flow velocity constant at 0.06 cm/s.

Flow-rate - Flow velocity of 0.03 cm/s, 0.06 cm/s and 0.12 cm/s were investigated while keeping the relative humidity at 5 % and the air temperature constant at 60 °C. In each case, the flow velocity refers to the average velocity of air passing through the cross section of the drying chamber.

Pellet size - The diameter of the pellets investigated were varied at 8 mm, 10 mm, 12 mm and 14 mm while maintaining the air temperature at 60 °C, the flow velocity at 0.06 cm/s and the relative humidity of 5 %.

At least three replicates were used for each set of drying conditions investigated to determine the repeatability of the experiments as well as to determine the statistical variance of the results obtained.

3.4 Physico-chemical characterisation of sludge

Characterisation of the sludge was performed to determine the effect of convective drying on the properties of sludge. The properties of interest were classified into two groups, which are the thermal and chemical properties. At least three replicate samples were used for each analysis conducted to enable the statistical analysis of the obtained results each

3.4.1 Moisture content and ash content

Moisture content was determined by drying samples in an oven at 105°C for about 24 h. The moisture content (M) of a product can be defined in either dry or wet basis. The moisture content is defined by equation (3-1) in dry basis, and by equation (3-2) in wet basis.

$$\%M_{db} = \frac{w_0 - w_f}{w_f} \times 100\% \quad (3-1)$$

$$\%M_{wb} = \frac{w_0 - w_f}{w_0} \times 100\% \quad (3-2)$$

Where w_0 is the initial mass of the sample before being oven dried

w_f is the dried mass of the sample

The two moisture content expressions are related by the following equation:

$$\%M_{db} = \frac{M_{wb}}{100 - M_{wb}} \times 100\% \quad (3-3)$$

The average initial moisture content for the sludge used in this study was found to be 80% (wet basis)

Ash content is the amount of material that remains after combustion in the oven at 550°C for 2 h. The ash content of a material represents the inorganic material that remains after the removal of water and organic matter by combustion. It is calculated by equation

$$Ash\ content = \frac{w_f - w_{f2}}{w_f} \times 100\% \quad (3-4)$$

Where w_{f2} is the mass of residue after combustion

3.4.2 Thermal analysis of faecal sludge

Thermal characterisation of the sample was performed as this data is relevant for the design of a dryer and for the use of the dried sludge as fuel. The properties that were analysed included the calorific value, heat capacity and thermal conductivity. The gross calorific value was determined by combusting a known mass of sample in an oxygen bomb calorimeter (*Parr 6200*). Thermal conductivity and heat capacity were determined using a thermal conductivity analyser (*C-Therm TCi*) which uses the Modified Transient Plane Source (MTPS) technique in describing the thermal conductivity, heat capacity and diffusivity of materials

3.4.3 Nutrient analysis

Studies conducted by Dail et al. (2007), Chapuis-Lardy et al. (2004) and (Sistani et al., 2001) showed that drying may have an impact on the concentration of nutrients available in poultry and pig excreta. Chemical analyses were performed on the dried product in order to ascertain the effect that convective drying might have on the agricultural value as a nutrient supplement. The nutrients that were investigated can be split into two categories, which are elemental and molecular nutrients. The elemental nutrients include sodium (Na), calcium (Ca), magnesium (Mg), potassium (K), and phosphorus (P), which are macronutrients essential for plant growth. The concentration of these nutrients in sludge were determined using Microwave Plasma-Atomic Emission spectrometer (*4200 MP-AES*) equipped with a concentric nebulizer.

The concentrations of the molecular nutrients were determined using a Spectroquant photometer (*Pharo 300*) and the relevant commercial test kits specific to the analysis. Prior to each test, a known mass of the sample was added to a known volume of water and centrifuged for liquid-solid separation. The resulting supernatant was taken and analysed. In essence, this analysis measures the amount of water soluble nutrients in the liquid fraction of sludge that is easily released after irrigation or rainfall. The compound ions investigated were ammonia (NH_3), nitrates (NO_3^-) and orthophosphates (PO_4^{3-}).

3.5 Data analysis

3.5.1 Moisture ratio

The moisture content of the solid material which is in equilibrium with the vapour contained in the drying agent, at a given temperature and humidity known as the equilibrium moisture content (M_e). Theoretically, a solid cannot be dried to moisture content below the equilibrium moisture content. The moisture ratio is the ratio of the moisture content at a particular time to the initial moisture content. It is calculated from equation (3-5):

$$MR = \frac{M - M_e}{M_0 - M_e} \quad (3-5)$$

Where M , M_0 , and M_e are the mass of the sample at any particular instant, the initial mass and the mass at the equilibrium moisture content respectively.

3.5.2 Drying curves

The sample mass was constantly monitored and logged on the computer. The drying rate, which is the amount of evaporated moisture over time, was calculated by dividing the decrease in mass of the sample between two subsequent measurements by the elapsed time as shown in equation (3-6). These were then normalised for differing surface areas by dividing by the surface area

$$Drying\ rate = \frac{w_t - w_{t+\Delta t}}{\Delta t \cdot A} \quad (3-6)$$

Prior to the calculation of the drying rate, the raw data was smoothened to eliminate the noise within the data by using a cubic spline fit over a number of adjacent data points as stipulated by Kemp et al. (2001). The Matlab code used to smoothen the data is presented in Appendix B and the comparison between the smooth data and the raw data is shown in Appendix C

3.5.3 Evaluation of the effective moisture diffusivity

The effective moisture diffusivity was calculated from Fick's second law of unsteady state diffusion which describes the internal mass transfer of moisture. The effective moisture diffusivity was evaluated only on the falling rate phase of the drying process. This analysis was conducted using flat slab geometry instead of pellets, as this offers more mathematical simplicity on the determination of the effective moisture diffusivity as compared to other geometries.

The temperatures for the analysis were 40, 50, 60, 70 and 80 °C while the air stream was operated at a constant air velocity of 0.06 cm/s and relative humidity of 5 %. The basic assumptions adopted for this analysis were:

- (a) The resistance to the flow of moisture through the sludge was uniformly distributed throughout the interior of the sludge.
- (b) The sludge could be viewed as a homogeneous mixture; hence it can be regarded to have uniform moisture distribution.
- (c) The movement of moisture due to thermal gradient within the sample is negligible therefore moisture movement may be regarded as a one-dimensional diffusion process.

(d) Negligible shrinkage of the sludge sample during drying.

These assumptions mean that the effective moisture diffusivity can be regarded as independent of the local internal moisture content. For purpose of this study, the equilibrium moisture content was regarded to be the moisture content of the sample after the drying experiment was achieved.

The diffusion coefficient was determined from equation (2-8) as described in section 2.2.2. However, the moisture ratio was evaluated on a mass basis as shown in equation (2-8)

$$MR = \frac{M - M_e}{M_0 - M_e} = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_{eff} t}{4z^2}\right] \quad (2-8)$$

Where M is the mass of sample (g),

M_e is the final mass of the dried sample (equilibrium mass) (g),

M_0 is the initial mass of sample (g),

z is the thickness of slab (m),

D_{eff} is the effective moisture diffusivity (m²/s)

t is the drying time (s).

Equation (2-8) was simplified by only considering only the first term of the summation series solution then linearizing the expression leading to equation (2-9).

$$\ln(MR) = \ln\left(\frac{8}{\pi}\right) - \frac{\pi^2 D_{eff} t}{4z^2} \quad (2-9)$$

Plotting a graph of $\ln(MR)$ vs t produces a straight line curve from which the effective moisture diffusivity can be determined by manipulating the slope of the graph.

The variation of the effective diffusivity of the water with temperature is classically represented by the Arrhenius equation (Chemkhi and Zagrouba, 2005), considering the fact that molecular kinetic energy increases when temperature increases.

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{RT}\right) \quad (3-7)$$

The activation energy E_a can be determined by linearizing equation (3-7) into equation (3-8) and plotting $\ln(D_{eff})$ versus $1/T$. The slope of the line corresponds to (E_a/R) and the intercept equals $\ln(D_0)$.

$$\ln(D_{eff}) = \ln(D_0) - \left(\frac{E_a}{RT} \right) \quad (3-8)$$

Where E_a is the activation energy (kJ/kmol)

D_0 is the pre-exponential factor (m²/s)

3.6 Fitting of drying curves to empirical models

One of the objectives was to test the fitting of various empirical drying models on the experimental results. The Newton model, Page model, modified Page model, Two-term exponential model and the Logarithmic model were the models investigated. Non-linear regression analysis, using Solver in Microsoft Excel, was conducted to evaluate the model constants at various drying conditions.

The drying models were evaluated through the comparison with the experimental data by using the goodness of fit statistical measure. This entails the determination of the coefficient of determination R^2 , which evaluates how well the model fits the experimental data, and the root mean square error ($RMSE$), which reflects the dispersion in the obtained data. A model represents accurately the experimental data for high R^2 close to 1 and a low value of $RMSE$.

The two statistical measures were evaluated as follows:

Root mean square error (RMSE)

$$RMSE = \sqrt{\frac{\sum_{i=1}^N (MR_{exp} - MR_{cal})^2}{N}} \quad (3-9)$$

Coefficient of determination (R^2)

$$R^2 = \frac{\sum_{i=1}^N (MR_{exp} - \overline{MR}_{exp})^2}{\sum_{i=1}^N (MR_{cal_i} - \overline{MR}_{exp})^2} \quad (3-10)$$

Where \overline{MR}_{exp} is the average moisture ratio

MR_{exp} and MR_{cal} are the experimental and calculated moisture ratio

N is the number of points used

4 RESULTS AND DISCUSSION

This chapter presents and discusses the experimental results obtained from convective drying of sludge from ventilated improved pit latrines. As explained in section 2.3.3, transport phenomena comprise of internal and external resistance to heat and mass transfer. Therefore, the resistance to the transport phenomena controls the rate of moisture removal within the solid (drying rate). By analysing the drying characteristics of faecal sludge, the predominant transport phenomena can be determined with the progression of drying.

This chapter is split into 3 main sections, the first detailing the effect of air temperature, relative humidity and flow rate as well as sample thickness on the drying kinetics of sludge. The second section highlights the effect of drying on the thermal properties as well as the nutrient content of the sludge, properties which are essential in the possible design of a sludge dryer and also vital for the agricultural sector. The last section encompasses the selection of an empirical drying model that best describes the drying data.

Unless stated otherwise, the results presented in this section are that of the cylindrical geometry in the form of extruded pellets. Particular focus was placed on pellets as studies have shown that pelletisation of bio solids is considered as a viable solution to producing a dust free product of high density. Siriwattananon and Mihara (2008) observed that pellets are effective in decreasing nutrient losses from the soil and also release nutrients at a steady rate compared with non-pelletized products.

4.1 Drying kinetics

This subsection details the drying curves obtained during the experiments in order to ascertain the effect of the parameters that were investigated - air temperature, air velocity, air humidity and sample size. Drying experiments were performed on the cylindrical geometry in the form of pellets, and also as a flat slab geometry. The shape of the rate versus time graphs produced at each data set were similar regardless of the configuration used on each operating parameter investigated. It was from these graphs that the different phases of drying were depicted. The variation of the mass with time during drying of the sample is presented as normalized mass or mass ratio to cater for the slightly different initial masses.

4.1.1 Effect of air temperature on drying rate

The effect of temperature on convective drying of faecal sludge was investigated by varying the air temperature while maintaining the diameter of pellets, air humidity and air flow constant. The temperatures used for this investigation were 40, 60 and 80 °C. For each experiment, an initial mass of

23 ± 2 g of pellets was extruded and placed into the drying rig. The typical drying curves depicting the evolution of mass ratio with time at different air temperatures is shown Figure 4-1.

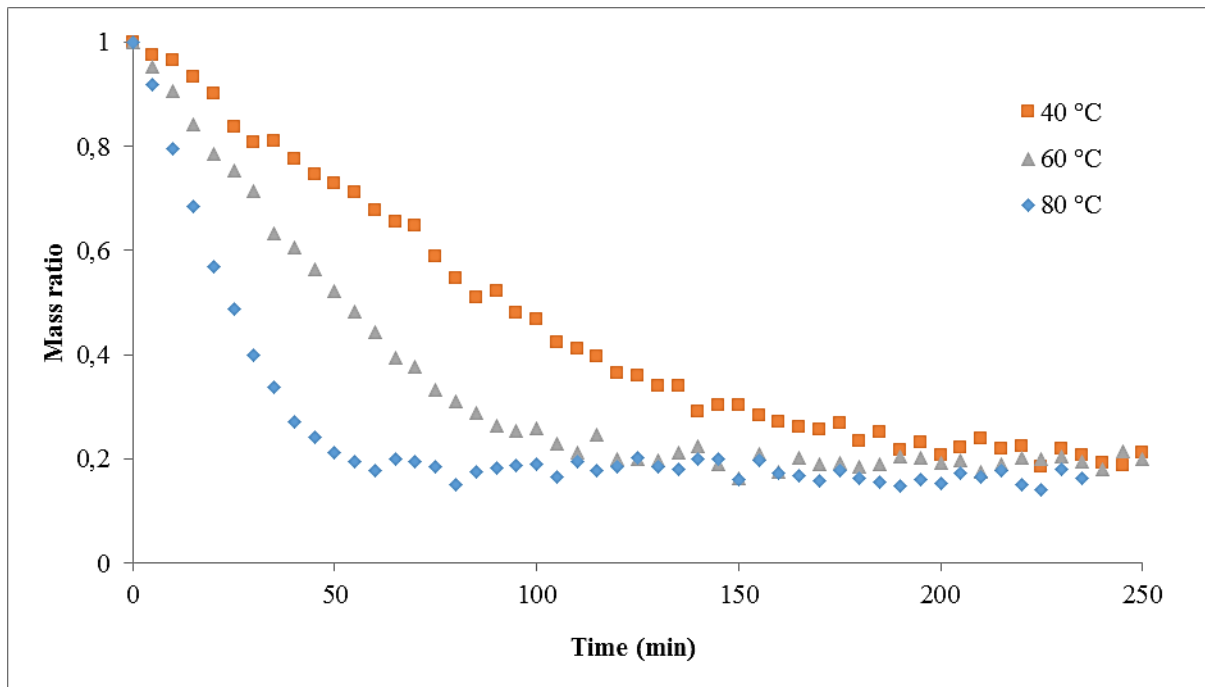


Figure 4-1: Influence of temperature on the sample mass evolution with time during convective drying of faecal sludge at constant pellet diameter of 8 mm, air relative humidity of 5 % and air flow velocity of 0.03 cm/s

As expected, the drying air temperature is one among the main factors that influence the drying kinetics. Drying sludge using higher air temperature, resulted in a notable decrease in the drying time, as can be deduced from Figure 4-1. Similar trends were observed for 10 mm, 12 mm, 14 mm pellets as well as a flat slab of 7 mm thickness. The times required to dry pellets with a diameter of 8 mm to an equilibrium moisture content of 10% (wb) at temperatures of 80 °C, 60 °C and 40 °C were 90 minutes, 135 minutes and 190 minutes respectively. Increasing the air temperature from 40°C to 80°C had an overall effect of reducing the total drying time by 53%.

The influence of temperature on the variation of the drying rate with amount of free moisture, which is represented by the moisture ratio, was investigated and the results are presented in Figure 4-2.

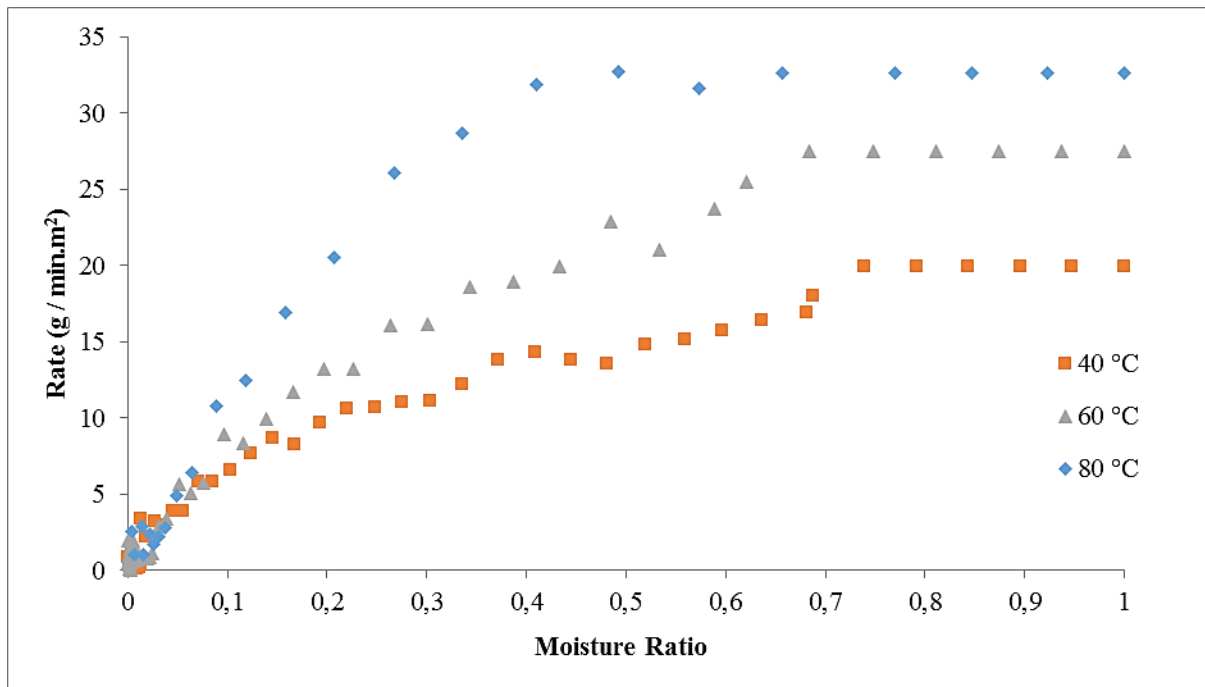


Figure 4-2: Influence of temperature on the drying rate with moisture ratio during convective drying of faecal sludge at constant pellet diameter of 8 mm, relative air humidity of 5 % and air flow velocity of 0.03 cm/s

The shape of the rate graphs produced clearly depicts the two phases of drying, which are the constant rate period and the falling rate period. The existence of a constant rate period means that the sludge had a high enough moisture content to maintain a saturated surface to allow for constant evaporation. The drying rates observed during the constant rate period whilst drying sludge pellets which are 8 mm in diameter, using air of 5% relative humidity and flow velocity of 0.03cm/s, at temperatures of 40°C, 60°C and 80°C were 20, 28 and 33 g/min.m² respectively. Using higher temperatures yielded higher drying rates during the constant rate period. Increasing the air temperature from 40°C to 80°C resulted in a 65% increase in the drying rate. This result is attributed to the fact that increasing the air temperature increases the rate of heat transfer into the sample of sludge. The greater the temperature difference between the solid and the drying air results in greater mass transfer (Doymaz, 2005, Saeed et al., 2008, Benali and Kudra, 2002, Tao et al., 2005, Stasta et al., 2006, Léonard et al., 2004).

The critical moisture content, which is the point at which there is a transition from the constant rate period to the falling rate period, was observed to be affected by the drying air temperature as depicted in Figure 4-2. The critical moisture content was observed at a moisture ratio of about 0.74 when drying using air with a temperature of 40°C, whereas the critical moisture content was found to occur at a moisture ratio of about 0.42 when drying with a temperature of 80°C. In other words, 26% of the free moisture contained within the sludge was removed during the constant rate period and the remainder of the moisture was removed during the falling rate period when drying at an air temperature of 40°C. In

contrast, 58 % of the moisture was removed during the constant period temperature of 80°C. This observed may be as result of increased internal moisture movement to the surface of the sludge due to increased vapour pressure that will then sustain the constant rate period until the sludge is much drier.

4.1.2 Effect of temperature on the effective moisture diffusivity, (D_{eff})

The existence of the falling rate period in the drying kinetics obtained in this study is evidence that the drying of faecal sludge is affected by the internal resistance to mass transfer. Moisture diffusivity, D_{eff} , is an essential transport property that describes the mass transfer of moisture in the falling rate drying period. It is assumed that the resistance to moisture migration to the surface of the solid because of water vapour gradients within the solid is the determining mechanism during this period.

At each set of investigated drying conditions, the moisture ratio was measured and the logarithm of the moisture ratio with time was plotted as shown in Figure 4-3.

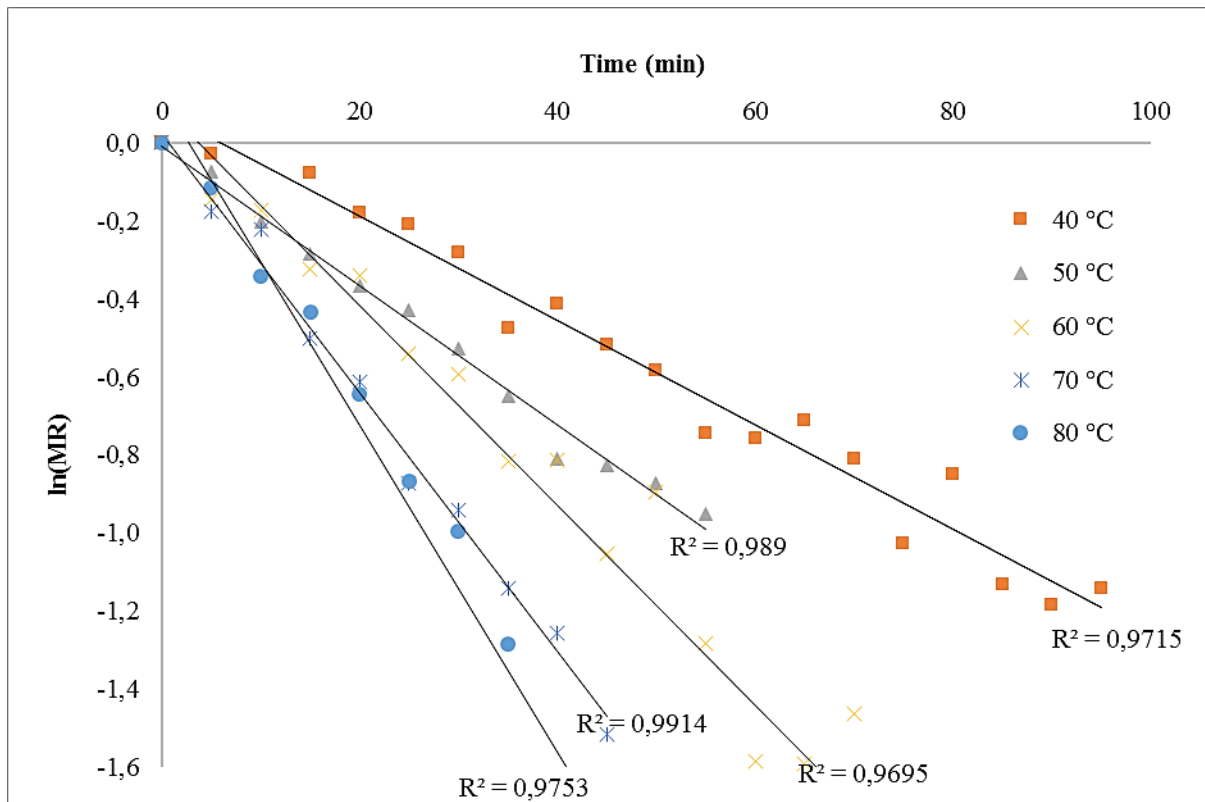


Figure 4-3: Logarithm of the moisture ratio versus time for the faecal sludge as flat slab at various air temperatures

Linear regression was only performed on the sections of the drying curves corresponding to the falling rate period. It can be observed from Figure 4-3 that the assumption of constant diffusivity during the falling rate period is validated, as a straight line describes the region with reasonable accuracy with a coefficient of determination, R^2 , at least 0.97. This observation implies that moisture transfer

mechanism during the falling rate period of the drying of sludge can be approximated by using the effective moisture diffusivity approach as detailed in section 3.5.3. In addition, the linear curve obtained from the logarithmic dimensionless moisture ratio versus drying time graph, Figure 4-3, indicates that the effective moisture diffusivity does not significantly depend on the instantaneous moisture content of the sludge sample during this drying period.

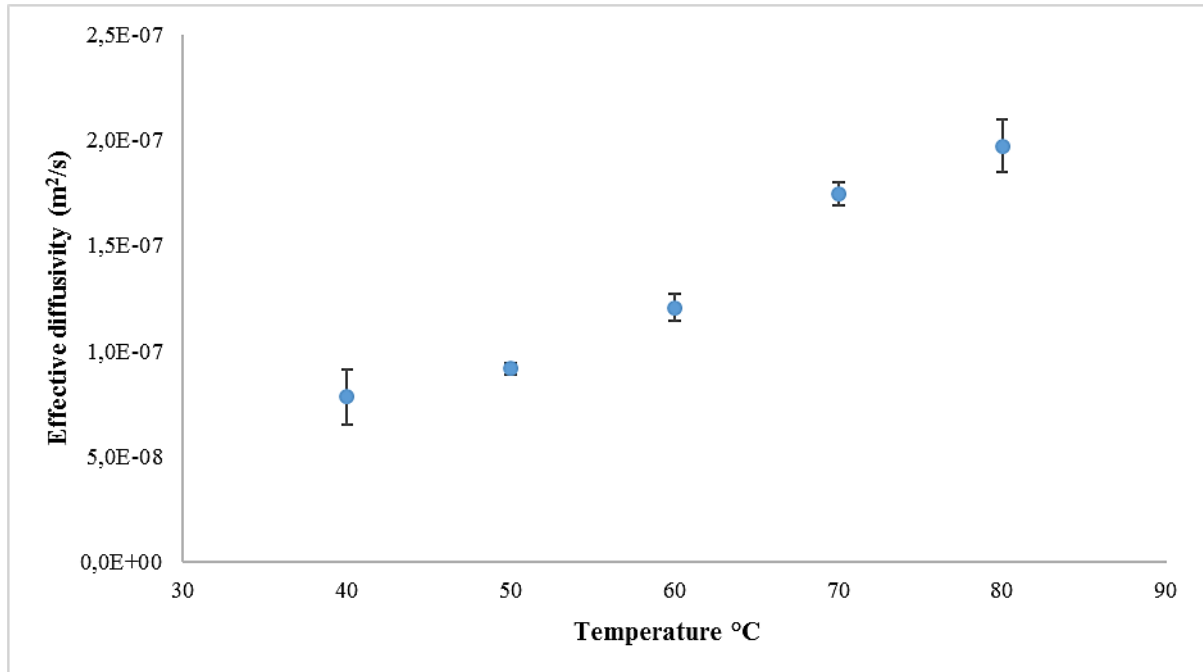


Figure 4-4: Effective diffusivity versus air temperature during drying of VIP faecal sludge as a flat slab

The variation of the effective diffusivities obtained as a function of temperature is illustrated in Figure 4-4. It can be observed from the plot that increasing the temperature considerably increases the effective diffusivity. This is because increasing the air temperature increases the internal temperature of the sample thus resulting in higher vapour pressure inside the sample. Higher internal vapour pressures results in faster moisture migration to the surface of the drying solid hence increased internal diffusion. The effective diffusivities ranged between 7.8×10^{-8} and 2.1×10^{-7} m²/s in the temperature range of this study. These values are in the same order of magnitude to those found in the study of wastewater sludge cited in Table 2-6.

Researchers in the drying field have found that the relationship between the temperature and effective diffusivity can be described by the Arrhenius equation (Celma et al., 2012, Vaxelaire et al., 2000, Léonard et al., 2004, Ruiz-López and García-Alvarado, 2007, Vega et al., 2007). To investigate whether the Arrhenius equation could be used to describe the relationship between the two properties for faecal sludge, the logarithm of the effective diffusivity was plotted versus the inverse of temperature as shown in Figure 4-5.

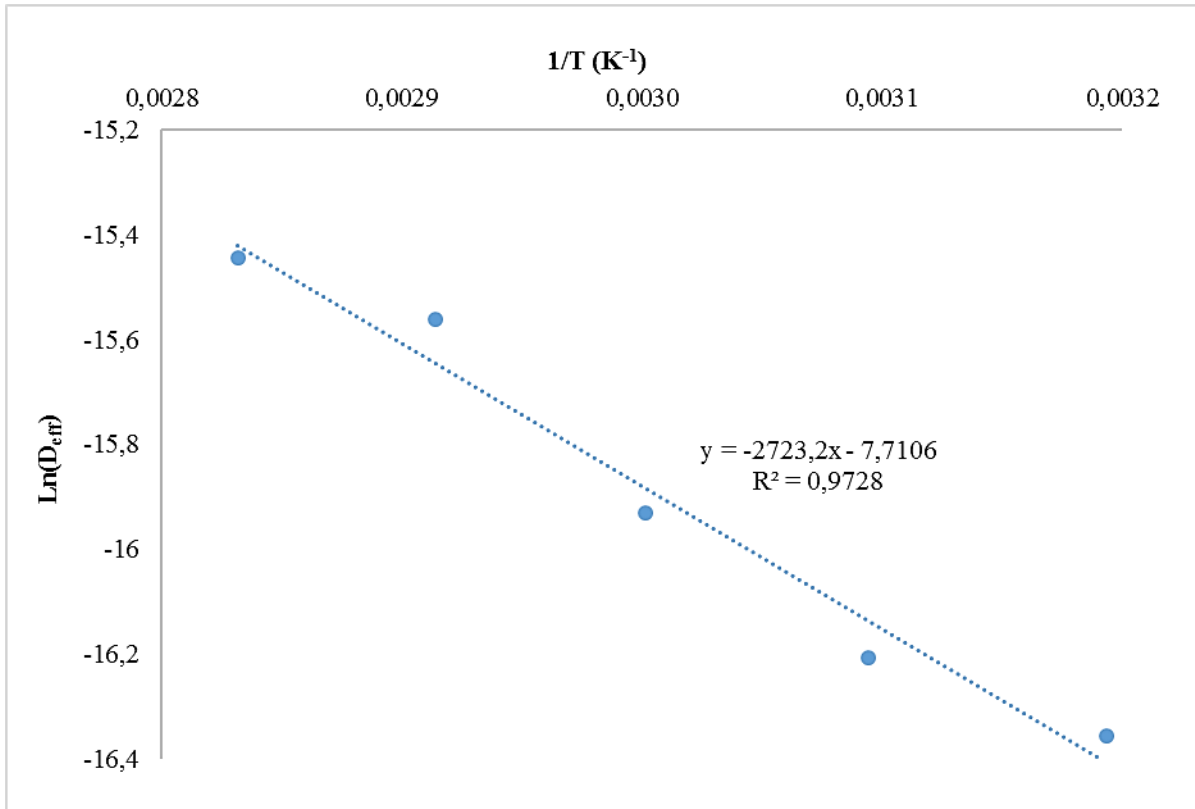


Figure 4-5: Variation of $\ln(D_{\text{eff}})$ versus $1/T$ to investigate the applicability of the Arrhenius equation in describing the relationship between drying temperature and the observed effective moisture diffusivity

A linear relationship, with a regression coefficient of 0.973, was found to satisfactorily describe the relationship between the logarithm of the effective diffusivity and the inverse of temperature as shown in Figure 4-5. This observation implies that the Arrhenius equation can be used to describe the dependence of the effective diffusivity of faecal sludge with the drying temperature in the range employed in this study.

After establishing the validity of the Arrhenius equation in describing the effective diffusivity, the activation energy of the sludge samples and the subsequent pre-exponential factor of the Arrhenius equation was evaluated. The activation energy was calculated from the slope of the straight line found in Figure 4-5, while the pre-exponential factor was evaluated from the y-intercept. The evaluation of the activation energy value is important as it is a measure of the temperature sensitivity of effective diffusivity. The activation energy E_a was evaluated at 21.78 kJ/kmol and the pre-exponential factor at $32.88 \times 10^{-8} \text{ m}^2/\text{s}$. Celma et al. (2012), in their work on the investigation of sludge from wastewater plants of tomato transformation industries, found that the activation energy varied between 30.15 kJ/mol and 36.70 kJ/mol. Reyes et al. (2004) also investigated the drying kinetics of biological sludge from a wastewater treatment plant and found the activation energy to be 30.07 kJ/mol and the pre-exponential

factor to be $41.73 \times 10^{-8} \text{ m}^2/\text{s}$. The values of activation and pre-exponential factor found in this study were in the same order of magnitude to the values obtained from wastewater sludge.

4.1.3 Effect of air relative humidity

The effect of relative humidity on the convective drying of VIP faecal sludge was investigated by varying the air relative humidity while maintaining the diameter of pellets, air temperature and air flow constant. The relative humidity employed for this investigation were 5%, 15% and 25% and the results are shown in Figure 4-6.

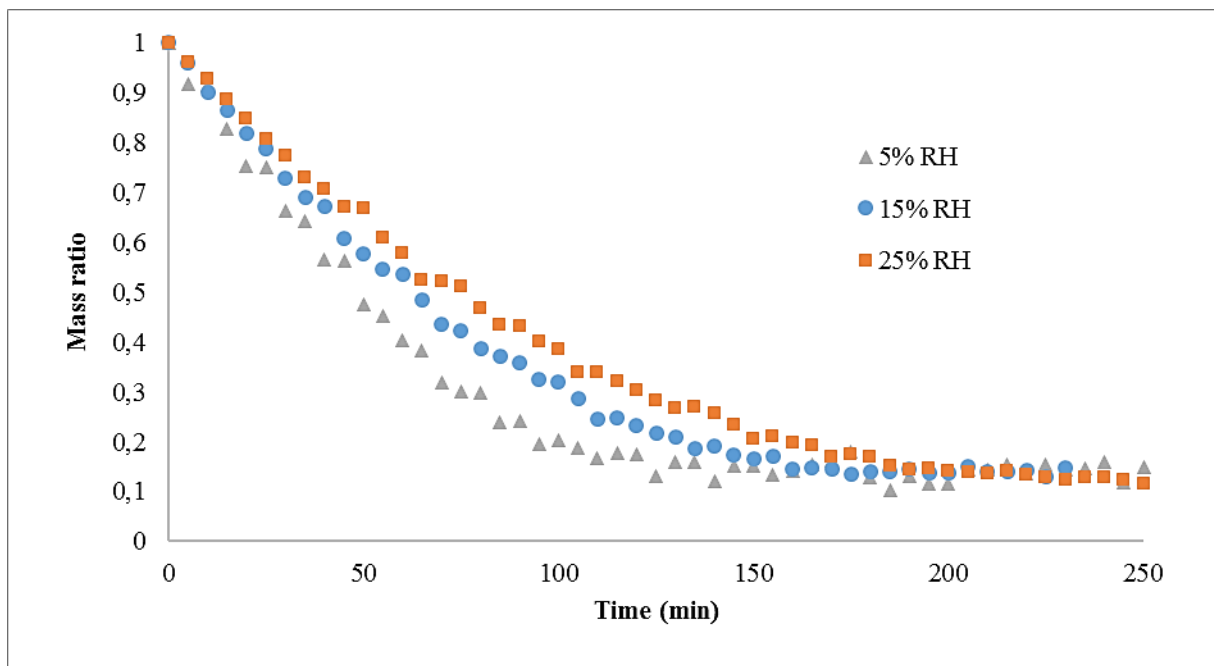


Figure 4-6: Effect of humidity on the sample mass evolution with time during convective drying of faecal sludge at constant pellet diameter of 8 mm, temperature of 60 °C and air flow velocity of 0.03 cm/s.

From the drying kinetics curves, it was observed that the air relative humidity had a considerable effect on the drying time of faecal sludge. At an air temperature of 60 °C and air velocity of 0.03 cm/s, increasing the relative humidity from 5% to 15% increased the drying time from 135 to 180 minutes and using air humidity of 25% increased the drying time to 220 minutes.

The influence of air relative humidity on the variation of the drying rate with the moisture ratio was investigated and the results are presented in Figure 4-7. The drying rate curves obtained give an insight on how the humidity affects the different drying rate periods. Both the constant rate period and the falling rate period are significantly affected by the change in relative humidity of the drying air. During the constant rate period, using air with a relative humidity of 5% resulted in the highest rate of moisture removal of around 30 g/min.m² compared to relative humidity of 15% and 25% which yielded

23 g/min.m² and 19 g/min.m² respectively. This observation was expected as the increase in the relative humidity of the drying air reduces the driving force of mass transfer rate, as explained in section 2.3.5.2. Increasing the relative humidity reduces the difference between the water content in the drying air and that on the surface of the solid undergoing the drying process.

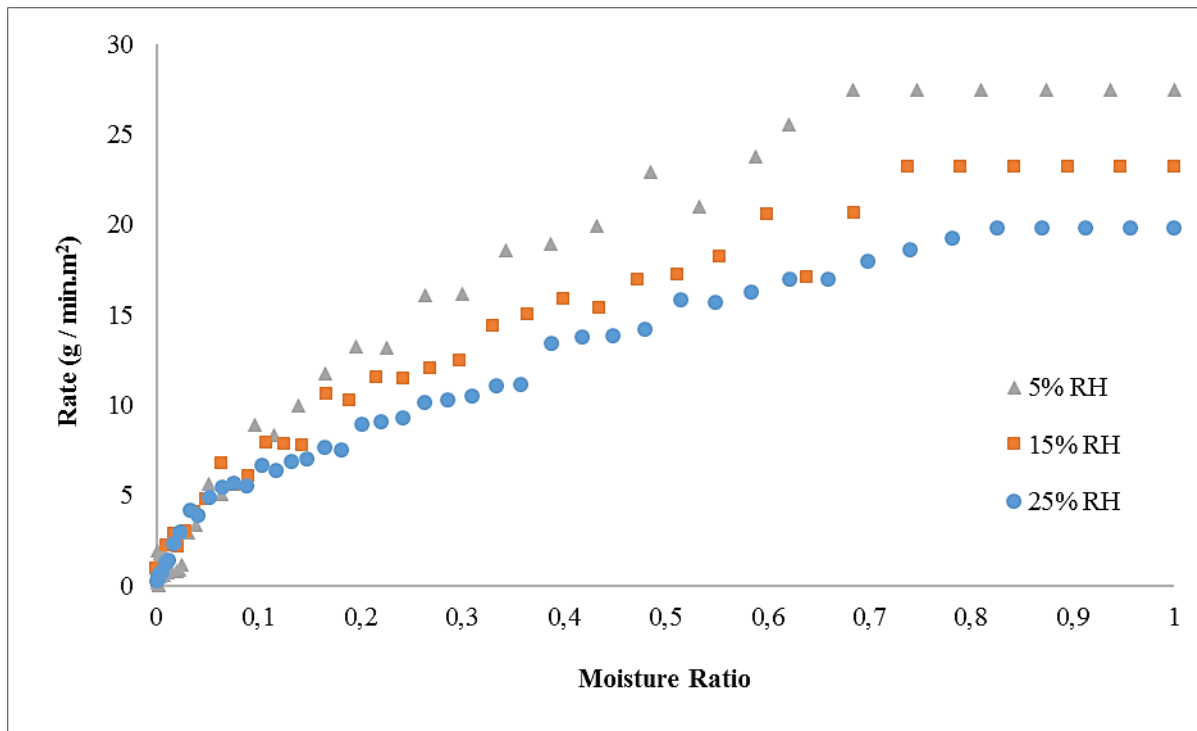


Figure 4-7: Effect of air relative humidity on the drying rate with moisture ratio during convective drying of faecal sludge at constant pellet diameter of 8 mm, temperature of 60 °C and air flow velocity of 0.03 cm/s

Figure 4-7 shows that the change in the relative humidity of the air used has an effect on the critical moisture content. Employing air with low relative humidity resulted in a prolonged constant rate period as compared to using air of higher relative humidity. The critical moisture content was observed at a moisture ratio of about 0.69 when drying using air with a relative humidity of 5%, whereas the critical moisture content was found to occur at a moisture ratio of about 0.83 when drying with a relative humidity of 25%.

4.1.4 Effect of sample thickness

In order to investigate the influence of sample thickness on the drying kinetics of faecal sludge, pellets of different diameters: 8 mm, 10 mm, 12 mm and 14 mm, were dried at an air temperature of 60 °C, air velocity of 0.06 cm/s and average relative humidity of 5%. The results are presented in Figure 4-8.

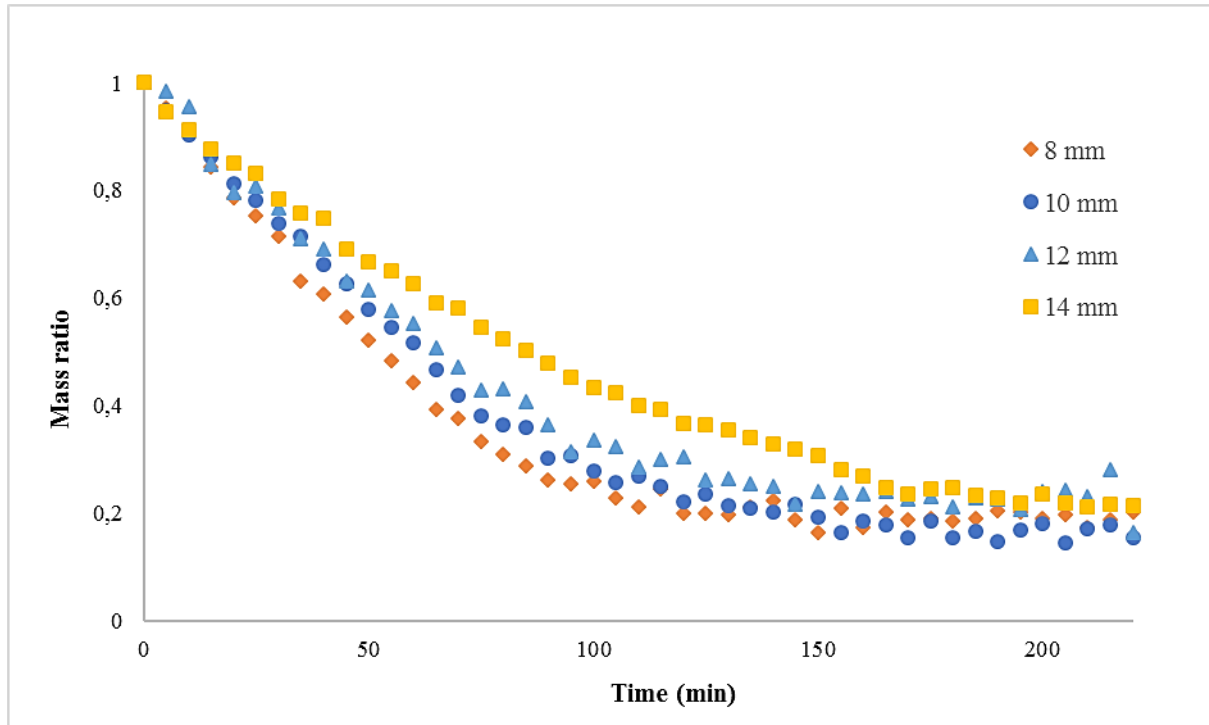


Figure 4-8. Effect of sample diameter on the sample mass evolution with time during convective drying of faecal sludge at constant temperature of 60 °C, relative humidity of 5% and air flow velocity of 0.03 cm/s.

It can be seen from Figure 4-8 that increasing the pellet diameter leads to an increase of the drying time as it takes approximately 135 minutes to dry an 8 mm sludge pellet as compared to the 200 minutes required to dry a 14 mm pellet under the same drying environment. It generally takes longer for larger samples to dry than smaller ones under the same operating conditions. This is an expected result as the distance that moisture travels within the sludge to the surface has a great contribution on the drying time.

To fully comprehend the effect of sample thickness on the different phases of drying as well as rate of moisture removal, drying rate curves were plotted and presented in Figure 4-9. It is immediately apparent from Figure 4-9 that sample size has a minor influence in the constant rate period.

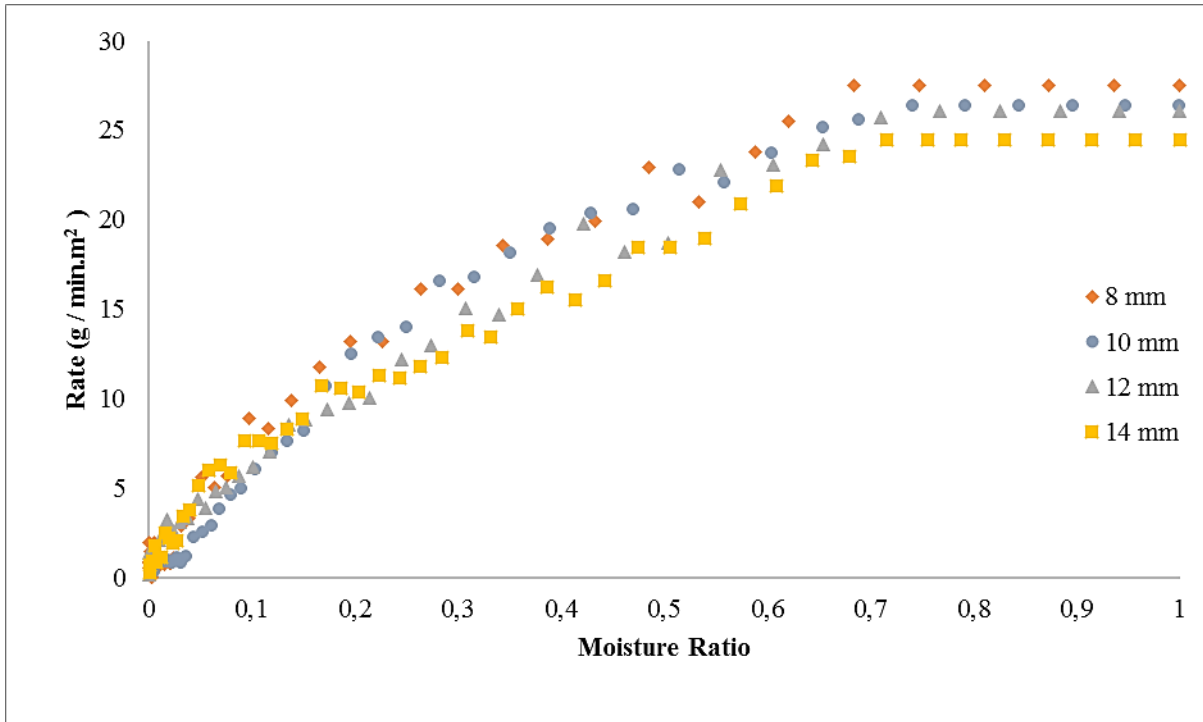


Figure 4-9: Effect varying pellet diameter on the drying rate with moisture ratio during convective drying of faecal sludge at constant temperature of 60 °C, relative humidity of 5% and air flow velocity of 0.03 cm/s

The drying rates during the constant drying period were close for the different pellet diameters, as the values ranged between 24.5 and 27 g/min.m². This is attributed to the fact that the constant rate period is mainly a convective mass transfer process which is primarily affected by the external drying conditions. The drying boundary layer occurs at the surface, therefore changing the pellet diameter would not affect the rate at which moisture is evaporated from the surface of the pellets thus yielding similar drying rate per surface area during the constant rate period. The difference in the total drying time indicates that the change in sample has a significant effect during the falling rate period. This shows that larger sample thickness causes a slower rate of moisture removal within the solid. Increasing the pellet diameter increases the mean path by which moisture has to migrate to reach the surface of the material for its evaporation.

The change in the pellet diameter did not affect the value of the critical moisture content as for it occurred around a moisture ratio of is 0.67. This observation may be due to the fact that the critical moisture content of a material is greatly influenced by external conditions.

4.1.5 Effect of air velocity

Many authors have studied the effect of air flowrate on convective drying and found that this depends on the material being dried and the air speed range, as discussed in section 2.3.5.3. The effect of air velocity on the drying kinetics of faecal sludge was investigated by conducting experiments using flow velocity of 0.03 cm/s, 0.06 cm/s and 0.12 cm/s. The other parameters were held constant, i.e. relative humidity of 5 %, temperature of 60 °C and sample diameter of 10 mm.

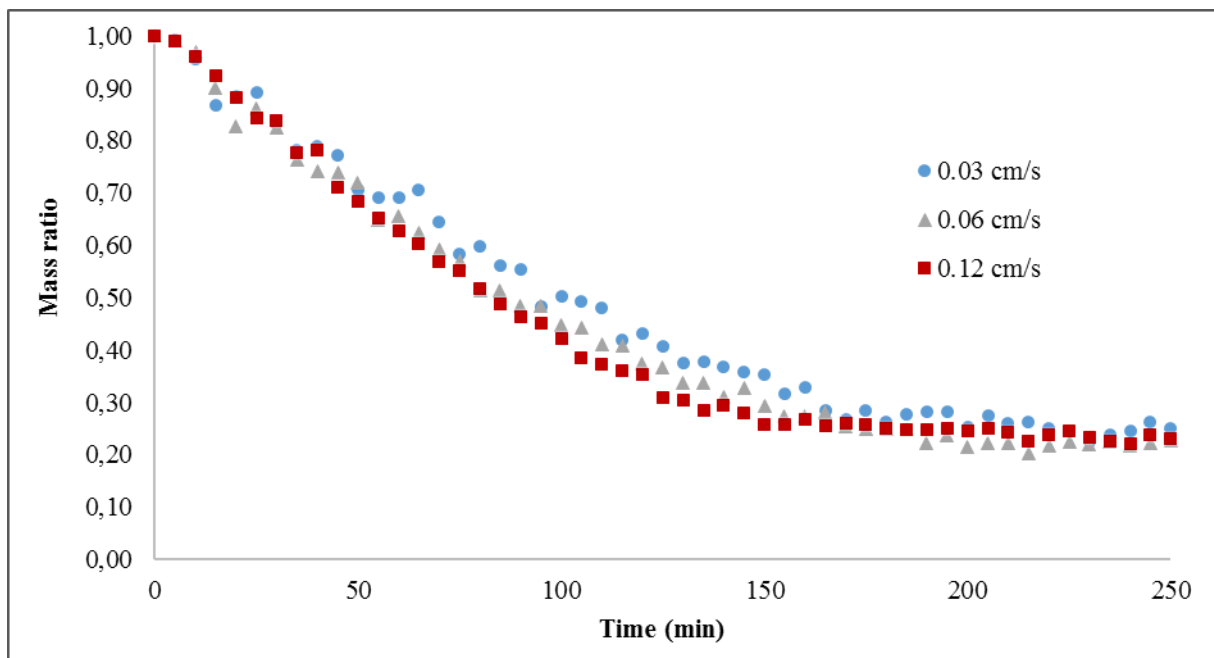


Figure 4-10: Effect of air velocity on the sample mass evolution with time during convective drying of faecal sludge at constant temperature of 60 °C, relative humidity of 5% and pellet diameter of 8 mm.

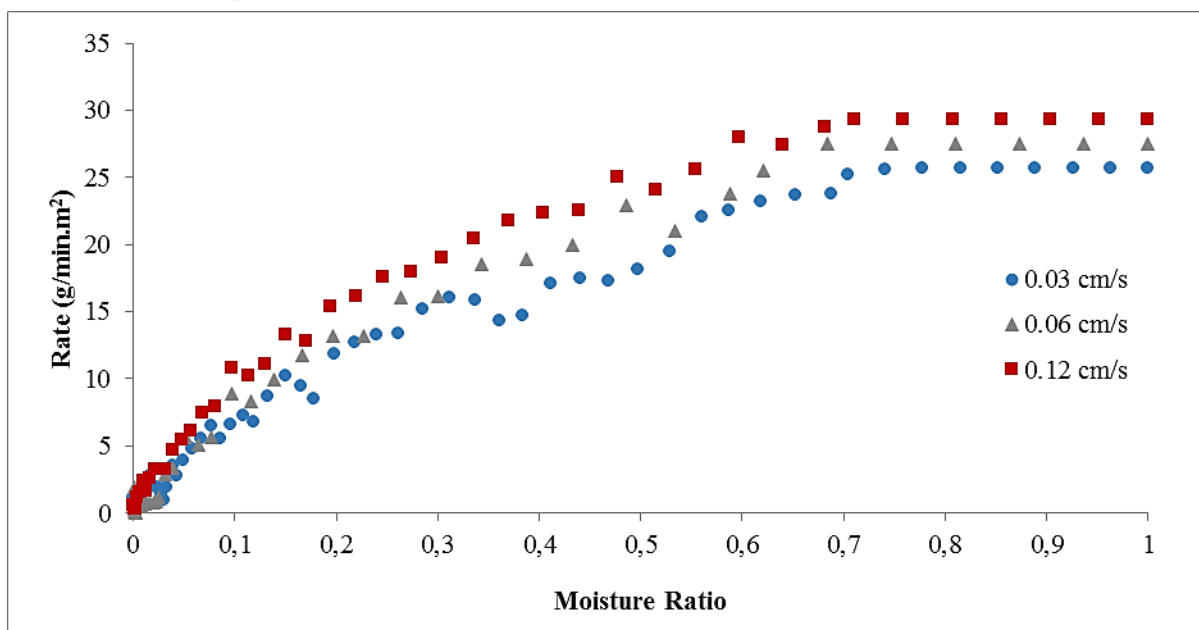


Figure 4-11: Effect of air velocity on the drying rate during convective drying of faecal sludge at constant temperature of 60 °C, relative humidity of 5% and pellet diameter of 8 mm

The effect of air velocity had a weak effect on the overall drying time which was approximately 200 minutes for the different cases. Figure 4-11 reveals that the air velocity has a notable influence on the constant rate. This behaviour is attributed to the moisture removal mechanism by convection during the constant rate period as increasing the velocity increases the Reynolds number and consequently the convective mass transfer coefficient. The effect of air velocity was significant during the falling rate period hence similar drying time were observed. Since diffusion is regarded as the main mechanism of moisture transfer during the falling rate, the mass transfer driving force is the moisture concentration gradient between the surface and the internal moisture. The concentration gradient is not a function of air flow but of air humidity. Looking at the effect of air velocity on the overall drying time, it can be concluded that for the particular case of VIP faecal sludge drying and within the range of study, increment in air speed does not have a considerable effect on the total drying time of the sample.

However, it should be noted that the average range of air velocities employed in this study were low in comparison to other convective drying studies. Air flow within the drying compartment was of a laminar flow regime, with a Reynolds number of less than 2100. Laminar flow results in low convective heat and mass transfer coefficient, which may have resulted in the perceived insignificant contribution of air velocity to the drying time.

4.2 Analysis of thermal properties

This section is aimed at characterising the thermal properties of product obtained from convective drying of faecal sludge from pit latrines. The key parameters focused on in this section are thermal conductivity, heat capacity and the calorific value. Data pertaining to thermal properties is critical in the design of a dryer as the storage and propagation of heat in sludge is governed by these properties.

4.2.1 Calorific value

One of the potential end uses of dried faecal sludge is as a biofuel for heating and energy production, therefore the calorific value is an essential parameter to characterise. The effect of the drying conditions on the calorific value was investigated by determining the calorific value of the samples dried at varying temperature and humidity. The effect of air flow rate on the calorific value was not investigated as many researchers on convective drying came to the conclusion that it had no considerable effect on the calorific value (Vesilind and Ramsey, 1996).

In order to ascertain the general effect of drying on the calorific value of sludge, an analysis was performed on raw sludge that was oven dried to ensure that all the moisture was evaporated. The calorific value of the raw sludge was found to average 17.5 ± 1 MJ/kg dry solids. This value was in the range of the calorific values found in literature. A study conducted by Komakech et al. (2014) to characterise municipal waste in Kampala found the average calorific value to be 17.3 MJ/kg dry solids.

Zuma et al. (2015) analysed the calorific value of 10 VIP latrines in the eThekweni municipality in Durban, South Africa and found the average calorific value of 14.3 MJ/kg dry solids.

The heat capacity of the dried pellets was similar to that of coffee husks and firewood, sawdust, but considerably lower than charcoal, used engine oil and diesel, presented in table 2.3.

Calorific values of dried pellets processed at different temperatures presented in Figure 4-12.

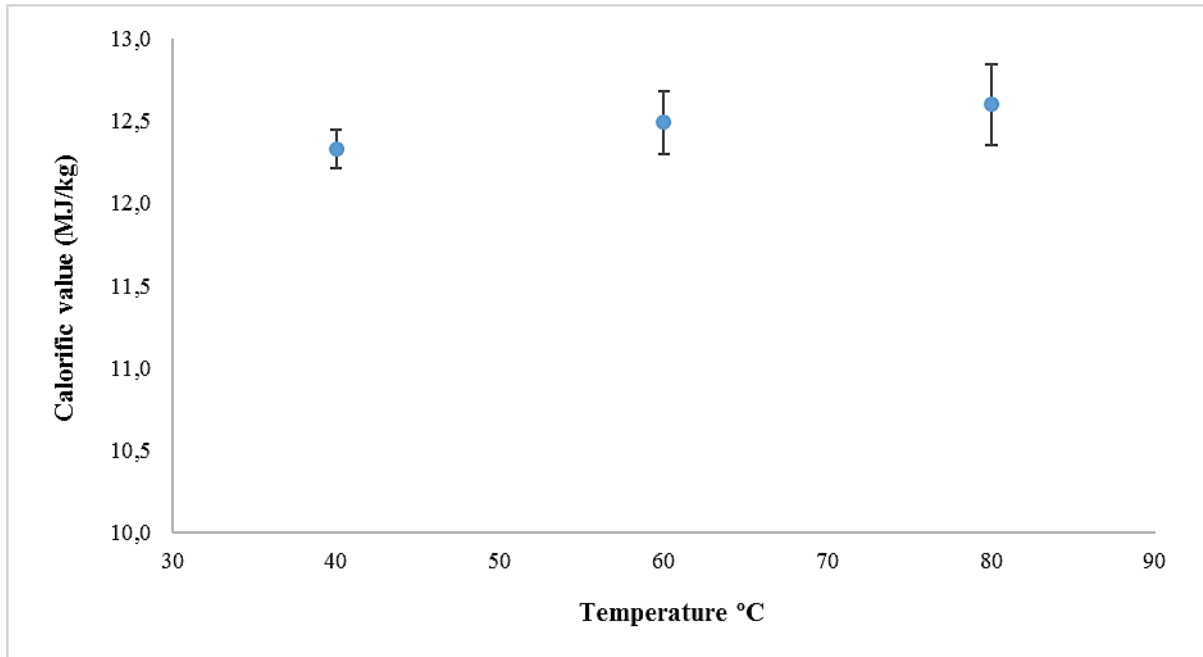


Figure 4-12: Influence of drying temperature on the resulting calorific value of sludge pellets dried at constant pellet diameter of 8 mm, relative air humidity of 5 % and air flow velocity of 0.03 cm/s

As shown in Figure 4-12, there was no significant variation in the calorific value of sludge dried using the different air temperatures with the average value around 12.5 MJ/kg wet solid. This observation maybe corroborated to the amount of volatile solids contained by the dried product, which can influence the calorific value of a solid fuel. An investigation of the effect of drying air temperature on the volatile solids was performed and the results depicted in Figure 4-13.

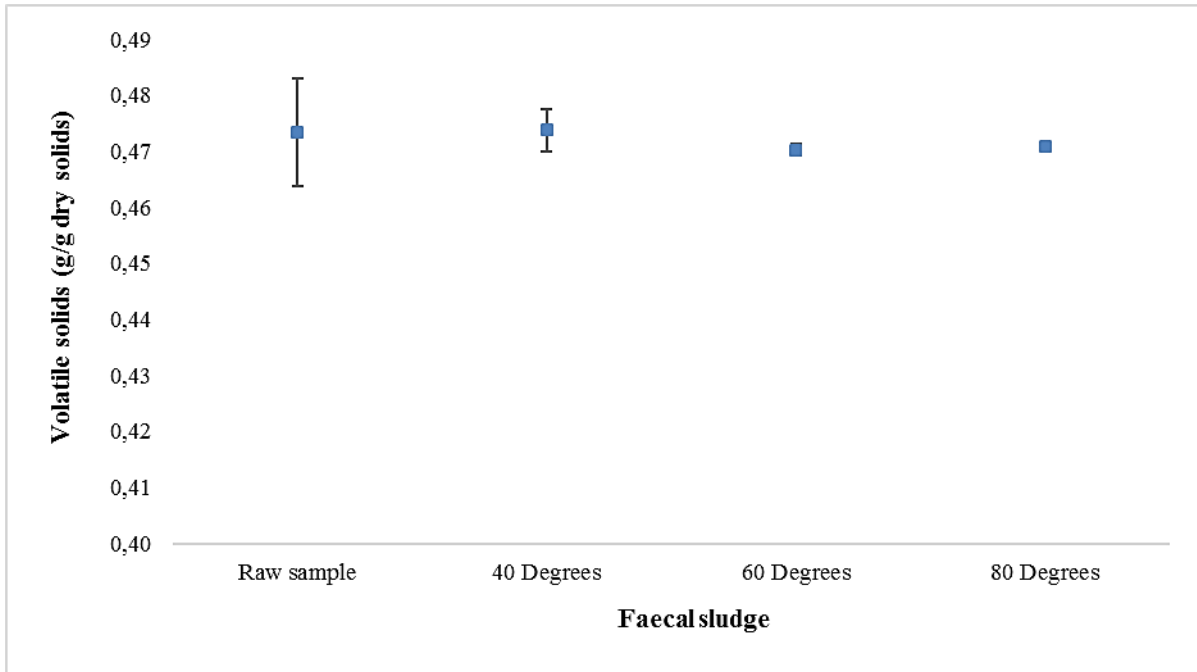


Figure 4-13: Graph displaying the variation of the volatile solids contained in the raw sample (wet) and the sludge pellets dried at different temperatures but at constant pellet diameter of 8 mm, air relative humidity of 5 % and air flow velocity of 0.03 cm/s

As depicted in Figure 4-13, there was not any significant change in the amount of volatile solids contained in the sludge whilst varying the drying air temperature. This observation can be attributed to the low air drying temperature range employed for this study which were below 100 °C. When sludge is exposed to higher temperatures, volatile solids are released from the sludge due to thermal degradation of the organic matter from the sludge. However, the effect of the thermal degradation becomes significant at temperatures higher than 105 °C. This implies that sludge exposed to temperatures greater than 105 °C can lose a significant amount of volatile solids to effect a considerable change in the calorific value. These finding corresponds with the literature in the thermal drying of wastewater sludge (Vesilind and Ramsey, 1996).

The effect that the air relative humidity has on the calorific value was investigated and the results are presented in Figure 4-14.

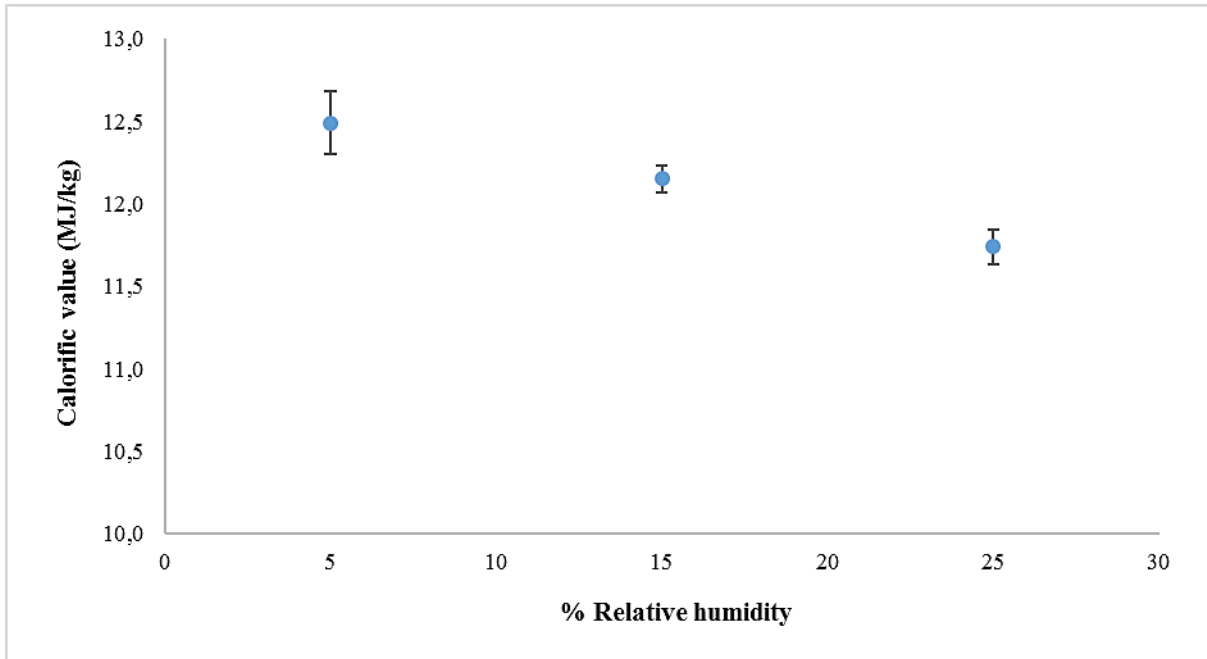


Figure 4-14: Influence of drying relative humidity on calorific value of sludge pellets dried at constant pellet diameter of 8 mm, temperature of 60 C and air flow velocity of 0.03 cm/s

As illustrated in Figure 4-14, it can be seen that the air relative humidity, under which this study was conducted, has an effect on the resulting calorific value of the dried product. This observation could be attributed to the fact that increasing the operating humidity of the drying air results in an increase of the equilibrium moisture content, therefore it lowers the calorific value as some of the heat released during oxidation would be employed to evaporate the residual moisture.

4.2.2 Thermal conductivity

Thermal conductivity is an intensive parameter essential in evaluating the ability of a solid material to conduct heat. One of the objectives of this study is to evaluate the thermal conductivity of the dried sludge and also evaluate its variation with the moisture content within the solid.

The initial value of thermal conductivity of wet faecal sludge before drying was found to be 0.55 W/m.K. This value is in the vicinity of the thermal conductivity of pure water of 0.6 W/m.K. This is due to the initial high water content of the sludge (80 % of total mass).

The thermal conductivity of the dried pellets was evaluated and the results are presented in Figure 4-15.

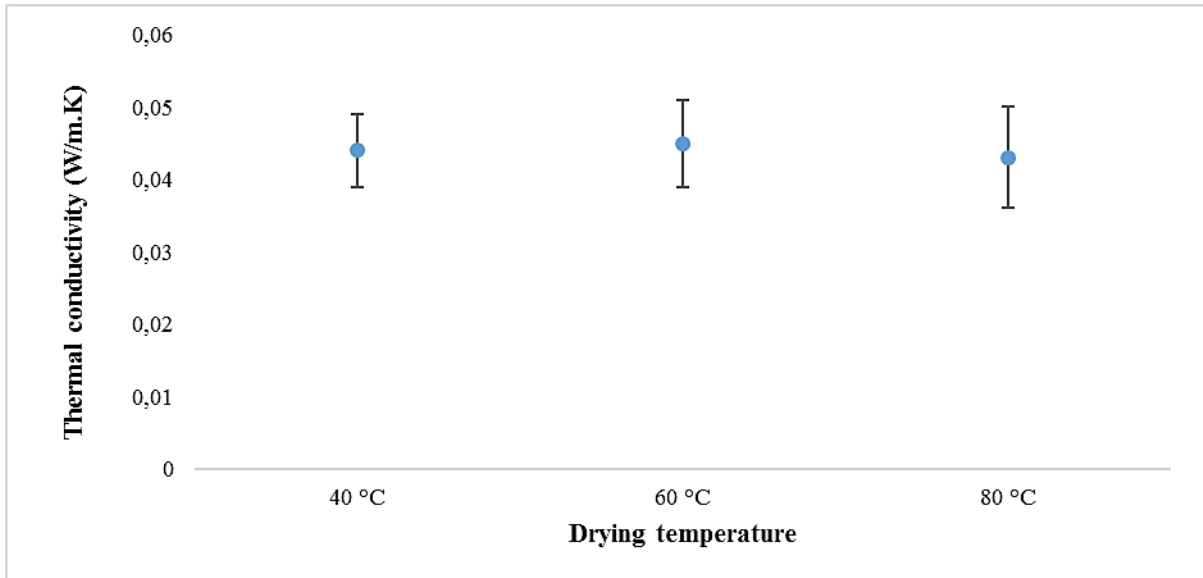


Figure 4-15: Influence of drying temperature on thermal conductivity of sludge pellets dried at constant pellet diameter of 8 mm, relative air humidity of 5 % and air flow velocity of 0.03 cm/s

There was no considerable variation in the final thermal conductivity of the pellets dried at different air temperatures, as shown in Figure 4-15. The average thermal conductivity was considerably lower than wet sludge at values as low as 0.044 W/m.K, a value comparable to that of air. This could be indicative of the large void spaces in the solid which are occupied by air after the sludge has been dried, leading to the decrease of the overall conductivity of the material (Hanson et al., 2000, Bart-Plange et al., 2009). The variation of thermal conductivity was analysed by removing the sludge at different time intervals during the drying process, and the moisture content and the thermal conductivity were evaluated. The variation of thermal conductivity of VIP sludge with the moisture content is presented in Figure 4-16..

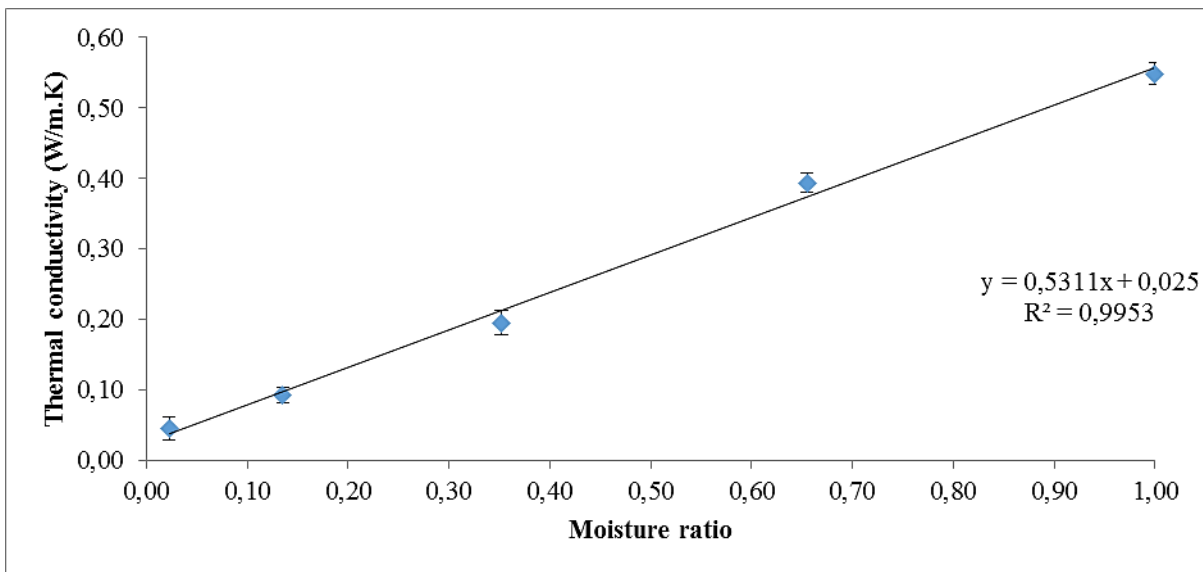


Figure 4-16: Influence of moisture content on thermal conductivity of sludge pellets dried at constant pellet diameter of 8 mm, relative air humidity of 5 % and air flow velocity of 0.03 cm/s

It is apparent that moisture content has a significant effect on the thermal conductivity. This is as a result of the contribution of water within the sludge. The thermal conductivity increased linearly with the increase in moisture content (with the coefficient of determination, R^2 equal to 0.995). This trend agrees with the results obtained by Hanson et al. (2000), on the study of high water content material drying.

4.2.3 Heat capacity

The ability to predict the heat capacity as drying of the sludge proceeds can be critical, especially for the calculation of energy requirements for the design of dryers that are aimed at processing faecal sludge. The heat capacity of raw sludge was initially determined to be 3.8 kJ/kg.°C, a value close to that of water (~ 4.2 kJ/kg.°C). This observation can be explained by high initial water content of the sludge (80 % of total mass).

The thermal conductivity of the dried pellets was evaluated and the results are presented in Figure 4-17

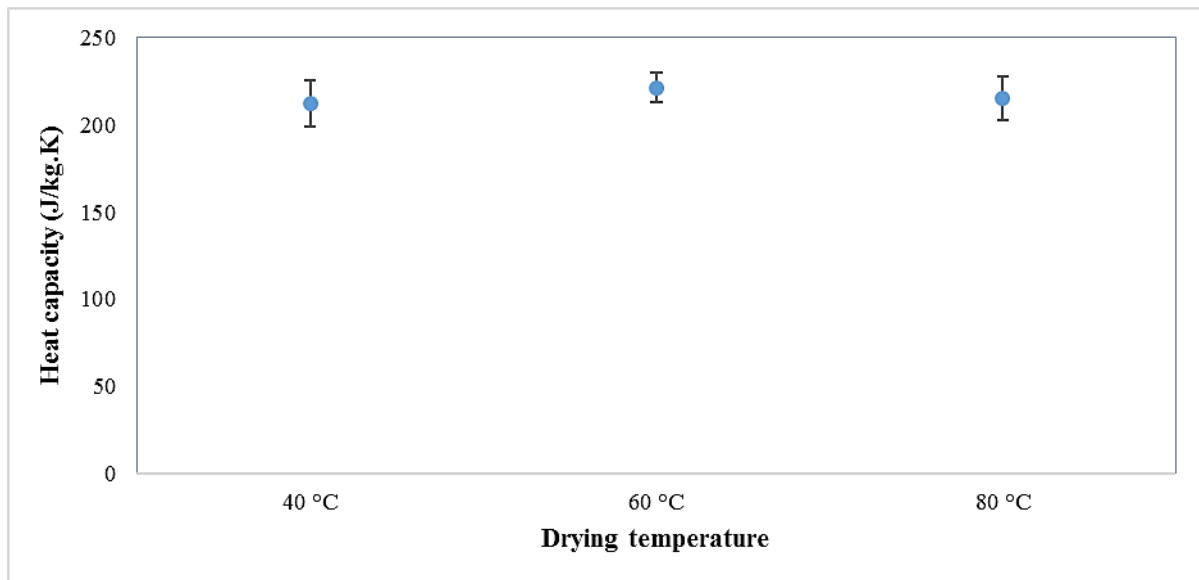


Figure 4-17: Influence of drying temperature on thermal conductivity of sludge pellets dried at constant pellet diameter of 8 mm, relative air humidity of 5 % and air flow velocity of 0.03 cm/s

As shown in Figure 4-17, there was no considerable variation in the heat capacity of the pellets dried at different air temperatures. The average values of heat capacity of the dried sludge ranged from 200 - 220 W/m.K. The heat capacity at different moisture contents was investigated by varying the duration of the samples in the dryer, and then evaluating their moisture content as well as heat capacity. The results are presented in Figure 4-18.

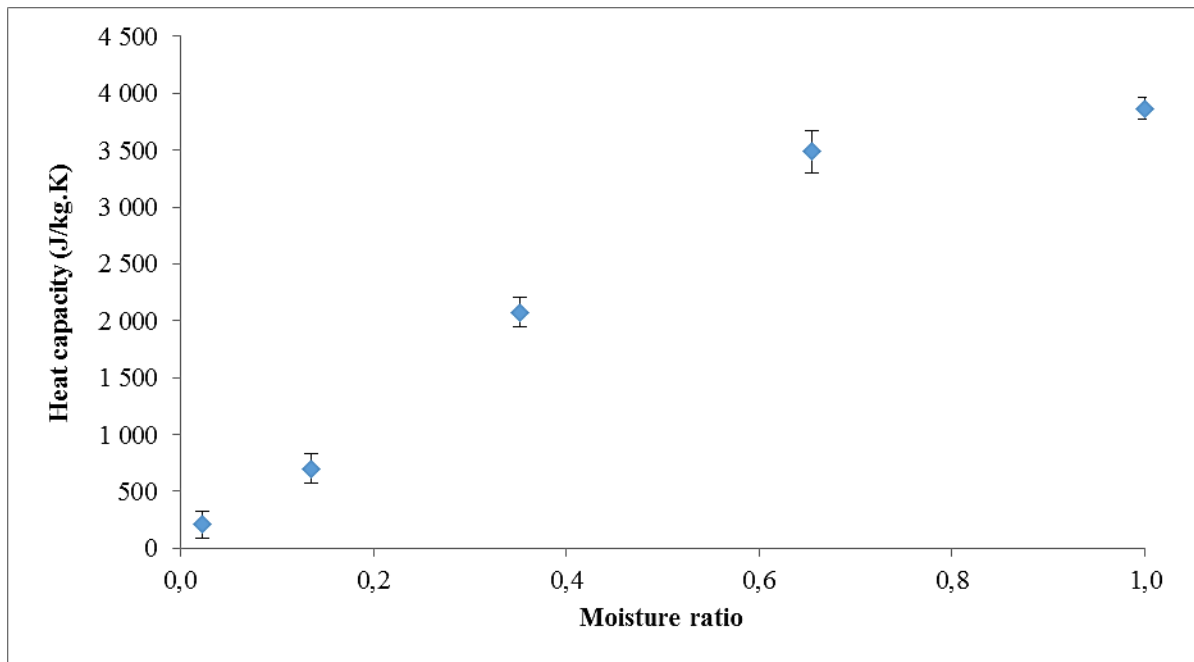


Figure 4-18: Variation of heat capacity with respect to the moisture ratio

It is apparent that moisture content has a significant effect on the heat capacity of sludge. The heat capacity decreased as the moisture content within the sample decreased. This is as a result of the contribution of water within the sludge.

4.3 Nutrient analysis

One of the possible uses of dried faecal sludge is in agriculture as a fertilizer, therefore it is of importance to determine the nutrient content and its variation with respect to the drying conditions. The concentration of the nutrients that were analysed to evaluate the value to agricultural of the dried sludge were K, P, Mg, Ca, PO_4^{3-} , NO_3^- , and NO_2^- . Only the effect of drying temperature was investigated as it was reported in literature as the main parameter that may alter the nutrient concentration within a sample (Sablani, 2006, Morris et al., 2004, Arslan and Özcan, 2008).

The effect of drying temperature on the total nutrient elements and nutrient ions are illustrated in Figure 4-19 and Figure 4-20 respectively.

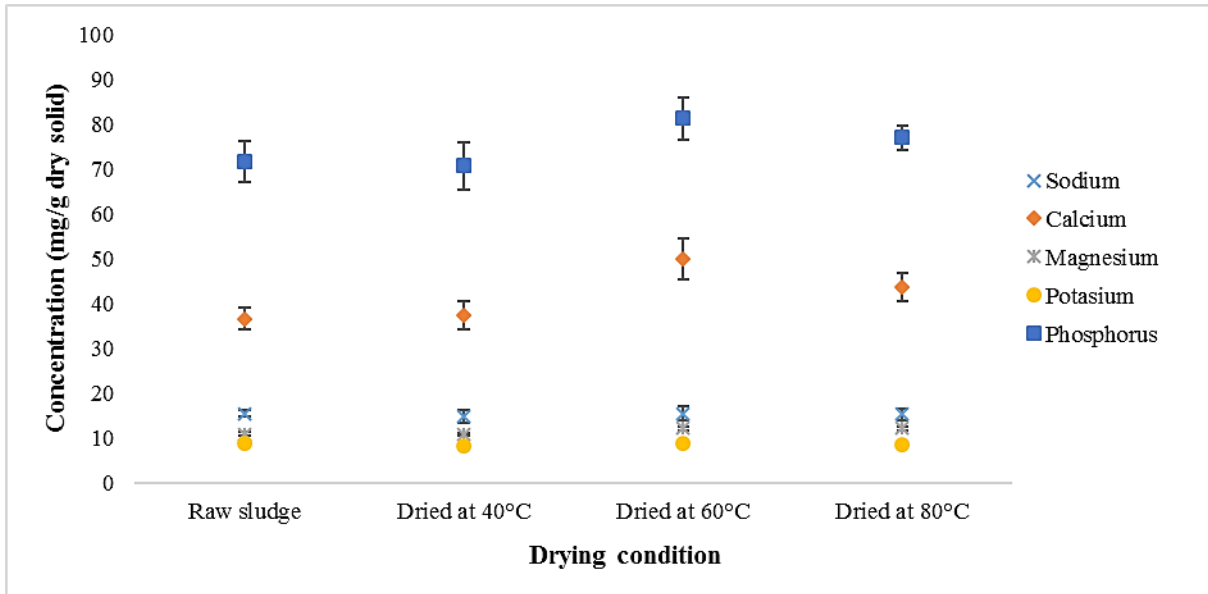


Figure 4-19: Variation of the concentration of the concentration of sodium, calcium, magnesium, potassium and phosphorus between raw sludge and sludge dried under different temperatures

Figure 4-19 shows that all the nutrient elements that were investigated did not exhibit any variation in concentration with drying temperature as the concentrations of nutrients in the raw sludge were similar those in the dried pellets. The concentrations for sodium, calcium, magnesium, potassium and phosphorus were 15.5, 42.1, 11.2, 8.6 and 4.7 mg / g dry solid respectively.

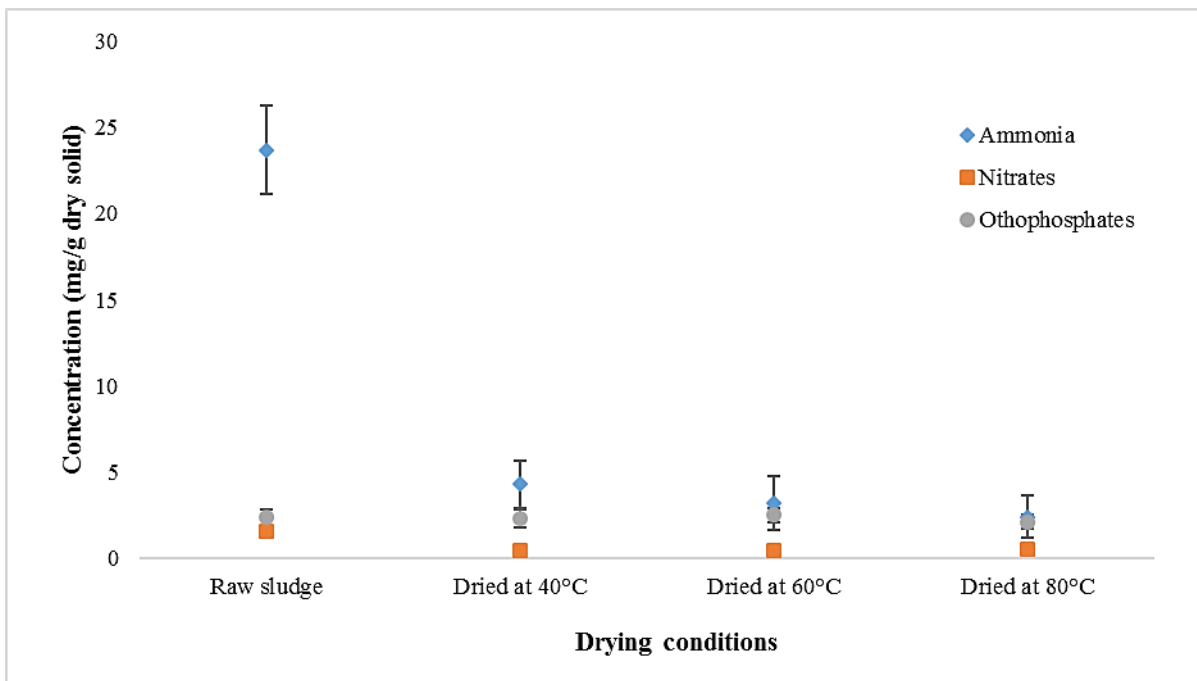


Figure 4-20: Variation of the concentration of ammonia, nitrates and orthophosphates between raw sludge and sludge dried under different temperatures

The concentration of orthophosphates did not show any variation between the initial concentration in raw sludge and that in the dried pellets. The average concentration of orthophosphates in the sludge was 2.5 mg / g dry solid. However, there was a reduction in the nitrates and ammonia concentration between the raw sludge and the dried pellets. The reduction in ammonia concentration was most significant as the concentration dropped from 24 mg / g dry solid in the raw sludge to around 4.5 mg / g dry solid. The concentration of nitrates decreased from 1.5 mg / g dry solid in the raw sludge to around 0.45 mg / g dry solid. These observed changes in the nutrient concentrations could have either been as a result of the increased volatilization of both ammonia and nitrates that occurs as temperature is increased or stronger bonding of the compounds to the solid matrix as temperature was elevated.

It is evident that faecal sludge contains nutrients that are of agronomical value. Table 4-1 summarises the results of this study and also compares with literature values.

Table 4-1: Comparison between nutrient concentration from literature and that found in this study

Nutrients	Raw sludge]	Dried product	Nikiema et al. (2013)	Mnkeni and Austin (2009)	Komakech et al. (2014)
	[mg/kg dry solid]	[mg/kg dry solid]	[mg/kg dry solid]	[mg/kg dry solid]	[mg/kg dry solid]
P	71	72	12.4	3	2.7
K	8.7	8.6	6.1	44	19.5
Ca	43.5	42.1		4	
Mg	11.2	11.2		7.9	
PO ₄ ³⁻	2.4	2.3			0.37
NO ₃ ⁻	0.5	0.5	2.9		
NH ₃	24	4.5			

The concentration of total phosphorus was significantly higher compared to that found in literature as showed in Table 4-1. The marked discrepancy could be as a result of the difference in diet as the amount on chemical is greatly influenced by the diet, health and age of individuals and therefore data available is greatly dependent on the source population. This difference can also be seen from the different values of total potassium presented in by Nikiema et al. (2013) in their study of using pellet faecal sludge as

fertilizer Mkeni and Austin (2009) when analysing the fertiliser value of contents from urine diversion toilets.

Compared to popular manure used for agriculture, the concentration of nutrients within the sludge found in this study were greater some the same nutrients detailed in Table 2-4. In particular, the nutrients concentrations are higher compared to cattle manure which is commonly used as supplements to inorganic fertiliser and also as a soil conditioner.

4.4 Empirical modelling of the drying of VIP sludge

Mathematical modelling is crucial for predicting and optimising drying process. Accurate and reliable models are useful in the design and utilisation of drying equipment. The objective of this section is to propose a mathematical model that describes the drying kinetics of faecal sludge as a function of the drying air parameters by investigating the applicability common empirical drying models in describing the experimental results. These empirical models are regarded by various authors in the drying field as simple alternatives to Fick's second law of diffusion (Yu et al., 2009, Vega et al., 2007, Simal et al., 2005). These models describe the relationship between the moisture content of the material and the drying time at various drying environments (air temperature, air relative humidity, air flow rate and sample size). Several empirical models have been widely used to describe isothermal convective drying by various authors, mostly in the food industry (Doymaz, 2008, Karathanos, 1999, Doymaz, 2005, Van Boekel, 2008, Vega et al., 2007) and also for the modelling of sewage sludge (Qian et al., 2011, Arlabosse et al., 2005, Yu et al., 2009). It is of interest to evaluate the applicability of these models in describing the drying of faecal sludge from VIP toilets.

The kinetic drying data obtained from the pellet and flat slab drying was fitted to some common empirical drying models, which were the Newton model, the Page model, the modified Page model, the Two-term exponential model and the Logarithmic model. These models were chosen because they are regarded as the most accurate in describing various food material as well as sludge from wastewater treatment (Léonard et al., 2005, Kucuk et al., 2014, Celma et al., 2012). An optimization tool, SOLVER (GRG2 method), included in Microsoft Excel 2013, was used for the regression of the parameters of each of the models by minimising the sum of the square differences between the experimental and the calculated values.

Figure 4-21 shows the variation between the predicted moisture content ratio from the models and the experimental values of pellets.

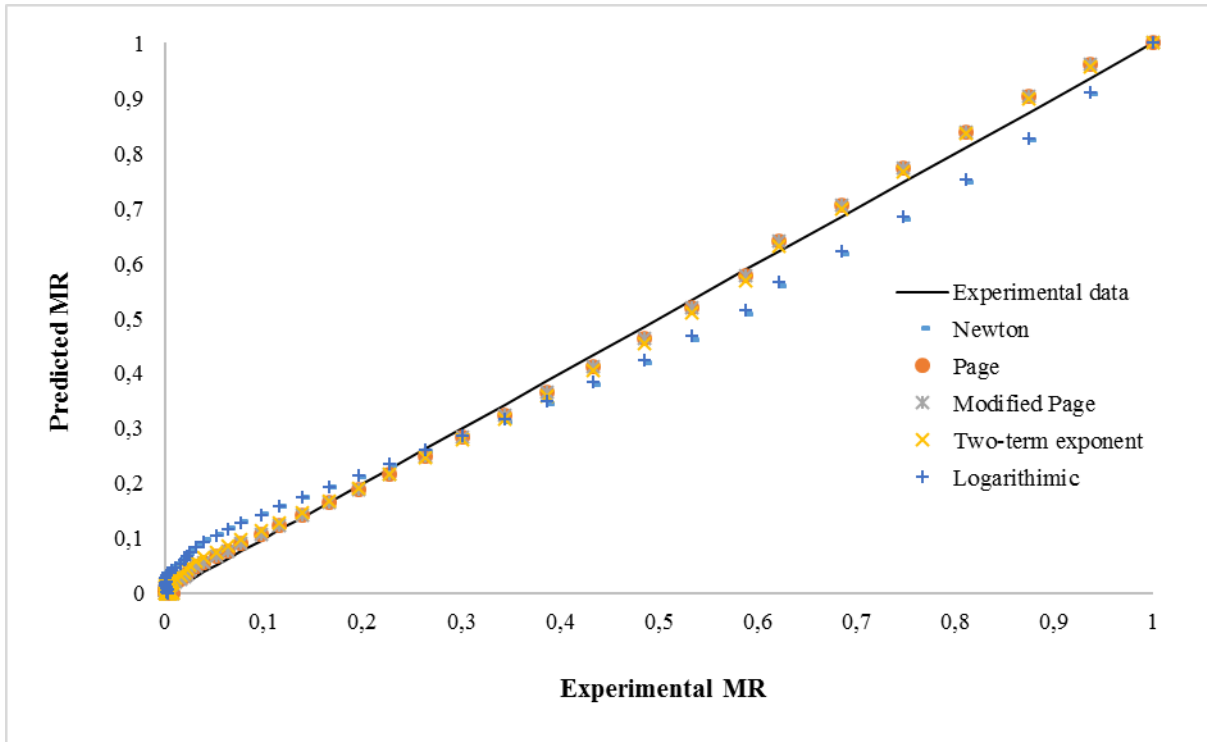


Figure 4-21: Graph predicting the Moisture Ratio versus experimental Moisture Ratio for different kinetic empirical models

From Figure 4-21, it can be observed that the Newton model exhibited the largest deviation from the experimental data, followed by the Logarithmic model. The Page model and the Modified Page were found to give better predictions of the experimental moisture as compared to the rest of the models with the root mean square error (*RMSE*) and the coefficient of regression (R^2) values used as the goodness of fit indicators. The Page model provided the best results as it had the lowest values of *RMSE* and highest value of R^2 for the different temperatures at each set of drying conditions for both configurations investigated. The Newton model was the least accurate with the largest *RMSE* and lowest R^2 values as can be seen in Table 4-2. This result agrees with the results obtained by Celma (2011) during the investigation of drying characteristics of sludge from tomato processing plants.

It can be observed from Figure 4-21 that the models tend to under or overestimate the experimental moisture ratio at various stages of the drying process. At an *MR* of 0.26 or lower, there is a transition of all models analysed from under estimation of the experimental data to overestimating it. This behaviour was also observed by Simal et al. (2005) when the empirical models were used to predict the drying kinetics of kiwi fruit. The Logarithmic and the Two-term models underestimate the experimental data from the initial stages of the drying process ($MR = 1$) to a region where the moisture ratio, *MR*, is approximately 0.26. Whilst the Page model and the Modified Page exhibit two transition points, one at *MR* of 0.77 and the other at 0.26.

Table 4-2: Regressed parameters of the empirical model analysed for pellets

Model	Temperature	Model constants			RMSE	R ²
Newton	40	k = 0.01489			0.06172	0.963
	60	k = 0.01934			0.05573	0.965
	80	k = 0.02892			0.04524	0.972
Page	40	k = 0.00285	n = 1.372		0.01306	0.997
	60	k = 0.00466	n = 1.341		0.01174	0.998
	80	k = 0.00482	n = 1.474		0.01729	0.996
Modified Page	40	k = 0.01340	n = 1.373		0.01306	0.997
	60	k = 0.01825	n = 1.341		0.01174	0.998
	80	k = 0.01589	n = 1.479		0.01729	0.996
Two-term	40	k = 0.02127	a = 1.876		0.02572	0.991
	60	k = 0.02758	a = 1.869		0.02013	0.993
	80	k = 0.04385	a = 1.993		0.01837	0.994
Logarithmic	40	k = 0.01279	a = 1.063	c = -0.0634	0.04057	0.970
	60	k = 0.01863	a = 1.014	c = -0.0142	0.04392	0.969
	80	k = 0.02671	a = 1.031	c = -0.0304	0.03591	0.977

The Page model described the drying data the best of all the models investigated and the model constants, k and n , were correlated with temperature and sample size. This was done with the goal of developing a simple correlation that predicts the constants that can be used to determine the moisture ratio for different air temperatures and pellet diameters. These two parameters were considered because it was observed from the drying kinetics that the temperature and pellet diameter had a significant effect on the drying rate. In contrast, the air velocity had a negligible effect on the drying time hence it was not considered. The contribution of air relative humidity was not considered because of the assumption that most driers operate with relatively dry air.

These assumptions simplified the mathematical formulation of the resulting correlation. The variation with temperature of the Page model's k constant was investigated on both the flat slab configuration and the pellets with the results shown in Figure 4-22.

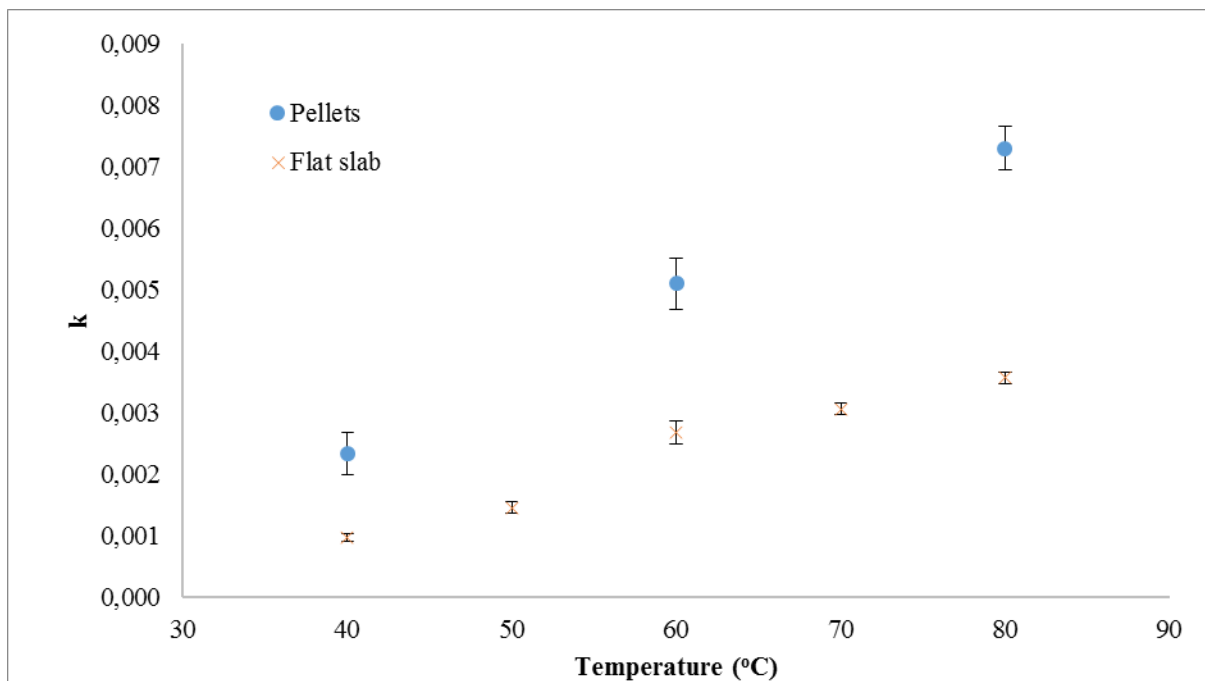


Figure 4-22: Variation of the k parameter from the Page model as a function of temperature for both flat slab configuration and pellets

The k values increased with increasing temperature. However, the n constant of the page model did not show any trend as the temperature and pellet diameter was changed as shown in Table 4-3. This observation is consistent with the results found by Doymaz et al. (2006) when they investigated the drying characteristics of dill and parsley leaves using air temperatures between 40 °C and 70 °C. Other studies that came to the same observation include the investigation of hot-air drying of cauliflower by Sharma et al. (2008) and the modelling of the hot-air drying kinetics of red bell pepper by Vega et al. (2007). These studies used temperatures between 40 °C and 80 °C, which is the same range of temperature used for this project.

Table 4-3: Variation of Page model constant, n , with the drying conditions

Drying conditions	Page model constant, n
Varying drying temperature at constant air velocity of 0.06cm/s, air relative humidity of 5% and pellet diameter of 8 mm	
40°C	1.37 ± 0.3
60°C	1.34 ± 0.3
80°C	1.47 ± 0.5
.Varying pellet diameter whilst drying at constant temperature of 60°C, air relative humidity of 5% and velocity of 0.06cm/s,	
10 mm	1.34 ± 0.2
12 mm	1.44 ± 0.6
14 mm	1.37 ± 0.4

The variation of the Page constants with temperature and diameter was investigated and equation (4-1) was proposed to determine the moisture ratio at a particular drying time, drying temperatures and pellet diameter. The formulation of the drying model is presented in Appendix D.

$$MR = ad^c T^b t^n \quad (4-1)$$

Where T is the drying temperature (°C)

t is the time (min)

a , b and c are constants

d is a pellet diameter (mm)

The model constants a , b and c were regressed for using non-linear regression coupled with multiple regression analysis and were found to be 5×10^{-5} , -1.081 and 1.6622 respectively.

The results of the developed model estimating the moisture ratio at different temperature and pellet diameter are shown in Figure 4-23 and Figure 4-24.

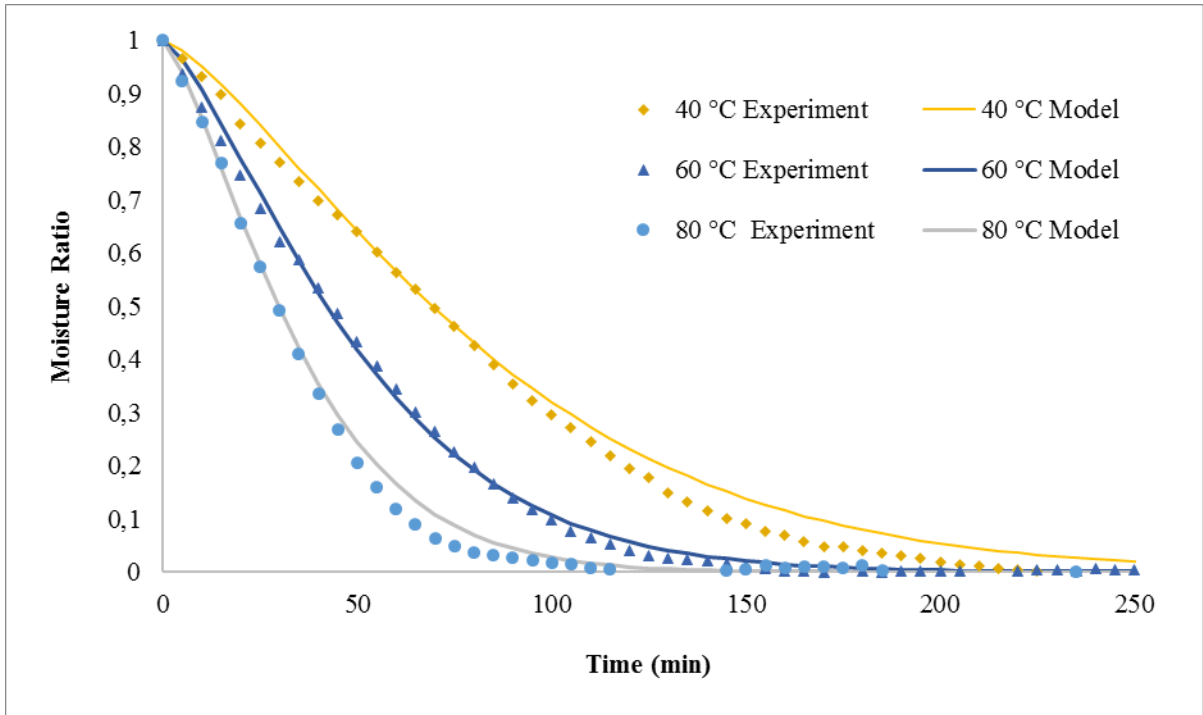


Figure 4-23: Values for the experimental and predicted moisture ratio for the developed model at different temperatures for convective drying of faecal sludge at constant pellet diameter of 8 mm, relative air humidity of 5 % and air flow velocity of 0.03 cm/s

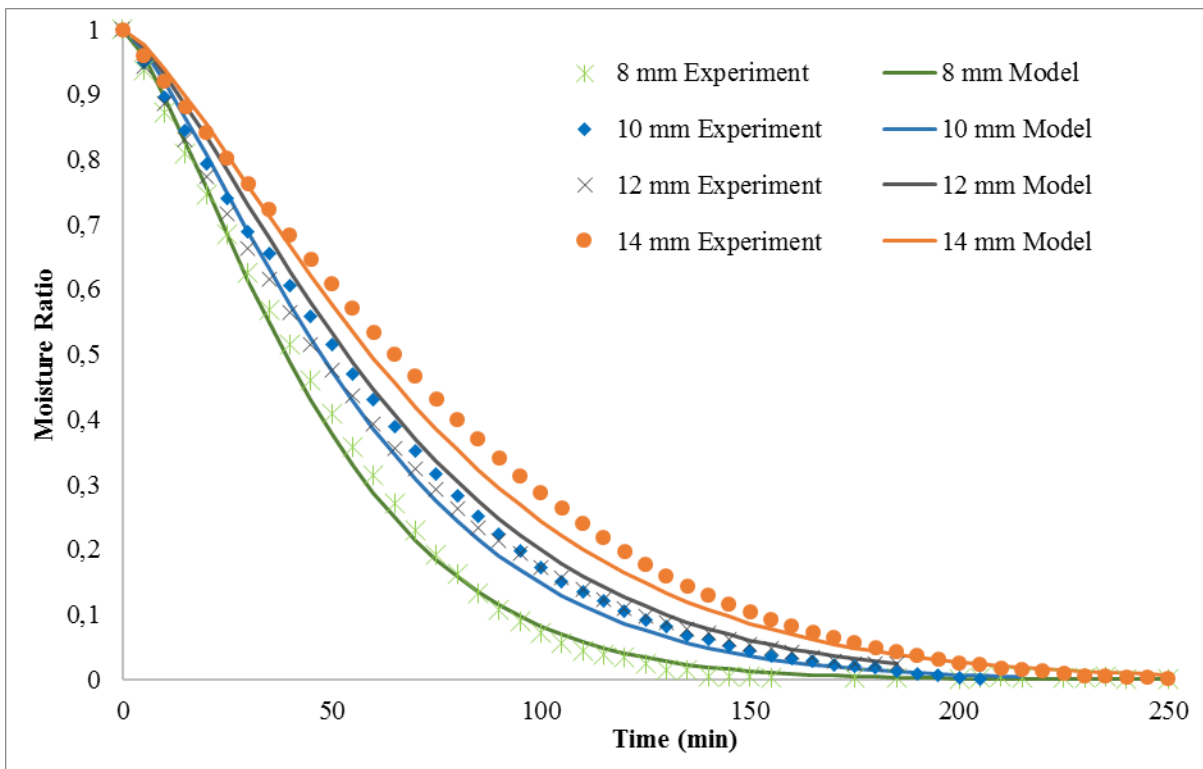


Figure 4-24: Values for the experimental and predicted moisture ratio for the developed model at pellet diameters for convective drying of faecal sludge at constant temperature of 60 °C, relative humidity of 5% and air flow velocity of 0.03 cm/s.

The model developed agreed well with the experimental results when using air with low relative humidity (5%) for the temperatures and pellet diameters investigated. The page model and the developed model are compared in Table 4-4

Table 4-4: Comparison of the goodness of fit parameters between the Page model and the developed model

Drying conditions	RMSE		R ²	
	Page model	Developed model	Page model	Developed model
Varying drying temperature at constant air velocity of 0.06cm/s, air relative humidity of 5% and pellet diameter of 8 mm				
40°C	0.01096	0.03157	0.997	0.979
60°C	0.01687	0.02047	0.998	0.992
80°C	0.01826	0.04371	0.996	0.971
Varying pellet diameter whilst drying at constant temperature of 60°C, air relative humidity of 5% and velocity of 0.06cm/s,				
10 mm	0.02174	0.03694	0.994	0.984
12 mm	0.01893	0.05893	0.995	0.965
14 mm	0.02627	0.09385	0.987	0.892

The developed model predicted the drying kinetics well at different temperatures despite having lower and higher *RMSE* values compared to the Page model. The lowest *R*² value for predicted model was 0.971 obtained at 80°C. This implies that the model is better compared to the Newton model which had the highest *R*² value of 0.971 as shown in Table 4-2. However, the deviations became larger when the pellet diameter was changed as shown in Figure 4-24 with the highest *R*² value being 0.965. The inability of the developed model to closely predict the experimental data could be as result of the assumption made during the formulation that the model parameter, *n*, had a constant value of 1.36. This assumption was made because the Page model parameter, *n*, did not exhibit any trend when the temperature and the sample diameter was varied hence an average value was used.

Despite the Page model predicting well the experimental data as compared to the developed model, its inability to make use of input such as the drying conditions (temperature and sample diameter) makes the developed model have more practical relevance.

5 CONCLUSIONS AND RECOMMENDATIONS

Convective drying of VIP sludge was investigated by varying the external drying parameters which were temperature, relative humidity and air velocity, as well as the size of the sample (changing the diameter of pellets). Faster drying was achieved by increasing air temperature and velocity, and also by decreasing air relative humidity and sample size. Under the explored conditions, the most influencing parameters on the drying kinetics of VIP sludge were the temperature, pellet diameter and air relative humidity. An increase of temperature from 40 to 80 °C reduced the drying time by 53%, from 190 to 90 minutes respectively. Increasing the relative humidity of the air from 5% to 25% resulted in lengthening the drying time from 135 to 220 minutes which corresponds to an increase of 63%. The air velocity had a relatively low effect on the drying time as increasing the velocity by fourfold resulted in less than 20 minutes decrease in the total drying time. However, it should be noted that the range of air velocity used in this study was low relative to various cases in literature. This may have resulted in the perceived observation of air velocity having a negligible influence on the total drying rate. In order to achieve high air speeds, the drying chamber of the rig could be reduced in volume.

The drying curve depicted two typical drying periods, the constant rate period and the falling rate period. Increasing the air temperature and velocity, whilst reducing the relative humidity resulted in an increase of the drying rate during the constant rate period. The critical moisture content was strongly affected by the drying temperature and relative humidity. High temperature and low humidity yielded prolonged constant rate period. This information is of consideration in the drying process as the constant rate period is easier to control through the operating conditions.

The effective diffusivity of faecal sludge was determined graphically through the analytical solution of Fick's second law of diffusion. The effective diffusivity of faecal sludge increased with the increase in temperature. The effective moisture diffusivities ranged between 7.8×10^{-8} and 2.1×10^{-7} m²/s in the temperature range of this study. However, the assumption of constant sample thickness during drying was made to determine the effective moisture diffusivity which was not the case according to visual observations. More accurate results of the effective moisture diffusivity could be evaluated by taking into account the eventual sample shrinkage.

The applicability of common empirical models that describe the moisture content of a sample as a function of time was investigated. The experimental data were fitted to the Newton's model, the Page model, the modified Page model, the two-term exponential model and the Logarithmic model. The Page model was found to most accurately predict the drying kinetics of sludge as suggested by the lowest *RMSE* value of not more than 0.017 and the highest *R*² value of at least 0.983. In order to take into account, the effect of drying air temperature and sample diameter on constants of the Page model, the

parameters of the model were correlated with respect to the temperature and pellet diameter. These had a strong influence on the k constant but non on the n value to which the average value was 1.36. The developed model predicted the experimental data well over the temperature and sample diameter range despite having lower R^2 values compared to the Page model. The validity of the model could be enhanced by increasing the temperature and pellet diameter.

The thermo-physical properties of the dried sludge studied in this project included the thermal conductivity, heat capacity and calorific value. The thermal conductivity of the fresh sludge (~80 %) was 0.55 W/m.K and exhibited a linear decrease as the sample was dried. The average conductivity of the final product was 0.04 W/m.K, a value similar to common insulators such as polyurethane, vermiculite and fibreglass which have thermal conductivities of 0.03, 0.07 and 0.035 W/m.K Heat capacity also showed a similar dependence with respect to the moisture content but without exhibiting a linear relationship. The heat capacity decreased from 3 866 to 211 J/kg.K. The average calorific value obtained was 12 MJ/kg, a value which is in the range with the calorific value of wood and coffee husks. This implies that faecal sludge can be a possible alternative source of energy.

The effect of the drying on the nutrient concentration of sludge was investigated in order to evaluate the potential reuse of the dried product in agriculture. The concentrations of K, P, Mg, Ca, PO_4^{3-} , NO_3^- and NO_2^- were analysed on a dry basis. The nutrient concentrations that were investigated did not show any significant variation between the raw and the dried samples except for ammonia, which decreased from 24 mg / g dry solid in the raw sludge to around 4.5 mg / g dry solid. The nutrient concentration of the macronutrients K, P, Mg and Ca, were generally higher than those of animal manure therefore dried faecal sludge can be used as fertiliser supplement.

Precaution should be taken in reusing faecal sludge as it contains harmful pathogens, *Ascaris* being the most resistant. The degree of pasteurisation of the dried sludge was not investigated due to the unavailability of *Ascaris* within the sample that was used. Future work should include the introduction of a known quantity of *Ascaris* eggs into the sample in order to evaluate the pasteurisation effect of convective drying.

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APPENDIX A STANDARD OPERATING PROCEDURES

A - a) Drying Rig

Drying is a mass transfer process consisting of the removal of a solvent by evaporation from a solid. A source of heat and an agent to remove the vapor produced by the process are often involved. In the most common case, a gas stream is used to apply the heat by convection and carries away the vapor. When a piece of wet material is exposed to hot air which is not saturated (its relative humidity is less than 100%), evaporation takes place from its surface. At a given temperature the rate of evaporation is dependent on the vapor pressure difference between the air close to the material and that of the more mobile air surrounding it. The drying rig allows control of drying air temperature, flow rate and moisture content.

Brief description of the rig

Dry air is humidified by counter current contact with water in the packed column. The humidity of the exit air can be varied by changing the water temperature and water flow rate entering the packed column. The air flow rate is determined using an orifice and the pressure difference across it is measured by a differential pressure transducer. The humidity of the air is determined by a humidity transducer. The humidified air is then heated to the desired temperature before it enters the drying chamber where the sample is to be dried. The drying pan is attached to a mass balance so that the mass of the sample may be monitored during the experiment. The mass balance is connected to a computer which will track the loss in mass of the sample with time.

Safety precautions

- Ensure that all electric cables are not in contact with water.
- Do not switch on the heater when there is no air flow.
- Ensure that there is enough water in the water bath and the heater is fully submerged.
- Ensure that the door of the drying chamber is completely closed during the experiment

Experimental procedure

Start up

1. Switch on the computer and open the appropriate software (Lab-view)
2. Open the exit valve for the packed column
3. Open the gas valve and set to the desired air flow rate. Allow the reading to stabilize
4. Switch on the water bath.
5. Set the water bath temperature and flow rate
6. When all set parameters are stable, place the sample in the drying chamber.
7. Start recording on the data logging software (Lab-view)

Shut down

1. Switch off the heaters
2. Switch off the water bath
3. Close the air supply

Microwave Plasma Atomic Emission Spectrometer

1. Scope and Application

Operation of the Agilent 4100 MP-AES involves the use of compressed air, high microwave energy and hazardous materials including corrosive fluids and flammable liquids. The plasma is extremely hot (about 6000 °C) and operates using high levels of microwave energy. The plasma emits high intensity light. The microwave excitation assembly is designed to reduce microwave radiation to safe levels while still allowing easy installation of the torch and viewing of the plasma.

The various indicator lights are color coded to represent the status of the instrument.

A green light indicates the instrument is in normal/standby mode

An orange light indicates that a potential hazard is present

A blue light indicates that operator intervention is required

A red light warns of danger or an emergency

The primary gas used is nitrogen, which is the supply gas for the plasma and nebulizer gas supply. Instrument grade quality required.

A small quantity of argon is used only in the plasma ignition cycle.

Oil free compressed air is used for the pre optics protection gas.

Pic page 24 and 25

Sample preparation

Weigh a well-mixed sample to the nearest 0.001g in the digestion vessels. For sludge, fecal samples use between 0.1g and 0.5g. For oil or oil contaminated samples use no more than 0.25g.

If the sample cannot be well mixed and homogenized, then oven dry at 60 °C or less and then grind the sample.

Add 12ml of Aquaregia (9ml Conc nitric acid + 3ml Conc hydrochloric acid) to the samples in the digestion vessels.

Dilute each sample to 50ml (then centrifuge) and test using the MP-AES.

The microwave program is set as follows:

04:00min @ 1000w @ 90°C

05:00min @ 1000 w @ 130°C

04:00min @ 1000 w @ 190°C

10:00min @ 1000 w @ 190°C

30:00min @ 1000 w @ 30°C

Total time 1:03:00

04:00min @ 1000w @ 90°C

60:00min @ 1000 w @ 130°C

10:00min @ 1000 w @ 40°C

30:00min @ 1000 w @ 30°C

To install the MP Expert Software

Log on to the instrument computer with Administrative rights. (Username-Admin/password-3000hanover).

Insert the MP Expert Software disk in your CD Rom drive. The software will automatically start.

Follow the instructions on the screen.

Click Yes to restart the computer if prompted.

Plug the USB cable into the USB port and then into a USB port on the computer.

Once installation is complete, you will need to register your software. To start the software, click the Windows START button, Then ALL PROGRAMS>Agilent>MP Expert.

Windows START button then choose Programs>Agilent>MP Expert>MP Expert Help.

Analysis Checklist

Turn on the MP-AES and software

Prepare for analysis

Calibrate the MP-AES

Create/open a worksheet

Develop a method

Run samples

Print a report

Turning on the MP-AES and Software

Check the exhaust and intake lines are secured to the MP-AES.

Ensure the gas lines are connected to the MP-AES and the gas supplies are turned on and set to the correct pressures (4-6 bars). Switch on the nitrogen gas generator.

Check that the USB and power cables are plugged on.

Switch on the compressor and the extractor system.

Instrument

Turn on the MP-AES.

Switch on the computer(Username-Admin/password-3000hanover)

To start the MP Expert software, click the Windows start button and then choose Programs>Agilent>MP Expert>MP Expert. The main index window will appear.

Click on instrument.

Red blinking zones on the instrument model on the pc stipulates errors. Check the error bar reading on the left. Purge the instrument to remove O₂.

Click-Start Purge(1-2mins to purge)

Blinking should stop after purging.

Torch

Check that the pre optics window is clean and correctly installed and that the interlocked is engaged.

Insert the torch with the outstanding lever facing you and completely close the torch handle. Do not touch the torch-this will create hotspots. Fit the spray chamber socket to the ball joint on the base of the torch and secure using the torch clamp. Monitor pic on pc-red zones will appear if it is inserted incorrectly.

Pump

Check that all tubing on the spray chamber, nebulizer and peristaltic pump are correctly connected.

See pic.

Inlet tubing should be placed in a beaker of 500ml de-ionised water.

Go to software-Click on –Run pump icon (the lower icon).

Click on fast run (run for 2 mins) and look for a flow.

Click on Pump Off.

Check icons on left are all green.

Plasma

Ensure that the Plasma Enable Switch is in the Enable state (pushed in).

Click-Plasma On(6000 degrees celsius)

Inspect flame-must taper uniformly. Must have only the following colors (orange on the outside-pink and the blue in the middle).

Switch off if it is not correct.

Leave the plasma on while continuing with the calibration.

Preparing for Analysis

Click the Plasma button in the MP Expert software, press F5 or choose Plasma on from the arrow under the plasma button.

Ensure that the peristaltic pump is correctly set up.

Place the sample tubing from the peristaltic pump into the rinse solution and the drain tubing into the drainage vessel.

Click the pump button in the MP-Expert software and choose Normal(15rpm) from the arrow under the pump button. The pump will be initialized and the solution will begin aspirating.

It will take approx 30 mins for the MP-AES to warm up.

The plasma will take 10-15 secs to ignite. If it fails refer to the troubleshooting guide.

The plasma cannot run without the spray chamber and the nebulizer gas supply connected. Doing so will damage the torch.

MP-AES Calibration

Ensure a standard glass concentric nebulizer, a single pass spray chamber and a standard plasma torch are installed. Use white/white peristaltic pump solution tubing and blue/blue tubing for the drain. The tubing tab color denotes the tubing size.

Place the solution inlet tubing into the wavelength calibration solution and allow the sample to reach the plasma

Click the instrument button

Click Optics Calibration

Click Calibrate Instrument. The torch will be aligned then a wavelength calibration and a calibration check will be performed automatically.

After a short while, an indication of the success or failure of the calibration check will appear, as well as an indication of the wavelength offsets.

If the calibration fails, check the sample introduction system. If the system seems fine, prepare a new wavelength calibration solution and try again.

Recommended values for the settings are given on the Conditions.

Test

Tick all tests except the last.

Click RUN TEST

Export to pdf and save.

Creating/Opening a Worksheet

Click NEW from the START page or the FILE menu.

A list of recently used files will appear otherwise you may BROWSE for more.

Opening an existing worksheet

- Click OPEN from the Start page or the File menu.
- A list of recently used files will appear otherwise you may BROWSE for more. The OPEN dialog box will be displayed in this instant.

Choosing rack type

Autosampler right click/Rack type/no rack

Go to bottom rack/right click/rack type/eg 11 samples.

Creating a new worksheet from a template

- Click NEW Form on the Start page or New Form Template from the file menu.
- A list of recently used files will be presented, otherwise you may BROWSE for more files. The New Form Template dialog box will be displayed in this instance.
- The Worksheet window will appear with the new worksheet loaded.

Turning off the Agilent 4100 MP-AES

- **Rinse the spray chamber by aspirating water for a few minutes. 1% nitric acid.**
- Extinguish the plasma by choosing 'PLASMA OFF' from the Plasma button drop down arrow or pressing SHIFT +F5 on the keyboard.
- Remove the solution tubing from the solution to prevent flooding of the spray chamber, Choose (normal-15 rpm) from the pump drop down button and pump any remaining liquid from the sample introduction system and inlet tubing. Turn the pump off when there is no more liquid flowing.

The green Plasma enable button, located in front of the MP-AES is intended for emergency only. If it is used you will have to reset the plasma enable button to 'on' position before the plasma can be re-ignited.

- To increase the pump tubing lifetime, loosen the pressure on the peristaltic pump tubing by releasing the pressure bars, and lifting the tubing out of the grooves in the tube retainer. PIC page 39
- Push up the pressure bar tensioners. This releases them from the pressure bars
- Allow the pressure bar to swing backwards
- Lift the tubing out of the grooves in the tube retainer.

Routine Maintenance

Daily:

1. Check and if necessary empty the drain vessel
2. Clean the surface of the equipment-clean all spills immediately
3. Inspect the pump tubing and replace if it has lost its elasticity,
4. Unclip the pump tubes when pump not in use.

Weekly

1. Inspect the torch for cleanliness. Clean as necessary.
2. Inspect the spray chamber for cleanliness. Clean as necessary. -soak nebulizer and spray chamber overnight in 400ml of Aquaregia which is used to remove organics.
3. 32% HCL 3:1 55% Nitric acid (300ml HCL:100ml Nitric acid)
4. Clean the nebulizer
5. Inspect the pre-optics window for cleanliness. Clean or replace as necessary

Monthly

Clean the air filter on top of your MP-AES

Perform an instrument calibration

Inspect the external gas supply system for leaks including the tubing connected to the MP-AES.

Replace any damaged leaking worn out components.

Standards Na, Mg, Ca, K. Making up 200ppm of each standard from 1000ppm.

- Take 50ml from concentrated 1000ppm individual standards and place in a 250 volumetric flask and top with distilled water to give you 200ppm.
- Combine all 4 individual standards (Na, Mg, Ca, K) to give you a stock solution of 1000ml(200ppm)
- Then make 5 different concentrations from the stock. See column 1 and 2. Concentration of the individual salts will be the concentration of the mixed standard divided by the number of salts (4).

Volume from stock(1000ppm) to dilute (mL)	Concentration of Mixed Standard (ppm)	Concentration of individual Standard (ppm)
50	200	50
200	160	40
100	80	20
50	40	10
25	20	5

Phosphate and total Phosphorus Analysis

(Cat. No. 1.14848); (Cat. No. 1.14543)

Scope and field of application

The measurement of total phosphorus and phosphate is essential for performance studies on the struvite reactor. The phosphate concentration in influent and effluent gives indication on the performance of the reactor operation whereas the total P values (influent and effluent) demonstrate the effectiveness of the filtration material used. The recovery can be calculated based on these measurements.

(Phosphate) Measuring range 0.02 – 11.46 mg/L P₂O₅

(Total Phosphate) Measuring range 0.11 – 11.46 mg/L P₂O₅

Principle

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

Interferences

Sample for phosphate analysis must be pre-treated 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

(Other interferences are mentioned in operational manual of test kits)

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

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

Phosphate Test

PO₄-1 – Sulphuric Acid (≥25% - <50%)

PO₄-2 – Non-Hazardous

Total Phosphate Test

P-1K – Sodium nitrate ($\geq 50\%$ - $\leq 100\%$)

P-2K – Sulphuric Acid ($\geq 10\%$ - $< 15\%$)

P-3K – Non-Hazardous

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 PO₄-P, the CombiCheck 10 also contains an addition solution for determining sample-dependent interferences (matrix effects).

Sample Preparation

Fecal samples are diluted by blending 1.8g -2g sample into 1L of distilled water, as described in detail below:

Weigh out 1.8g – 2g faecal sample using an analytical balance and add to a blender with 100mL distilled water and blend.

Add blended sample to a 1L volumetric flask and dilute to 1L using distilled water.

Swirl flask until sample is completely dissolved.

Filtration

Filter paper dimensions: diameter = 47mm, pore size = 0.45 microns

Filter the diluted solution using a Buchner funnel.

Collect the filtrate for analysis.

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 (diluted and filtered) sample into a test tube.

Reagent PO₄-1 5 drops Add and mix.

Reagent PO₄-2 1 level blue microspoon, add and shake vigorously until the reagent s 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 1 dose 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.

.....

Sample Analysis

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

Ortho-Phosphate measurement:

Pipette 8.0 mL distilled water into a test tube.

Add 0.5 mL pretreated sample with a micro-pipette and mix.

Add 0.5 mL Reagent PO4-1 with a micro-pipette and mix.

Add 1 dose Reagent PO4-2 and shake vigorously until the reagent is completely dissolved.

Leave to stand for 5min (reaction time), and then fill the sample into the cell (10-mm cuvette) 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 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 1 dose 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.

Procedure (Using Standard Solution - Reagent R-1)

Note: The error caused by the photometric measurement system and the mode of operation can be determined by means of the standard solution. This is used **without dilution** in place of the sample solution.

Basic Procedure: Proceed according to the instructions given in the package insert of the respective test kit and in the manual of the photometer used (as described in the total P measurement procedure using UD samples). In this case, however, use **undiluted reagent R-1** in place of the sample without adjusting the pH!

Detailed Procedure:

Total P measurement using a standard solution (reagent R-1):

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

Pipette 5.0 mL **undiluted reagent R-1** into a reaction cell.

Add 1 dose Reagent P-1K, close 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 1 dose 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 **standard sample** in the photometer.

Disposal of waste

Dilute 10 ml into 1000ml.

Slowly add NaCO₃ until ph 6-8 is reached.

Flush down the sink with excess water.

Calculations

$$\text{Wet Sample Concentration (g/g)} = \frac{A}{1000} \times \frac{V}{M}$$

$$\text{Dry Sample Concentration (g/g)} = \frac{\text{Wet Sample Conc. (g/g)}}{\text{Total Solids (g/g)}}$$

Where:

A – Spectroquant Reading Concentration

V – Volume of Dilution (L)

M – Mass of Sludge used in sample preparation (g)

Total Solids - 2540

Introduction

Solids refer to matter suspended or dissolved in water, wastewater and fecal sludge. Solids may affect water or effluent quality adversely in a number of ways. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

Total Solids is the term applied to material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids includes total suspended solids, the portion of solids retained by a filter and total dissolved solids, the portion that passes through the filter of 2.0µm or smaller. Fixed Solids, is the term applied to residue of total, suspended or dissolved solids after heating to dryness for a specified time at a specified temperature. The weight loss on ignition is called volatile solids.

Total Solids Dried at 103-105°C

1. Scope and Field of Application

Total Solids are determined in a wide variety of liquid and semi-liquid materials. These include portable waters, domestic and industrial waters, polluted waters and faecal sludge produced from treatment processes. It is of particular importance for the efficient operation of a treatment plant.

A known volume of well-mixed sample is evaporated to dryness in a porcelain crucible in a hot air oven at 105°C, the solids remaining are cooled and weighed. The residual material in the crucible is classified as total solids, and may consist of organic, inorganic, dissolved, suspended or volatile matter

2. Interferences

- Highly mineralized water with a significant concentration of calcium, magnesium, chloride and sulphate may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing.
- Exclude large, floating particles from the sample if it is determined that their inclusion is not desired in the final result.
- Disperse visible floating oil and grease with a blender before withdrawing sample portion for analysis because excessive residue in the dish may form a water-trapping crust.

3. Sampling

- Mix the sample well to suspend solids uniformly.
- Remove the test portion rapidly before any settling of solid matter occurs.
- Use a measuring cylinder and not a pipette for sludge and wastewater samples.
- Use a crucible for feces.
- Use a volume or mass of sample to ensure a measurable residue- limit sample to no more than 200mg residue
- Suitable aliquots: Liquid samples – 100ml, Sludge -30ml, Faeces 10-20g

4. Safety Precautions

- . Always use safety goggles, gloves and laboratory coat while working in laboratory
- Wear gloves suitable for withstanding high temperatures when removing crucibles from the oven.
- 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.

5. Apparatus

- 50ml capacity evaporating porcelain crucibles
- Desiccator
- Drying oven
- Analytical Balance

6. Reagents

- None

7. Calibration

- Check the temperature throughout the oven area by placing a calibrated thermometer on each shelf, after 30mins, check temperature at each level against oven setting.
- Adjust oven setting if necessary.
- If temperatures are uneven on the shelves, check insulation.

8. Procedure

Prepare Crucible

- If volatile solids are to be measured ignite clean crucible at 550°C for 1hr in the furnace. If only total solids are to be measured, heat clean crucible to 103-105°C for 1h. Store and cool dish in a desiccator until needed. Weigh immediately before use..... W_1g

Sample Analysis

- Measure out appropriate volume (30ml) /minimum mass (10-20g) that will yield a residue between 2.5 and 200mg of a mixed sample using correct volume measuring cylinder or analytical balance.... V_{ml} ... W_g . Transfer quantitatively to the weighed crucible, rinsing the cylinder with small volumes of distilled water to dislodge heavy particles. Add washings to the crucible.
- Place in hot oven at 103-105°C for 24hrs.
- *Dry sample for at least 1hr in an oven 103-105°C, to desiccator to balance temperature and weigh. Repeat cycle of drying, cooling and weighing until a constant weight is obtained, or until weight change is less than 4% of previous weight or 0.5mg, whichever is less.*

Remove the next day and cool for 15 minutes and weigh..... W_2g

9. Calculation

$$\text{Total Solids in Sample (mg/l)} = \frac{(W_2 - W_1)g \times 100\,000}{V_{\text{sample}} (ml)}$$

$$\text{Total Solids in Wet Sample (g/g)} = \frac{(W_2 - W_1)g}{W_{\text{sample}} (g)}$$

$$\text{Moisture Content (g)} = W_{\text{sample}}(g) - [(W_2 - W_1)]g$$

Total Suspended Solids Dried at 103-105°C

1. Scope and Field of Application

Suspended solids are useful determinants in the analysis of polluted, re-use and waste waters. It is used to evaluate the strength of domestic/industrial waste waters and to determine the efficiency of treatment units, such as settling tanks, biological filters, and the activated sludge. Use of glass fiber filter pads is preferred to crucibles because of the saving in filtration time and the only prior preparation necessary is drying in an oven for 30mins at 105°C.

A measured volume of well shaken is vacuum filtered through a dried pre-weighed 110mm diameter glass fiber filter. The filters and residue is dried to a constant weight at 103-105°C. The increase in weight of the filter represents the total suspended solids.

1. Interferences

- Exclude isolated large floating particles.
- Samples high in dissolved solids must be washed adequately.
- Loss in mass of the rinsed glass fiber filters must be taken into the final calculation.
- The larger the sample, the smaller the factor applied in the calculation, but avoid prolonged filtrations.

3. Sampling

- Take the sample at a point of turbulence to ensure that it is truly representative.
- Mix sample thoroughly and remove test portion rapidly before segregation occurs.
- Use appropriate volume measuring cylinder and not pipettes.

4. Safety Precautions

- Exercise care when using glassware, vacuum pumps and ovens.
- Good housekeeping and cleanliness are essential for obtaining accurate results.

5. Apparatus

- Four- place Analytical balance
- 110mm diameter funnel and flask
- Vacuum pump

6. Reagents

- Nil

7. Calibration

- The analytical balance and ovens are checked and serviced weekly.

8. Sample Preparation – Faecal Sludge

- Weigh out between 1.8g and 2g of well mixed faecal sludge sample.
- Place the weighed out sample into a blender with 250ml of distilled water.
- Blend for 30 seconds.
- Transfer the blended mixture into a volumetric flask and top up to 1L with distilled water.
- Transfer the 1L solution to a plastic bottle and store in the cold room.

9. Procedure

Dry Filter Paper

- Use 110mm glass fiber filter paper Whatman No 4(20-25um) for sludge.
- Use 20ml sample on a 40mm, 0.45um glass fiber filter for waste water and urine.(change to a 1um pore size if the dried residue is more than 200mg.
- Use a smaller pore size if the dried residue is lower than 2.5mg.
- Mark the filter paper with a pen
- Place papers on the stainless steel mess of appropriate size
- Position on top shelf in oven at 105°C for 30min .
- If volatile solids are to be measured ignite at 550 °C for 15min in a furnace.
- Transfer to desiccator
- Cool for 20 minutes before weighing

Weigh Filter Paper

- Transfer filter paper rapidly to balance
- Note mass (**W1**) grams, to fourth decimal place

Prepare for Analysis

- Place filter paper into a 110mm diameter funnel, with the marking on the lower side
- Measure out appropriate volume to yield between 2.5 and 200mg dried residue of well mixed sample
- Place funnel into flask with side arm attached to a vacuum pump.
- Apply pump
- Wet paper with distilled water to seal edges of the paper to surface of the funnel
- Pour sample onto the filter paper, keeping sample in the middle of the paper.
- When filtration is complete. Remove paper by placing the end of a small thin spatula along the edge of the filter paper and lift slowly.
- Remove the paper with a pair of tweezers, taking care not to tear the paper.
- Fold paper twice to form a triangle enclosing sample residue. This seals the residue in the filter paper and protects it from contact with air.

Dry and Weigh

- Place triangles on a stainless steel mess
- Place in oven at 105°C for 2hrs
- Remove from oven and place in desiccator
- Cool to room temperature
- Weigh after 20 mins, as rapidly as possible
- Note mass (**W2**) grams

11. Calculation

$$\text{Total Suspended Solids (g/ml)} = \frac{(W_2 - W_1)}{V_{\text{sample}}(\text{ml})}$$

$$\text{Total Suspended Solids in Wet Sample (g/g)} = \text{TSS (g/ml)} \times \text{DF}$$

$$\text{Total Suspended Solids in Dry Sample (g/g)} = \frac{\text{TSS}_{\text{wet sample}}}{\text{Total Solids (g/g)}}$$

W_1 = weight of filter paper before oven (105°C) (g)

W_2 = weight of residue + filter paper after oven(105°C) (g)

DF = Dilution Factor

Fixed and Volatile Solids Ignited at 550°C

1. Principle

The residue from the above methods is ignited to constant weight at 550°C. The remaining solids represent the fixed total, dissolved or suspended solids while the weight lost on ignition is the volatile solids. The determination is useful in control of wastewater treatment plant operation because it offers a rough estimate of the amount of organic matter present in the solid fraction of wastewater, activated sludge and industrial wastes.

2. Interferences

- Negative errors in the volatile solids may be produced by loss of volatile matter during the drying.

2. Apparatus

- Muffle Furnace
- As above

4. Procedure

- Ignite residue from the total solids to constant weight in a muffle furnace at a temperature of 550°C.
- Have furnace up to temperature before inserting sample.
- Usually 2 hr for VIP and sludge samples, 15-20min for waste water (200mg residue)
- Let the crucible cool partially in air until most of the heat has dissipated
- Transfer to a desiccator for final cooling. Do not overload the desiccator
- Weigh dish as soon as it has cooled to balance temperature.

5. Calculation

$$\text{Volatile Solids in Wet Sample (g/g)} = \frac{(B - C)}{W_{\text{sample}}(g)}$$

$$\text{Volatile Solids in Dry Sample (g/g)} = \frac{VS_{\text{wet sample}}}{\text{Total Solids(g/g)}}$$

$$\text{Fixed Solids in Wet Sample(g/g)} = \frac{(C - D)}{W_{\text{sample}}(g)}$$

$$\text{Fixed Solids in Dry Sample(g/g)} = \frac{FS_{\text{wet sample}}}{\text{Total Solids(g/g)}}$$

B = weight of residue + crucible before ignition - 550°C (g)

C = weight of residue + crucible after ignition -550°C (g)

D = weight of crucible (g)

APPENDIX B MATLAB CODE USED TO SMOOTHEN RAW DATA

The experimental curves produced had a noise which would make it difficult to generate rate curves. A Matlab code was generated to eliminate the noise within the data. The function of the code was to generate a smoother curve by using a cubic spline fit over a number adjacent data points

```
clear
clc

stepRatio = 0.08           % Ratio "Step length / Total length", it has to be < 1
                            % Adjustment parameter [0.01 - 0.1]
data = load(['manip1']);    % manip should contain time and mass in that order(column vector)
data=data.(['manip1']);
t=data(:,1);
M=data(:,2);
degree=1;                  % degree of fitting polynomial
n=int16((length(t)-1)*stepRatio); % integral number of values for each step
Mp=[];%empty vector before iterations
tp=[];%empty vector before iterations
    for i=n+1:length(t)-(n+1)
        ts=t(i-n:i+n);
        Ms=M(i-n:i+n); %
        [a,S,MU]=polyfit(ts,Ms,degree); % fitting polynomial of degree
        tp(i)=ts(n+1);%definitive vector values
        Mp(i)=polyval(a,tp(i),S,MU);%definitive vector values
    end
    % Smoothing first section of curve
    ts=t(1:n);
    Ms=M(1:n);
    [a,S,MU]=polyfit(ts,Ms,degree);
    for i=1:n % Iterations to build the first points from tp and Mp vectors
        tp(i)=ts(i);
        Mp(i)=polyval(a,tp(i),S,MU);
    end
```

```

%figure(1)
% xlabel('Time (min)')
% ylabel('Mass (g)')
% plot(t,X,'-b',ts,Xs,'*r',ts,polyval(a,ts,S,MU),'-g');
% axis([ts(1) ts(n) min(Xs) max(Xs)]);
finaltable = [tp;Mp]'
subplot(1,2,1)
plot(tp,Mp)
title('change in mass')
xlabel('Time(min)')
ylabel('mass (g)')

% plotting of instantaneous rate curve
ii = 1: length(Mp)-1;
for in = ii
    rate(in) = (Mp(in)-Mp(in+1))/5 ; % change in mass per unit time
end

subplot(1,2,2)
plot(tp([ii]),rate,'r+')
title('Rate plot')
xlabel('Time(min)')
ylabel('rate ')

```

APPENDIX C COMPARISON OF RESULTS

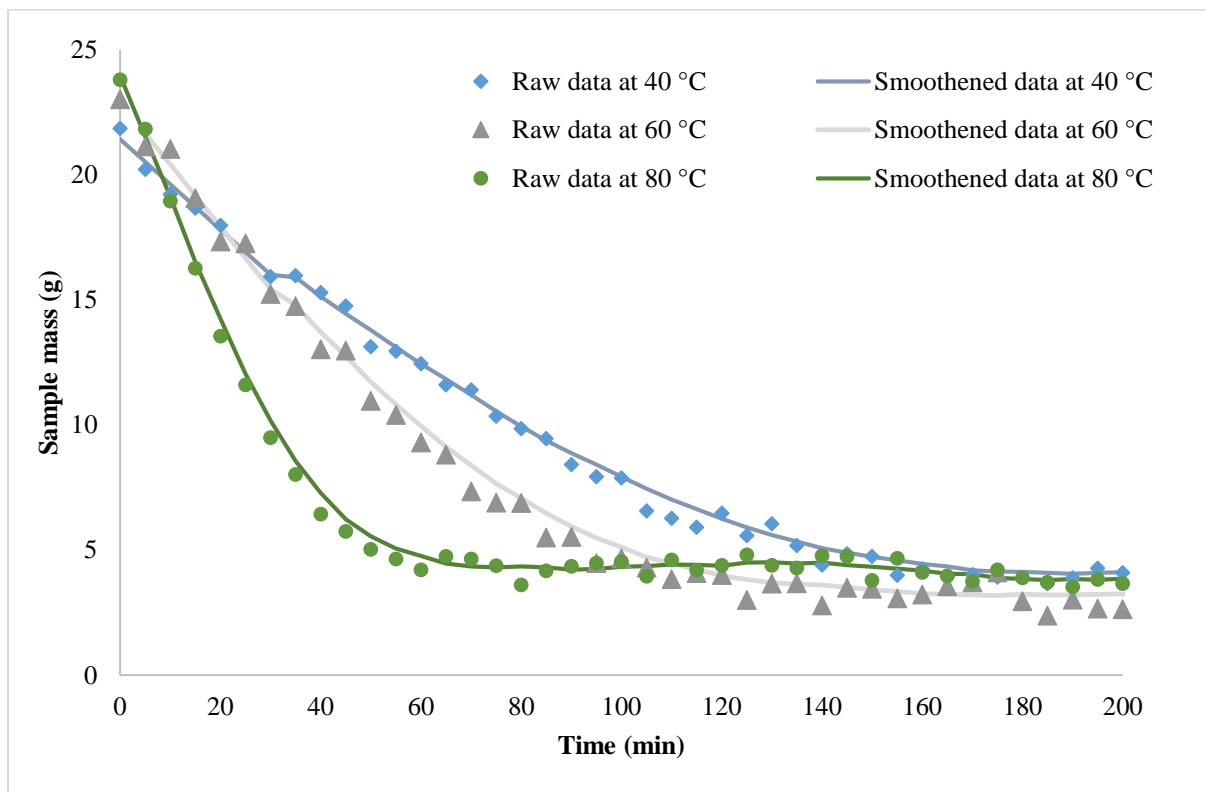


Figure C. 1: Comparison between raw data and smoothed data of pellets at varying temperatures whilst at constant relative humidity, pellet diameter and air velocity

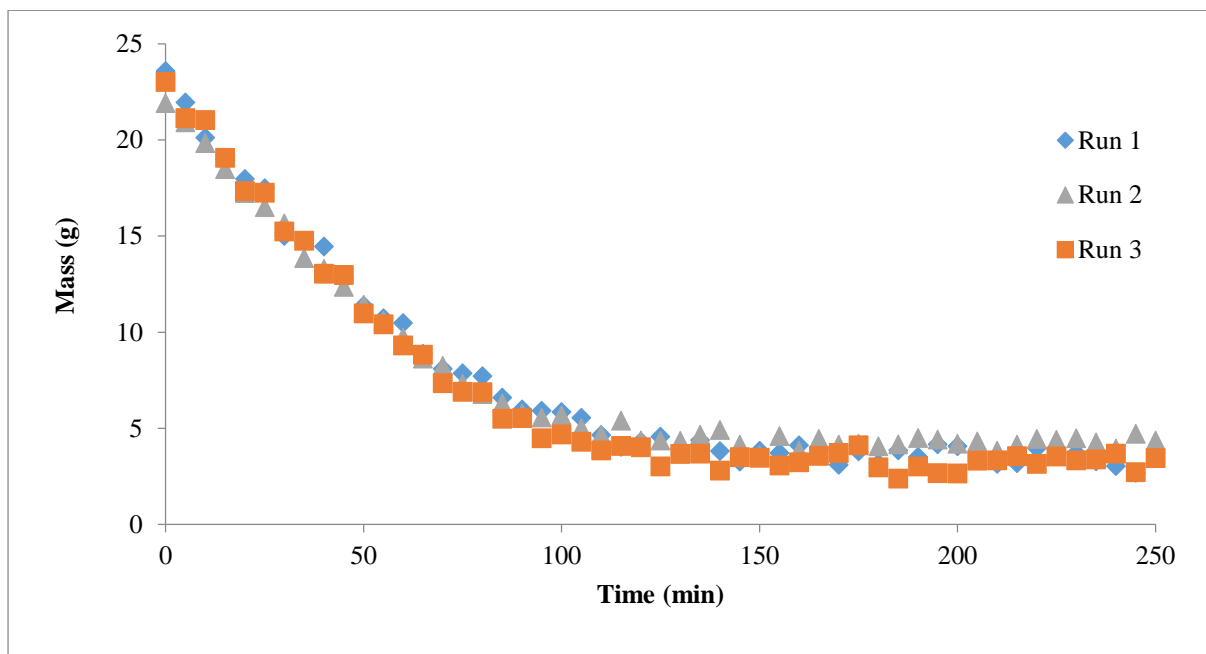


Figure C. 2: Three drying experimental results at the same conditions of 60°C, 5% relative humidity, pellet diameter of 8 mm to evaluate the reproducibility of the raw data

APPENDIX D Formulation of an empirical drying model

Analysis of the coefficients found for the Page model was done as it was the model that described the experimental data well of all the models investigated. The temperature variation of the k constant for the flat slab and cylindrical configuration was evaluated and represented in the figure below. The variation of the k constant was regressed to a power law of the form

$$k = AT^b$$

Where A and b are constants, T is the temperature (°C)

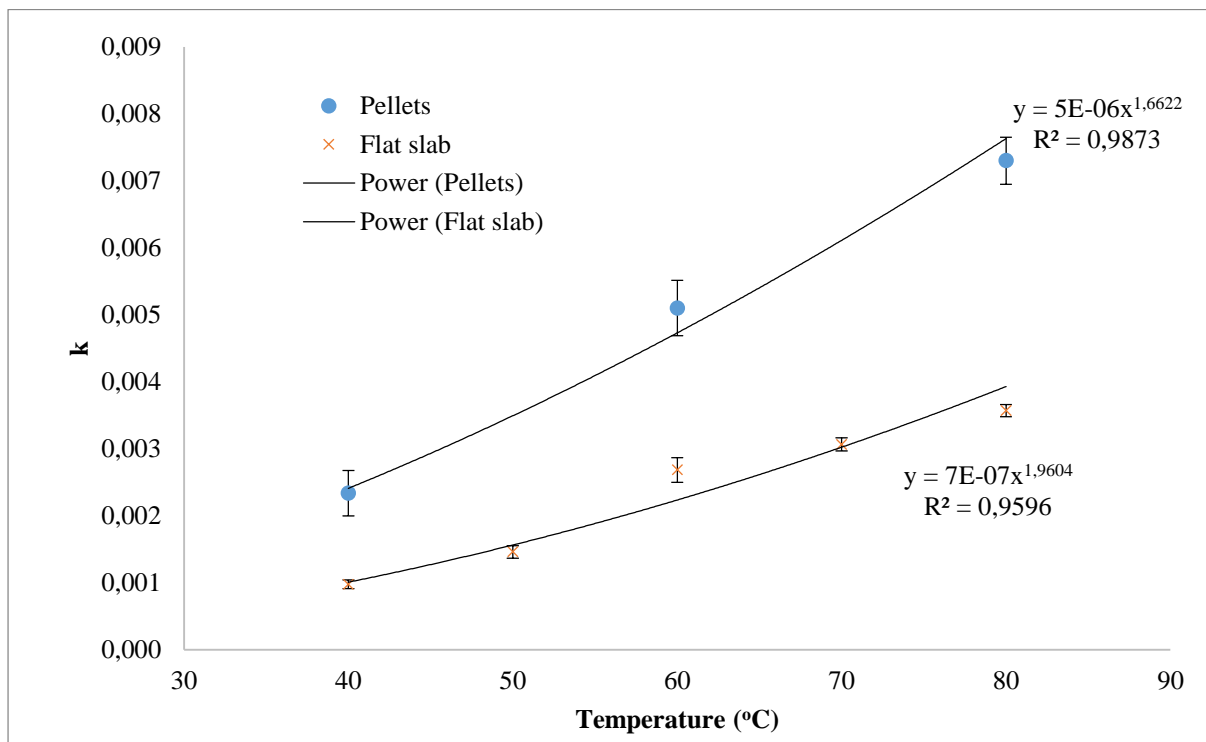


Figure D. 1 variation of the constant, k , of the Page model with temperature of both the flat slab and cylindrical configuration

It was assumed that the constant A was a function of the sample thickness, of which results from changing pellet diameters were used. The variation of the A constant with the pellet diameter was assumed to follow a power relationship of the form

$$A = Cd^e$$

Where C and e are constants, d is the pellet diameter (mm)

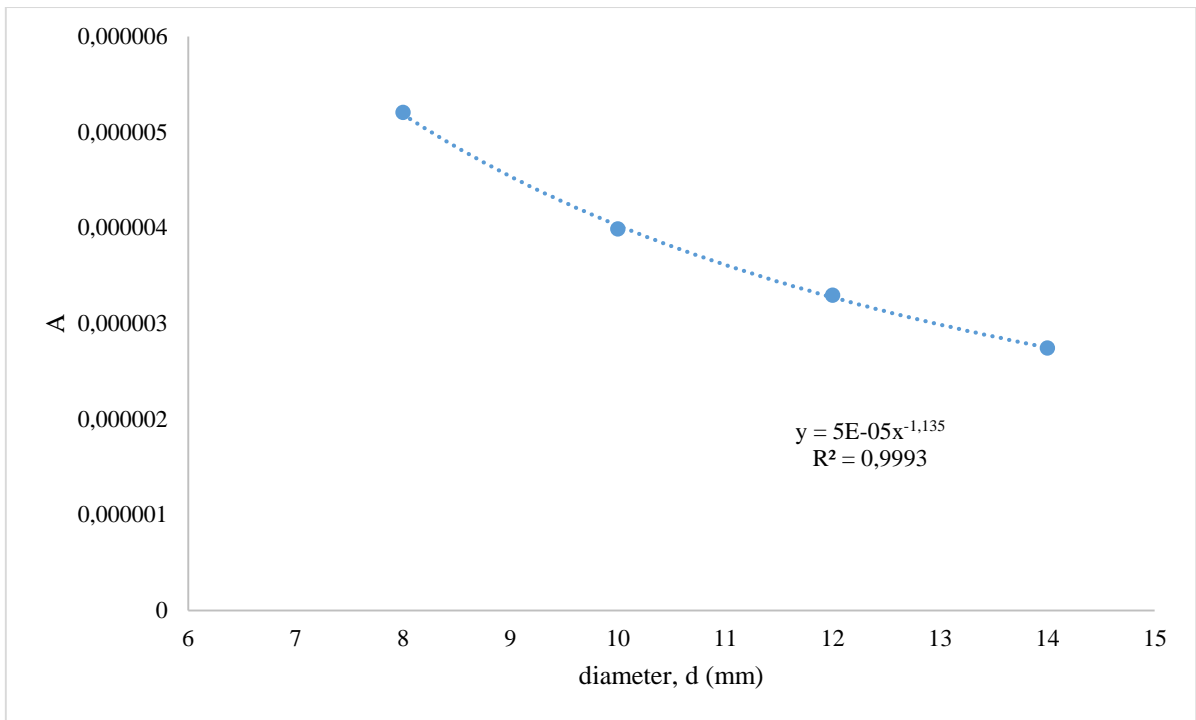


Figure D. 2 variation of the A constant with the pellet diameter obtained by regressing the k , constant of the Page model with temperature of cylindrical configuration

APPENDIX E Drying data

Table E- 1 Drying data used to evaluate the dry rate at a temperature of 40°C, relative humidity of 5%, velocity of 0.6cm/s and pellet diameter of 8 mm

Final mass (g)	3,20		Surface area (m ²)	0,002262
Time (min)	Mass (g)	Rate (g/min)	Rate/ SA	Moisture Ratio
0	22,89	0,2486	27,473	1
5	21,65	0,2486	27,473	0,936
10	20,40	0,2486	27,473	0,873
15	19,16	0,2486	27,473	0,810
20	17,92	0,2486	27,473	0,747
25	16,68	0,2486	27,473	0,684
30	15,43	0,2309	25,519	0,621
35	14,78	0,2149	23,755	0,588
40	13,71	0,1901	21,014	0,533
45	12,76	0,2072	22,901	0,485
50	11,72	0,1805	19,953	0,432
55	10,82	0,1715	18,951	0,386
60	9,96	0,1679	18,553	0,343
65	9,12	0,1459	16,122	0,300
70	8,39	0,1456	16,092	0,263
75	7,67	0,1193	13,189	0,226
80	7,07	0,1195	13,204	0,193
85	6,47	0,1061	11,730	0,165
90	5,94	0,0899	9,932	0,138
95	5,49	0,0751	8,297	0,116
100	5,12	0,0804	8,886	0,097
105	4,71	0,0515	5,688	0,076
110	4,46	0,0459	5,069	0,063
115	4,23	0,0508	5,615	0,051
120	3,97	0,0303	3,345	0,039
125	3,82	0,0263	2,903	0,031
130	3,69	0,0103	1,135	0,024
135	3,64	0,0075	0,825	0,022
140	3,60	0,0073	0,801	0,020
145	3,51	0,0069	0,762	0,015
150	3,42	0,0062	0,691	0,010
155	3,35	0,0054	0,597	0,007
160	3,26	0,0049	0,545	0,002
165	3,23	0,0047	0,516	0,001
170	3,21	0,0045	0,501	0,000
175	3,18	0,0042	0,465	-0,001
180	3,23	0,0047	0,516	0,001

Table E- 2 Drying data used to evaluate the dry rate at a temperature of 60°C, relative humidity of 5%, velocity of 0.6cm/s and pellet diameter of 8 mm

Final mass (g)	4,13		Surface Area (m ²)	0,002262
Time (min)	Mass (g)	Rate (g/min)	Rate/ SA	Moisture Ratio
0	21,42	0,1804	19,9338	1,000
5	20,52	0,1804	19,9338	0,948
10	19,61	0,1804	19,9338	0,896
15	18,71	0,1804	19,9338	0,844
20	17,81	0,1804	19,9338	0,791
25	16,91	0,1804	19,9338	0,739
30	16,01	0,1629	18,0039	0,687
35	15,90	0,1533	16,9471	0,681
40	15,14	0,1487	16,4339	0,637
45	14,45	0,1426	15,7563	0,597
50	13,80	0,1367	15,1050	0,559
55	13,11	0,1339	14,7955	0,520
60	12,44	0,1229	13,5871	0,481
65	11,83	0,1248	13,7934	0,445
70	11,21	0,1292	14,2797	0,409
75	10,56	0,1251	13,8229	0,372
80	9,93	0,1104	12,2019	0,336
85	9,38	0,1007	11,1261	0,304
90	8,88	0,0996	11,0049	0,275
95	8,42	0,0971	10,7282	0,248
100	7,93	0,0960	10,6103	0,220
105	7,45	0,0873	9,6525	0,192
110	7,02	0,0748	8,2672	0,167
115	6,64	0,0781	8,6356	0,146
120	6,25	0,0695	7,6778	0,123
125	5,91	0,0597	6,6020	0,103
130	5,61	0,0523	5,7767	0,086
135	5,35	0,0529	5,8504	0,070
140	5,08	0,0355	3,9199	0,055
145	4,90	0,0348	3,8462	0,045
150	4,73	0,0269	2,9768	0,035
155	4,59	0,0293	3,2420	0,027
160	4,45	0,0197	2,1810	0,019
165	4,35	0,0305	3,3747	0,013
170	4,20	0,0103	1,1347	0,004
175	4,15	0,0037	0,4126	0,001
180	4,13	0,0077	0,8547	0,000
185	4,09	0,0071	0,7810	-0,002

Table E- 3 Drying data used to evaluate the dry rate at a temperature of 80°C, relative humidity of 5%, velocity of 0.6cm/s and pellet diameter of 8 mm

Final mass (g)	3,89		Surface area (m ²)	0,002262
Time (min)	Mass (g)	Rate (g/min)	Rate/ SA	Moisture Ratio
0	23,15	0,2952	32,6268	1,000
5	21,68	0,2952	32,6268	0,923
10	20,20	0,2952	32,6268	0,847
15	18,73	0,2952	32,6268	0,770
20	16,53	0,2952	32,6268	0,656
25	14,94	0,2863	31,6431	0,573
30	13,36	0,2961	32,7262	0,492
35	11,78	0,2882	31,8555	0,410
40	10,34	0,2596	28,6872	0,335
45	9,05	0,2356	26,0346	0,268
50	7,87	0,1856	20,5084	0,206
55	6,94	0,1531	16,9225	0,158
60	6,17	0,1127	12,4524	0,119
65	5,61	0,0978	10,8068	0,089
70	5,12	0,0578	6,3858	0,064
75	4,83	0,0440	4,8631	0,049
80	4,61	0,0253	2,7999	0,038
85	4,49	0,0200	2,2105	0,031
90	4,39	0,0153	1,6947	0,026
95	4,31	0,0218	2,4070	0,022
100	4,20	0,0089	0,9824	0,016
105	4,16	0,0258	2,8491	0,014
110	4,03	0,0093	1,0316	0,007
115	3,98	0,0231	2,5543	0,005
120	3,87	0,0089	0,9824	-0,001

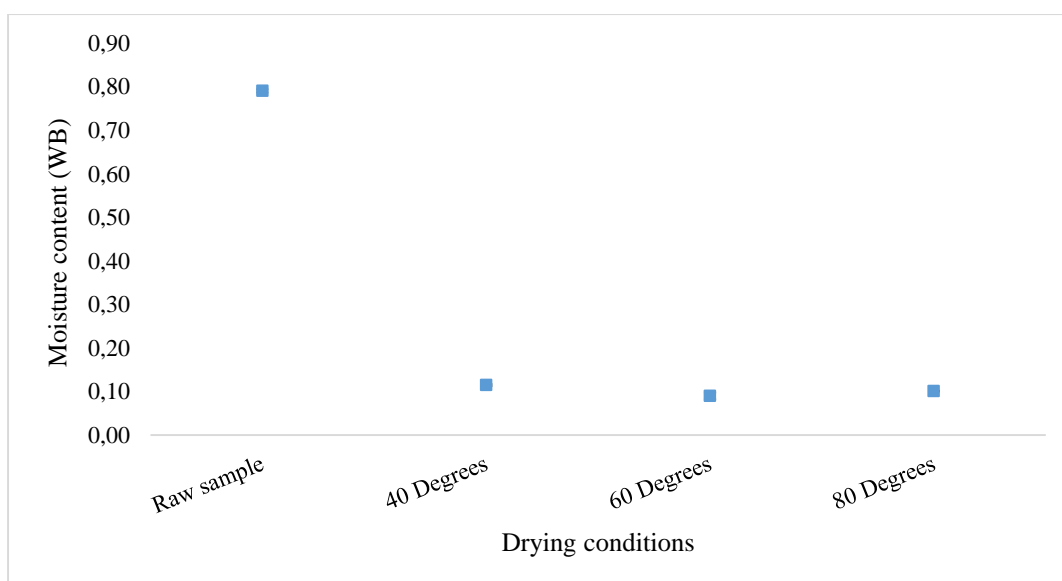


Figure E- 1 Evaluation of the moisture content of the raw sludge and the dried pellets

Table E- 4: Calorific value measurements from the Bomb Calorimeter

Sample ID	mass (g)	Gross heat (MJ/Kg)	EE value	average heat	standard deviation
40T-0.1F-5RH	0,508	12,45	234		
40T-0.1F-5RH	0,507	12,22	2345	12,33	0,117
40T-0.1F-5RH	0,5047	12,32	2345		
60T-0.1F-5RH	0,508	12,33	2345		
60T-0.1F-5RH	0,5091	12,70	2345	12,49	0,192
60T-0.1F-5RH	0,5078	12,44	2345		
80T-0.1F-5RH	0,5053	12,87	2345		
80T-0.1F-5RH	0,5063	12,39	2345	12,67	0,2445
80T-0.1F-5RH	0,5068	12,73	2345		
60T-0.1F-15RH	0,5077	12,15	2345		
60T-0.1F-15RH	0,5059	12,15	2345	12,15	0,0043
60T-0.1F-15RH	0,5014	12,14	2345		
60T-0.1F-25RH	0,5024	11,64	2345		
60T-0.1F-25RH	0,5044	11,85	2345	11,74	0,103
60T-0.1F-25RH	0,5048	11,72	2345		