

Solar Thermal Dewatering in Faecal Sludge Management

Elinor Jane Winrow

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Declaration of Interests

DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.



Date 27/08/2021

STATEMENT 1

This thesis is the result of my own investigations, except where otherwise stated.

Other sources are acknowledged by footnotes giving explicit references. A bibliography is appended.



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Abstract

This research project focused on understanding the way in which faecal sludge dries on a material level. Faecal sludge pose's huge health and environmental problems within the developing world, but by implementing faecal sludge management systems these issues can be reduced. This research is of great importance as understanding the way in which faecal sludge dries can improve the efficiency and effectiveness of new and developed drying techniques. In turn this will mitigate many of the health and environmental issues facing the developing world.

The aim of this project was to investigate how faecal sludge dries under differing conditions to help predict how efficiently newly developed drying technologies will work.

Faecal sludge from ventilated pit latrines (VIP), urine diversion toilets (UDDT) and anaerobic baffled reactors (ABR) along with fresh faeces (HF) were analysed using both UV-Vis-NIR and STA-FTIR analysis to identify their drying properties.

UV-Vis-NIR spectroscopy was used to analyse the reflectance and transmission properties of faecal sludge, identifying how it interacts with the radiation produced from the whole solar spectrum. This allowed the depth of penetration to be identified along with the absorbance potential of each type of faecal sludge. When analysing the whole solar spectrum VIP, UDDT, ABR and HF had a total absorbance 87%, 86%, 85% and 65% respectively, indicating that solar thermal drying was an excellent drying process.

To understand the drying trends and energy demand needed to remove moisture from faecal sludge, STA-FTIR was carried out. This demonstrated the variability found within the moisture retention properties of faecal sludge, along with the importance of fully understanding the drying system used to ensure that no energy is wasted within the process. pH and nutrient content were found to play an important role within a faecal sludge ability to dry, meaning that UDDT sludge reached the lowest final moisture content of ~23% due to the removal of nutrients within the urine diversion process, while VIP sludge dried to the lowest moisture content (~8%) due to it having a neutral pH.

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Nomenclature

MDG - Millennium Development Goal

SDG – Sustainable Development Goal

FSM – Faecal Sludge Management

VIP – Ventilated Improved Pit Latrine

UV – Ultra Violet

NIR - Near InfraRed

IR – InfraRed

MIR – Mid InfraRed

COD – Chemical Oxygen Demand

UDDT – Urine Diversion Toilet

ABR - Anaerobic Baffled Reactor

HF - Human Faeces

LaDePa – Latrine Dehydration Pasteurisation

PSS – Particle System Separation

UV-Vis-NIR – Ultra Violet – Visible – Near InfraRed Spectroscopy

STA-FTIR - Simultaneous Thermal Analysis coupled with Fourier Transform Infrared

Spectroscopy

STA – Simultaneous Thermal Analysis

FTIR - Fourier Transform InfraRed Spectroscopy

TGA – Thermal Gravimetric Analysis

DSC - Differential Scanning Calorimetry

SOP – Standard Operating Procedure

Chapter 1 Literature Review

1.1 Faecal Sludge Management

In 1923 Gandhi said, "sanitation is more important than independence" and to this day it still rings true. Adequate sanitation, together with good hygiene and safe water are fundamental to good health, along with social and economic development (1). Despite bold efforts being made by a range of actors to improve sanitation, a significant majority of urban households in developing countries have yet to gain access to decent, dignified, healthy and affordable sanitation choices. Slums all over the world are hidden from public view. These are located far from highways, on slopes, dumps, wetlands and in the shadow of the sun. Within Kenya alone, around 55% of the urban population dwell in slums which amounts to around 6 million people (2). Unfortunately, many people within these communities are either being forgotten about, or the sanitation options being developed are not appropriate for their needs and lifestyles.

In 2000, the international community adopted the Millennium Development Goals. The seventh Millennium Development Goal (MDG) aims to ensure environmental stability, with the 10th MDG aiming to halve the proportion of the population that lacks sustainable access to safe drinking water and basic sanitation (3). These goals were then updated in 2015 to create the 2030 Agenda for Sustainable Development (SDGs), with Goal 6 ensuring available and sustainable management of water and satiation for all (4). While access has increased substantially due to funding from external aid, there is still a lot of work needed to achieve this target when considering safe access to water and sanitation resources in Sub-Saharan Africa and the world at large.

Around 2.7 billion people worldwide currently lack access to adequate sanitation contributing to around 10% of global diseases. By creating improvements within this area, we can reduce the morbidity and severity of the various diseases and improve the quality of life within developing communities (1). Faecal Sludge Management (FSM) systems are being put in place to help combat these sanitation problems facing the developing world.

Faecal Sludge Management (FSM) was created as there were no management systems to deal with the faecal sludge produced from onsite sanitation systems.

The result was that waste typically ended up dumped into the environment, causing significant health and environmental implications. Creating an FSM infrastructure and public services that work for everyone, whilst keeping faecal sludge out of the environment is a huge challenge.

Implementing a similar treatment system to the ones found within the developed world is unfortunately not an option due to the flush toilet being a cost, resource, and energy intensive system. It requires the use of at least 3 litres of water per flush which can quickly increase to up to 20 litres depending on the toilets design and user's behavioural pattern. It also requires sewer connections for transportation, treatment and disposal of excreta, wastewater, and grey water leading to an average of 285 L of sewage sludge being produced per person per day in the UK (5). Comparing this to the average daily water allowance that is commonly found within the developing world of approximately 25 – 30 litres a day in total it is easy to see why complex sanitation processes are not a viable option within these communities. These processes also present huge energy requirements leading to environmental burdens for both urban and rural communities (6).

In order to achieve a complete sanitation chain which is also known as the sanitation value chain (Figure 1.1), human excreta management systems should follow the management pathway listed below (7):

- a) Collection of excreta
- b) Transportation of excreta to a suitable location
- c) Storage and/or treatment of excreta
- d) Reusing and/or returning excreta to the environment

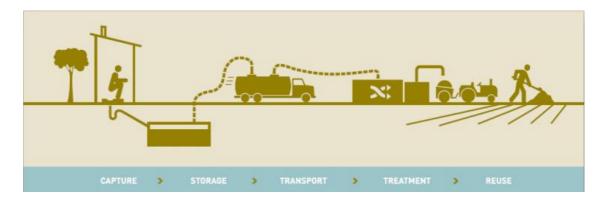


Figure 1.1: Sanitation Value Chain (8)

The sanitation value chain clearly breaks down the full waste system into 5 simple steps. The first stage of the sanitation value chain is capture, where how and where people defecate is investigated. New toilet systems are developed through schemes such as the Reinvent the Toilet Challenge in 2011 and 2014 (9). Alongside capture, sanitation practitioners also consider the second stage of the sanitation value chain. Understanding how the faecal sludge is going to be stored is as important as how it is going to be captured as without the faecal sludge being easily accessible to empty once the toilet system is full it will not be used again. The third stage of the chain is transport. This is where sanitation practitioners put in place affordable methods for households to have their toilets emptied and the waste transported to a treatment site. The last two steps of the sanitation value chain - treatment and reuse - are often classed as one step. Creating treatment systems is a vital part of the sanitation value chain as it allows the faecal sludge to be turned from a environmentally hazardous waste material into a useful end product that can either be safely disposed of within the local environment or re-used within the local community.

It is this chain that is now being followed to develop new and improved onsite sanitation options for developing communities. It is a common misperception that onsite decentralised technologies only fulfil sanitation needs for rural areas, as there are around one billion onsite facilities worldwide in urban areas. In many cities, onsite technologies have much wider coverage than sewer systems.

An example of this would be to look at Sub-Saharan Africa where 65-100% of sanitation access in urban areas is provided through onsite technologies. Even where sanitation needs are met through onsite technologies, for a vast number of people in urban areas of low- and middle-income countries, there is typically no management system in place for the resulting accumulation of faecal sludge (Figure 1.2).

~2.7 billion people worldwide are served by sanitation methods that need fecal sludge management

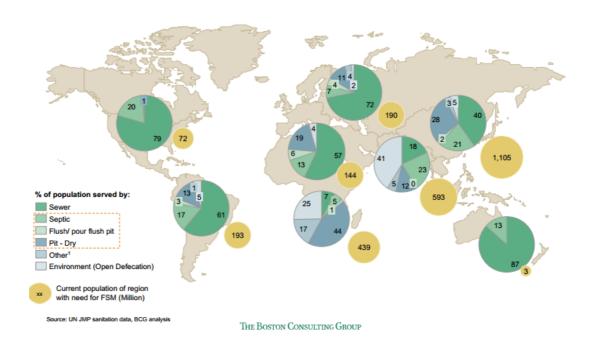


Figure 1.2: Percent of population served by onsite sanitation technologies as of 2013 (10)

Increasing access to sanitation is a global priority. Currently one in five children die from diarrheal related diseases, which is more than AIDS, malaria, and tuberculosis combined (11). Progress towards the SDGs have been successful in increasing access to improved sanitation facilities, however providing adequate access to

sanitation facilities does not end when onsite technologies are built. The promotion of onsite technologies has greatly reduced open defecation, but without solutions or funding to maintain their functionality through appropriate FSM, this can result in a sludge management crisis, leading to significant impacts on human and environmental health. Improved FSM are defined as systems that hygienically separate human excreta from human contact, includes flush toilets, connection to a piped sewer system, connection to a septic system, flush/pour-flush to a pit latrine, ventilated improved pit (VIP) latrines, and composting toilets. Onsite technologies can represent viable and more affordable options, but only if the entire service chain including collection, transport, treatment and safe end use or disposal is managed adequately. Unfortunately, this is where the sanitation value chain often breaks down and without an FSM structure in place, when the containment structure fills up, the untreated faecal sludge most likely ends up directly in the local environment. This will result in the pervasive contamination by pathogens, hence not providing a protective barrier to human contact and removing the protection of public health (10).

India provides a good example of why FSM infrastructures are so important to the health of communities worldwide. In 2012, 626 million Indians defecated in the open and out of a total of 7935 towns in India, only 162 had access to sewage treatment plants. This indicates that as of September 2012 more Indians had access to a mobile phone than a toilet.

One of the most common reasons for this was that when houses in India were built it was unusual to include a toilet due to them being seen as unclean. Many Indians believed that it was cleaner to defecate in the open, rather than have a toilet within their homes and that open defecation was often associated with strength and good health.

Fast forward to 2019 and the Indian prime minister Narendra Modi has declared India to be open defecation free. More than 110 million toilets have been provided to the 626 million people who previously did not have access to them. Although this is a massive step forward to create solutions to the first two steps of the sanitation value chain, there are still issues surrounding the collection of treatment of the

faecal sludge once the latrines become full. This again leads to issues as many experts claim that pronouncing India 100% open defecation free is misleading, as once the toilets fill there are no easy disposal methods, therefore leading people to being forced once again to defecate in the open (12,13).

1.2 Drying

Increasing urbanisation and industrialisation have resulted in a dramatic increase in the volume of municipal wastewater and sewage produced around the world. Within wastewater treatment systems, the sewage passes through a series of treatment steps that use physical, biological, and chemical processes to remove nutrients and solids, break down organic materials, and destroy pathogens. The end products are treated water and sludge. The former is released to streams and rivers, and the latter is generally disposed of by land filling, land application or incineration.

Thermochemical technologies are becoming an attractive method to dry faecal sludge in comparison to the more conventional disposal methods due to the volume reduction and minimized environmental impacts. The end products can be used as a fertiliser, low-cost carbon, bio-oil, syn-gas as a gaseous fuel or chemical feedings (14).

There are many ways to dispose of the resulting dried faecal sludge, however they all have their disadvantages. The use of digested sewage sludge on farmland on one hand is limited by the uptake capacity of the soil and on the other is not reasonable due to the growing concentrations of toxic organic constituents and heavy metals in the sludge resulting from increasing industrialization. The disposal of dewatered sludge in sanitary landfills incur inherent chemical energy loss and surveillance for an indefinite period is needed. Ocean dumping disturbs, at least locally, the ecology of the biosphere and should be avoided (15). A combination of all these factors indicates why a greater understanding of the drying properties of faecal sludge is needed. There is currently limited information available on the drying of faecal sludge, but the same material principles can be applied when looking at different industries such as food and paint.

Drying can be defined as a unit operation in which the supply of heat accomplishes a liquid-solid separation, with the separation resulting from the evaporation of liquid. It is important to note that in the majority of cases, when water is evaporated solvent evaporation also occurs (16). The drying process is an essential unit operation with diverse applications in chemical, agricultural, biotechnology,

food, polymer, ceramics, pharmaceutical, pulp and paper, mineral, and wood processing industries (17). Drying involves the application of heat to a material which results in the transfer of moisture within the material to its surface, and then the removal from the material into the atmosphere (18). It is thought that there are currently around 200 different ways to dry a material with new techniques being developed on a regular basis. This indicates that fully understanding the drying properties of a material are increasingly valuable to insure that the right technique is chosen (19).

When looking at the different techniques to use it is important to consider the composition of the material, its size and shape, the optimum drying temperature, along with the climatic conditions of the environment including humidity and room temperature. Temperature is one of the most decisive factors when choosing a drying technique. It is usually beneficial to use higher temperatures as it will allow drying times to be accelerated, however the process then becomes drastically more energy intensive (18,20).

When investigating the most effective method to dry a material it is important to consider the two types of water present. Moisture held in loose chemical combination present as a liquid solution within the solid, or even trapped in the microstructure of the solid, which exerts a vapour pressure less than that of pure liquid is referred to as bound moisture. Moisture in excess of bound moisture is known as unbound moisture. When a wet solid is subjected to thermal drying, two processes occur simultaneously:

- 1) Transfer of energy (mostly heat) from the surrounding environment to evaporate the surface moisture. The removal of water as vapour from the material surface depends on the external conditions of temperature, air humidity and flow, area of exposed surface, and the pressure.
- 2) Transfer of internal moisture to the surface of the solid and its subsequent evaporation due to process 1. The movement of moisture internally within the solid is a function of the physical nature of the solid, the temperature, and its moisture content.

The rate at which drying is accomplished is governed by the rate at which the two

processes proceed, with one process being the limiting factor governing the rate of drying (17).

The heat required for drying can be supplied by the mechanisms of convection, conduction and radiation.

Convection – a carrier gas (usually air) supplies the heat for the evaporation of the liquid by the conversion of sensible heat into latent heat. The carrier gas subsequently entrains the volatile matter.

Conduction – the heat is supplied indirectly, and the carrier gas serves only to remove the evaporated liquid. Typically, the airflow is approximately 10% of the airflow used in a convective process. Conduction of heat is the heat transport mechanism at contact drying.

Radiation – this type of drying can in principle be nonpenetrating, such as the drying of paint by infrared radiation, or penetrating, such as the drying of food or pharmaceuticals by dielectric drying. Dialectic drying (radiofrequency dying and microwave drying) is the only process in which heat is developed in the material being dried rather than being diffused into the material. Again a carrier gas is required to remove the evaporated liquid (16).

The separation operation of drying converts a solid, semisolid or liquid feedstock into a solid product by evaporation of the liquid into a vapour phase through the application of heat (14).

Drying is thought to be the oldest, most common, and diverse of chemical engineering unit operations. It competes with distillation as the most energy-intensive system due to the high latent heat of vaporisation and the inherent inefficiency of using hot air as the most common drying medium.

When a material is exposed to air at a particular temperature and humidity, the material will either gain or lose water until an equilibrium condition is achieved. Essential features of the drying process are the phase changes, leading to a solid phase as the end product (17).

Understanding the thermodynamic aspects of a drying process is vital when determining how best to dry different types of faecal sludge. Drying is driven by the difference in the thermodynamic activity between both water as vapour in the air

and water as moisture in the wet solid. Drying occurs when the thermodynamic activity of the moisture in the solid is higher than the vapour water in the air stopping the thermodynamic equilibrium. In comparison if the activity of the water vapour is higher than the activity in the moisture in the solid then the solid can gain moisture due it being hygroscopic in nature.

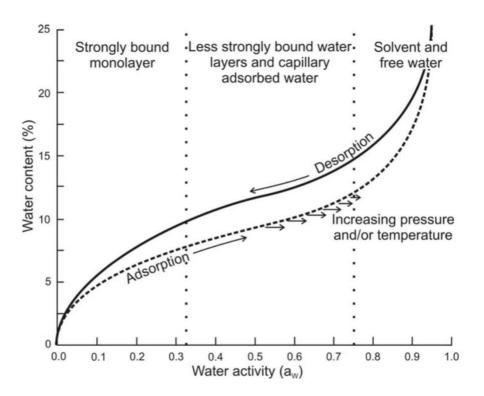


Figure 1.3: Sorption isotherms (21)

1.2.1 Moisture held in Faecal Sludge

The moisture in sludge can be roughly divided into four categories: free water, interstitial water, vicinal water, and water of hydration (Figure 1.4). Free water is surrounded by sludge particles and water does not directly combine with the sludge making it easy to be separated. Free water generally accounts for around 70 % of the total water content of sludge (22). Interstitial water refers to water that is trapped in the crevices and interstitial spaces of the flocs and microorganisms. Some portion of water held within the floc structure can be released when the microbial cells are disrupted. Some of the interstitial water may not behave physically as bulk water. Vicinal water is associated with the multiple layers of water

molecules held tightly to the particle surface. This water can be within cells as well if it is associated with a solid surface. Vicinal water is not free to move but adheres to solid surfaces; while interstitial water is free to move when physical confinement is eliminated. And finally water of hydration is water that is chemically bound to the particles and can only be removed by thermal destruction of the particles (23).

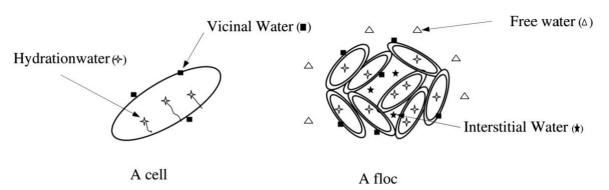


Figure 1.4: Schematic model of the various forms of water in bio sludge (24)

The energy of water molecules is reduced as the water bonds chemically with other molecules and structurally via Van der Waals forces.

When a sample is heated the vapour pressure rises allowing the water to begin to evaporate. This only works with unbound water as bound water has a lower vapour pressure than the 'pure water' or unbound water that had previously been removed.

To determine whether the water is bound, or unbound it is important to look at the type of material that the water is held in. Hygroscopic materials such as faecal sludge attract water and find ways to bind the moisture, while non-hygroscopic or hydrophobic materials keep water out of the capillaries, fibres, and chemical reactions.

Collagens can provide a useful in site into a hygroscopic material dries. The binding of water to molecules within collagens has been investigated leading to a triple helix as a protein motif being found within the fibril-forming collagen (25). X-Ray diffraction and nuclear magnetic resonance (NMR) studies have demonstrated that most proteins are surrounded by bound water. Dielectric measurements, dynamic mechanical spectroscopy and heat capacity measurements have shown that the structure of the water in the collagen fibrils is very different from the bulk water in

terms of restricted mobility and asymmetric orientation (25,26). Within the bonding there are two free carbonyl groups which allow for hydrogen bonding to bind water. The water that bridges the amide and carbonyl groups at the site of substitution are called interstitial waters which can be proven to be present thermodynamically (27). The presence of a hydrogen bond can be demonstrated spectroscopically and through comparable crystallographic analysis of organic compounds (28). The most frequent biological explanation assumes water structures extend multiple layers from surfaces of compactly folded macromolecules to explain large amounts of perturbed water (29). Interstitial waters can also affect the nutrient levels found within the sample with enriched nutrients found where interstitial waters were prevenient (30).

The binding of water has played an important role within bio-drying. Bio-drying allows for a lower moisture content of faecal sludge to be reached under a high temperature with the biodegradation of the organic matter present. It has been found that under aerobic conditions, microbes found within easily degradable organic matter will release a large amount of heat through aerobic respiration, part as the process for its own metabolism and the rest in the form of heat. The microbial activity within the sludge can cause bound water activation, reducing the bondage of the water state and making it easier to be heated to steam out (22).

1.2.2 Pathogens

When drying faecal sludge, it is important to consider that it contains germs, eggs, and other living organisms, some of which create pathogens and parasites. The pathogens and parasites found within human excreta result in several illnesses including diarrhoea, malnutrition, poor growth, and iron and vitamin A deficiencies (13). Urine is usually sterile and only poses a risk in special cases, with the majority of pathogens present being found to contribute towards typhoid, paratyphoid, and bilharzia. Within fresh excreta there are four main groups of organisms which can cause harm to humans: bacteria, viruses, protozoa, and helminths (31). Bacteria and viruses are immediately infectious once defecated, while protozoa are excreted primarily as cysts, allowing them to survive outside of the human body. The eggs produced by helminths are resistant to environmental conditions and require a period outside of the body, with some parasites such as bilharzia, requiring an intermediate host before they can become infectious (32).

When a person defecates, a pathogen - which cannot be contained or destroyed - causes the environment in which it appears to become contaminated. A contaminated environment puts people at risk of exposure to pathogens which in turn can lead to infection and disease (Figure 1.5) (33). Therefore, proper containment of faecal sludge is vital to human health.

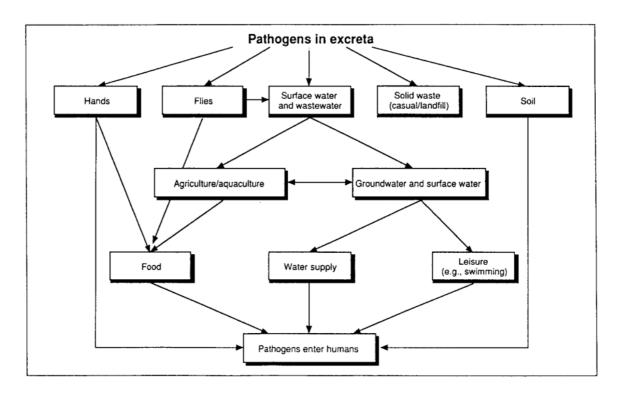


Figure 1.5: Transmission routes of Pathogens (33,34)

Figure 1.6 gives the temperature-time combination required for the inactivation of pathogens. A treatment process with time-temperature found within the safety zone should be lethal to all pathogens. The disinfection time needed to be in the safety zone is estimated at one week at a temperature of around 46 °C, one day at a temperature of around 50 °C, one hour at a temperature of around 62 °C and only a few minutes with a temperature of above 70°C.

The presence of pathogens within faecal sludge has led to numerous studies into the thermal pre-treatment of faecal sludge and biogas production. This is a major problem when anaerobic digestion is used to stabilise the sludge as it is insufficient at inactivating all the pathogens. This is a popular method compared with other methods of waste treatment, such as land filling, incineration, and composting, as it has the advantages of reducing the amount of sludge total solids and generating biogas, which is a renewable energy source (35).

There are several different drying technologies that could be used to remove water from faecal sludge, whilst also removing the pathogens, some of which will be discussed here.

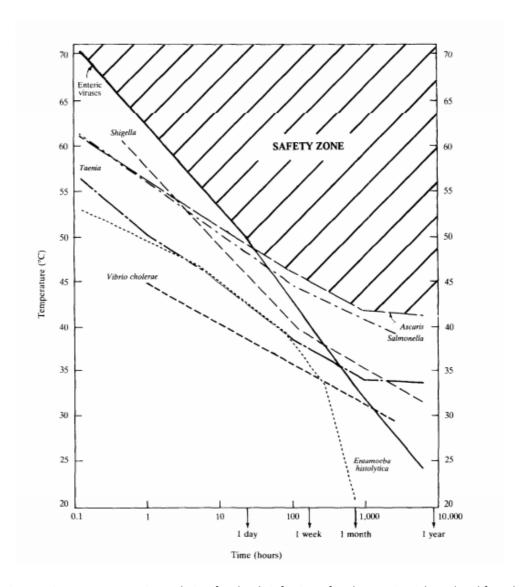


Figure 1.6: Temperature-Time relation for the disinfection of pathogens in night soil and faecal sludge (36)

1.3 Drying Techniques

1.3.1 Drying Beds

Drying beds are currently the most popular method used to dry faecal sludge due to their ease of use and ability to remove all pathogens providing that the sludge is left in the beds for long enough. There are two types of drying beds, planted and unplanted (Figure 1.7).

Unplanted sludge drying beds are shallow filters filled with sand (10-15 cm thick) and gravel (25-30 cm thick) with an under-drain at the bottom to collect leachate. Sludge is discharged onto the surface for dewatering, and two different drying processes take place.

- 1) Dewatering this is where the water will filter through the gravel. This process only takes a matter of days if not hours. This is a quick way to remove a large volume of the liquids present which usually lack pathogens.
- 2) Drying this is where water leaves through evaporation. This process can take several weeks or months depending on the time of year.

Depending on the faecal sludge characteristics, a variable fraction of around 50 - 80 % of the sludge volume drains off as a liquid which then needs to be collected and treated prior to discharge. Once dry the sludge is mechanically or manually removed from the dry bed for further processing to ensure complete pathogen removal (10) (Figure 1.8).

Planted drying beds, are beds of a porous media that are planted with emergent macrophytes. The drying beds are loaded with layers of sludge that are subsequently dewatered and stabilised through multiple physical and biological mechanisms (10). There are however limited examples of planted drying beds being used within faecal sludge management, although current research has produced promising results and it is expected they will be implemented round the world, especially in tropical regions of low-income (10). They are a promising technology for faecal sludge treatment in low-income countries having low energy requirements, low operating and maintenance costs, and can generate revenue to offset treatment costs through resource recovery as fodder and soil amendments (37).



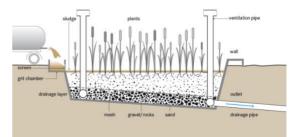


Figure 1.7: Planted and Unplanted Drying Beds (10)

Sustainable treatment options for faecal sludges dewatering is still a crucial problem in developing countries, though on site sanitation facilities remain the most predominant system within these regions (38).

Drying beds are usually used for small to medium sized communities (39) due to the technology depending upon land availability. Climate factors and the quantity and composition of the sludge are also important factors to consider when installing a drying bed (40).

Fresh faecal sludge, derived from bucket or public latrines with a high emptying frequency (once per month or more often), is more difficult to dewater and produce odours during digestion. In order to overcome this problem, fresh faecal sludge can be exposed to a chain of treatment technologies with a digestion step first (10).

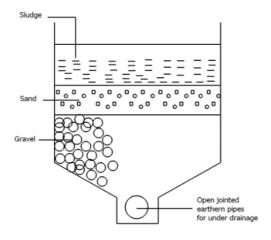


Figure 1.8: Design of a drying bed for the dewatering of sludge (40)

1.3.2 Ultra Violet Radiation (UV)

UV curing is a photochemical process in which high-intensity ultraviolet light is used to instantly cure or 'dry' inks, coatings and adhesives. The UV curing system relies on UV irradiation onto the surface and therefore cannot guarantee the curing degree of shadowed areas (41). UV curing is commonly used to cure polymer coatings and films due it being a photo initiated radical polymerisation reaction. Coatings include a small amount of photo initiator, which when exposed to UV radiation generates free radicals that initiate crosslinking (42). The rate of polymerisation depends on the intensity of the radiation used. Coatings cured by this method are limited to clear or lightly pigmented systems and a radiation penetration depth of 2cm. The process gives a low energy, environmentally friendly rapid cure with low emissions of volatile organic compounds that has applications in many settings particularly in fast drying varnishes and printing inks and quick setting adhesives and composites (43).

1.3.3 Near Infrared Radiation (NIR)

NIR is a radiative curing technique, however unlike UV curing it is essentially a fast-thermal method of curing (44). NIR curing has been used within the coil coating industries to produce pre-painted galvanised steel strips for use within the construction sector due to their wavelengths resulting in fast thermal heating of metal substrates (45). Within the print industry, typically IR radiation is used to directly heat the ink to induce particle sintering. A drawback to using IR is that the polymer substrate itself is easily damaged as it too has a high absorbance in the IR region of the electromagnetic spectrum. Therefore, the NIR region of the electromagnetic spectrum is used as typically polymer compounds do not have strong absorbance's at these wavelengths preventing the polymer sheets from being damaged (46).

The wavelengths of NIR radiation are just longer than the wavelengths of visible light and just shorter than IR, ranging from 750 nm to 3000 nm with a peak at round 1000 nm where typically polymer compound do not have a strong absorbance (45) (Figure 1.9).

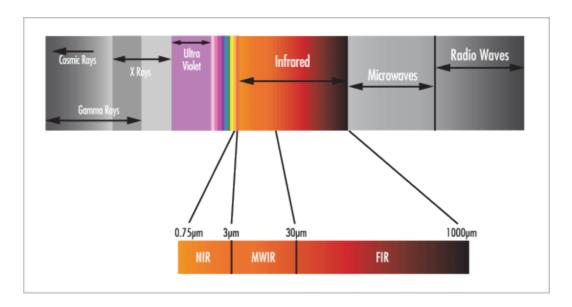


Figure 1.9: Electromagnetic Spectrum (47)

NIR has also been used to determine the soluble solids and dry matter content with materials using a handheld instrument working at a wavelength range of 729 to 975 nm. It is a cost effective, and non-destructive method of analysis and is often used within industries concentrated on quality control (48). It offers energy efficiency advantages because the heating is produced by direct absorption by the material itself meaning there is no need to preheat an entire oven and changes in the settings can feed back almost instantly on panel temperature (44). Water is also highly absorbing within the NIR region indicating that NIR drying could be used as a potential faecal sludge treatment technology. NIR can also penetrate deeper into organics than other types of radiation such as UV, so can be used where deeper penetration of the radiation is required as within faecal sludge treatment sites.

1.3.4 Mid Infrared Radiation (MIR)

As with NIR drying, MIR is considered a promising drying technique as it has the advantage of high energy efficiency, short drying times, uniform heating of material and easy control of the materials temperature (49). MIR radiation focuses in at the wavelength range 3-30 μ m which is where a lot of the current dewatering of faecal sludge research is focused. One of the main problems found when using any form of infrared drying is that crusting is often formed at the surface (50). MIR drying is often used within the food industry as it allows for the radiation to be absorbed by the material, without heating the surrounding air (51). It is often used in

conjunction with other drying techniques to improve the drying efficiency and improve the quality of the end product (52).

1.3.5 Deep Dewatering

Deep dewatering is another method used and can be found by adding inorganic and chemical conditioners. The disadvantages of using these conditioners is that they can bring about incomplete combustion, equipment corrosion and secondary pollution, the main disadvantage is that they will greatly reduce the heat value of the sludge cake. In order to prevent these problems, the system is often conditioned with sawdust or lime. Both sawdust and lime can help to speed up the sludge dewatering process, however sawdust is thought to be the superior conditioner as it improves the calorific value and has the ability to air dry (53,54). Faecal sludges moisture content is also decreased when conditioned with sawdust, which decreases the compressibility, the bound water content and improves the porosity of the sludge cake (53). This then makes drying the sludge cake by absorption a more feasible option (55).

1.3.6 Separation Techniques

Water in faecal sludge can be available in both free and bound forms. This difference is important to consider within the understanding of different separation methods, as free water is easily removed, and the removal of bound water is much more difficult. As shown in Figure 1.10 bound water includes interstitial, surface, and intracellular forms of bound water (10). By removing the safe liquid, it can lead to easier and cheaper pathogen removal of the solids.

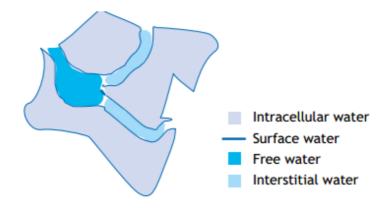


Figure 1.10: Water forms in sludge floc (10)

1.3.6.1 Gravity Separation

Within FSM, gravity separation is one of the most employed method of liquid-solid separation. It works by separating the suspended particle within unbound water. This simple method works by allowing the heavier particles to settle under quiescent conditions at rates based on the size of the particles, suspended solids concentration and flocculation (10).

1.3.6.2 Centrifugation

This method is traditionally used for the liquid-solid separation of wastewater sludge but can also be employed for faecal sludge for the partial removal of bound water. The process works by rotating the sludge at a high speed, with the centrifugal forces accelerating the sedimentation process. Solids settle out at the centrifuge walls, where they are pressed and concentrated (10,56).

1.3.6.3 Filtration

Another commonly applied mechanism for liquid-solid separation within FSM is filtration. This is the separation method found within planted and unplanted drying beds. The process uses a filter media to trap solids on the surface of the filter bed, whilst the liquid percolates through the filter bed and is collected in a drain or evaporates from the solids.

The main physical mechanisms found within filtration are shown in Figure 1.11.

These methods cannot be individually quantified so therefore the design of drying beds rely on empirical calculations (10).

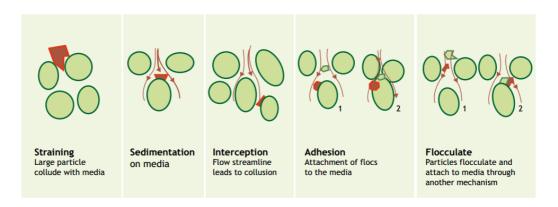


Figure 1.11: Schematic presentation of the mechanisms affecting flow in filter media (10)

1.3.6.4 Evaporation and Evapotranspiration

Evaporation occurs when water is released into the air as a vapour, and transpiration is the process by which plants release water vapour to the air as a part of their metabolic processes.

Evapotranspiration is the combination of these two mechanisms. In addition to filtration, dewatering in drying beds also occurs through evaporation, and with planted drying beds through evapotranspiration. For both mechanisms to occur, the surrounding environment needs to have an evaporative demand, which means that the air is not saturated (10).

1.3.6.5 Heat Drying

Heat drying is used to evaporate and dewater wastewater sludge beyond what can usually be achieved by more conventional passive methods. Currently heat drying is applied more to wastewater sludge processing than for faecal sludge, but this technology should be transferable.

Heat drying achieves both weight and volume reductions as water is lost in the form of vapour. The temperature of the sludge is increased through energy transferred from an external heat source. This allows the free water at the sludge surface to evaporate at a rate that depends on the ambient air temperature, humidity, flow and pressure, and the exposed sludge surface (10).

1.3.6.6 Screening

Screening is another important mechanism in FSM. This method removes the municipal waste and large solid objects from the faecal sludge, it prevents clogging and pump failures, and enhances the value of treatment end products. Bar screens installed in a vertical or inclined position against the incoming flow make a physical barrier that retains coarse solids. The distance between the bars are set such that the liquid and small solid particles can flow through while the larger solids are trapped (10).

1.3.7 Summary

There are many different drying techniques that can be used within the Faecal Sludge Management field, some which are currently widely used, and some which are at the beginning stages of investigation for use within the field.

The most commonly used are drying beds as these provide an inexpensive drying option for communities with limited resources. The main issue with the technique is that they require a large area of land and release unpleasant olfactory's into the environment. Separation techniques are also invaluable within the FSM field as they allow water to be easily removed from the faecal sludge often at very low costs.

1.4 Faecal Sludge

An important distinction to make between sewage sludge and faecal sludge is that sewage sludge is relatively homogenous, whereas faecal sludge is highly variable due to it coming from several different sources.

Sewage sludge can be defined as the residue generated from the treatment of wastewater. The two most commonly found types of sewage sludges are primary sludge (1°) and secondary sludge (2°). 1° sludge is the material collected from the primary settling tanks employed in wastewater treatment plants. 2° sludge, also known as biological sludge, is the sludge generated from the biological treatment of the wastewater drained from the settling tanks.

Chemical sludge is another form, and constitutes sludge that has been produced with the aid of chemicals, which are typically used to either facilitate the precipitation of the hard-to-remove substances, or to improve suspended solid removal (57).

Faecal sludge comes from onsite sanitation technologies such as pit latrines and septic tanks, that has not been transported though a sewer. It is a raw or partially digested slurry or semisolid and results from the collection, storage, or treatment of combinations of excreta and Blackwater, with or without Greywater (10). It contains excreta from an on-site sanitation technology, any water used (flush water, greywater, anal cleansing water etc) and other materials such as paper. If a composting latrine is used it will also contain a cover material such as ash or sawdust. Faecal sludge can also include any solid wastes produced within a house that have been added to the pit or containment vessel (31).

Excreta is made up of water, faeces and urine containing a number of components (Figure 1.12 & Figure 1.13).

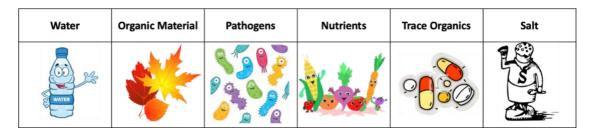


Figure 1.12: The 6 main components found within excreta (31)

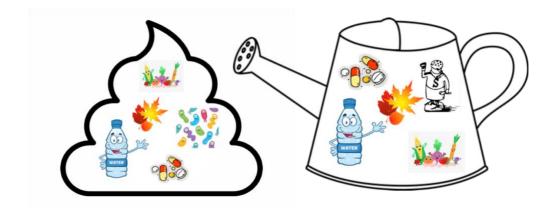


Figure 1.13: The components found within faeces and urine of a healthy person (31)

Looking at Figure 1.13, the urine produced does not contain a traceable level of pathogens, if the two components can be successfully separated, the urine will not require a pre-treatment step, and can be reused safely.

1.5 Types of latrines

It is important to analyse faecal sludge from several different sources. Within this research three different types of faecal sludge were analysed along with human faeces.

1.5.1 Ventilated Improved Pit Latrines (VIP)

One of the most common ways to decrease open defecation within developing countries is to install Ventilated Improved Pit Latrines (VIP) as part of faecal sludge management schemes. In South Africa, 31.3% of households have their sanitation needs met by a pit latrine; 12.5% are VIPs while 18.8% are pit latrines without ventilation (58).

The South African Department of Water Affairs and Forestry (DWAF) defines a sanitation system as the process by which waste is managed from the generation point to the disposal or recycle point (59). This is achieved using a sanitation facility who's infrastructure allows handling, storage, removal, and treatment of faecal sludge (60). Due to the latrines being permanent structures, the pits need to be emptied when they become full. The eThekwini Municipality empties the pits on a five year cycle with no cost to the households, however the sludge then needs to be treated (58).

There are thought to be two different sanitation systems used within the developing world, flush and discharge, or drop and store (32). Where the population of an area is high, traditionally flush and discharge systems are found as there is an adequate supply of water. This water system requires the construction of a wastewater treatment plant or septic tank to handle the resultant waste. In most African countries the drop and store system is more common due to water access being a major problem especially within the rural and peri-urban areas (32). A ventilated improved pit latrine (VIP) is fitted with a bent pipe which facilities airflow within the pit and the removal of bad odour. The pipe is installed with a fly screen at the top, which stops flies from leaving or entering the pit latrine (61).

1.5.1.1 Accumulation and Content

Depending on the demographic, geophysics and biological factors, there are variations within the content of a VIP latrine (62). Sludge accumulation rates in pit latrines and septic tanks will vary with a variety of factors, the most important are the number of users, the degree to which the pit is drained, and whether the pit is used for disposal of another household waste. In practice sludge accumulation rates vary from as little as 10 litres per user per year, to as much as 100 litres per user per year. The average accumulation rate has been found to be around 25-30 litres of sludge per user per year (63).

Figure 1.14: Manual Pit Emptying (64,65)

The content of the pit latrines varies dependent on the users feeding habitats and anal cleansing materials used (66).

Figure 1.15 a) shows the four proposed stages that are present within the pit latrine content.

- i) this is the layer which is located at the top, just below the pit hole. It shows all the readily biodegradable species originating from faeces is aerobically degraded by naturally occurring micro-organisms within a very short time of arriving on the surface of the pit;
- ii) this layer is at the top of the pit although not directly below the pit hole where a significant portion of the remaining biodegradable material is aerobically degraded

before being covered over by new pit contents;

- iii) the remaining biodegradable material, including organic residual from dead cells within micro-organisms and from the original faeces are slowly converted into soluble products, methane gas and carbon dioxide in the buried layers of the pit contents;
- iv) this is the lowest layer in the latrine and after a long residence time the material that remains in the pit is largely non-degradable (67).

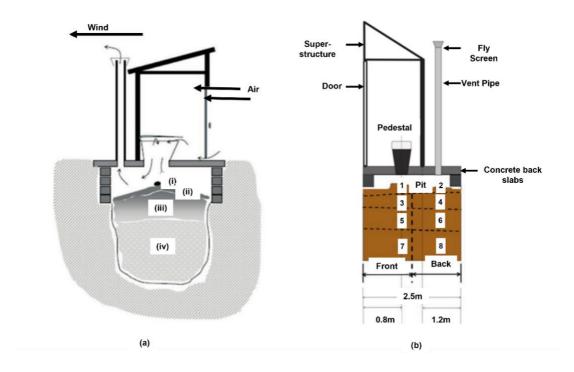


Figure 1.15: a) Diagram of a pit latrine showing the different conceptual layers (i) fresh stool; (ii) partially degraded aerobic surface layer; (iii) partially degraded anaerobic layer beneath surface; (iv) completely stabilised anaerobic layer.

b) Diagram of a VIP latrine showing the modified layers where the faecal sludge was split into front and back sections (58).

In 2013 Zuma et al (58) proposed a new layout for the layers within a pit latrine as seen in Figure 1.15 b) with the sludge being split into 8 sections, 4 in the front section and 4 in the back section (58), which is now the more accepted layout. By analysing the chemical and physical properties it is possible to analyse the biological stabilisation by looking at the COD, volatile solids, and the biodegradable COD. (62,66).

Using these results, it is possible to form a picture of the life cycle of the pit. This

can show when the pit was first commissioned and emptied, how fresh the material added to the pit is, and how stable the content within the pit is (62).

1.5.1.2 Management of the Sludge

While in use, VIP latrines eventually fill up and will need to be emptied or discarded. The most common practice is for the hole to be covered up and a new pit dug. This is not a sustainable solution in densely populated areas due to the lack of space to relocate the pit, so an alternative solution is to empty the pit and the removed sludge to be disposed onsite or transported for disposal (68). The emptying methods used are not always safe or clean.

There has been extensive research carried out in order to identify other solutions for dealing with faecal sludge. These have investigated the possible issues associated with pit emptying and its link to the sludge consistencies. This can be affected by the amount of water added to the pit, the ability for the water to leave or enter the pit, the type of anal cleansing material used, the diet of the users, and the presence of other solid or liquid wastes in the pit. Other avenues explored include cost, safety, sustainability and effectiveness (69).

Alternatives to the traditional methods found include anaerobic digestion to produce biogases, treatment within a waste water treatment works, the use of black solder flies, deep row entrenchment, composting, and energy recovery through different thermochemical processes (69,70). Other methods to recover energy from faecal sludge include hydrothermal processes such as hydrothermal carbonization and supercritical oxidation (71).

1.5.2 Urine Diversion Toilets (UDDT)

Urine diversion toilets (UDDT) are systems designed to separate urine and faeces so that they can be managed independently. Inputs into the system can include faeces, urine, cleansing water and dry cleansing materials.

The UDDT configuration ensures that the faeces, cleansing water and/or dry cleansing materials are captured in a portable container. This can then be easily removed and transported to be treated using either motorized or manual transport. Depending on the demand for urine end use and local requirements, the UDDT can divert the urine to the ground for infiltration through a soak pit, or alternatively it

can be directed into a portable container where it is stored before being transported for its intended use (72) (Figure 1.16). This is a valuable technology as typically the urine does not contain any of the pathogens present within the faecal sludge (73), however it does contain the valuable nutrients (Figure 1.13) so being able to separate this at the source allows for an easier capture of these nutrients.

These types of toilets are a good alternative to the more common VIP as they:

- a) Allow for cheaper emptying.
- b) After a sufficiently long dehydration time, faeces could be safely disposed of on-site.
- c) No new pits need to be periodically excavated.
- d) The risk of environmental pollution is limited (74).

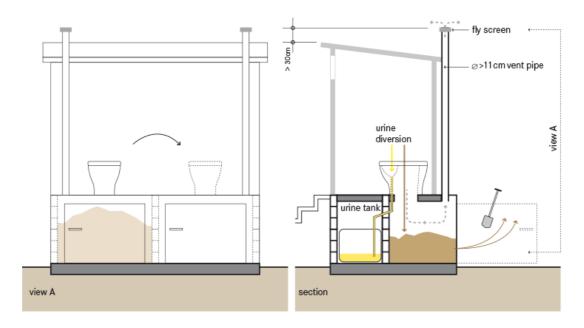


Figure 1.16: Schematic of a UDDT with two vaults

There are two main types of UDDT's, the single vault which has only one faeces vault, and the double vault type which has two faeces vaults that are used alternately. A UDDT can be configured either as a sitting toilet or a squatting toilet. A dry cover material is usually added to the faeces vault directly after each defecation event in order to help improve the aesthetics along with controlling flies, reducing odour, and speeding up the drying process.

Urine is separated at the user interface, drained through a piping system and either infiltrated into the soil for disposal or collected, stored, and sanitised in containers for use as a fertiliser. Faecal matter and anal wiping materials are collected into a ventilated vault directly below the user interface. Following defecation, the user covers the fresh faeces with a small volume of dry cover material in order to absorb moisture, control initial odour and prevent insect infestation.

1.5.2.1 Management of the Sludge

One of the most useful processes that are undertaken within a UDDT is its ability to remove most of the harmful pathogens found within the faeces. When properly designed, built, and maintained UDDTs can effectively contain pathogens from human contact and reduce the pathogen content in the faeces to enable reasonably safe handling of the faecal matter once the vaults have been emptied. It is important to note that a complete pathogen removal, including inactivation of all helminth eggs, cannot be guaranteed under ordinary circumstances with any type of UDDT (73).

1.5.3 Anaerobic Baffled Reactor (ABR)

Anaerobic baffled reactors (ABRs) are upgraded septic tanks with a series of baffles along the treatment chamber which the wastewater is forced to flow through (75). The ABR consists of alternating hanging and standing baffles, which compartmentalise the reactor and force the liquid flow up and down from one compartment to the next. This allows settling to occur in each of the up flow regions retaining high concentrations of biomass meaning that high treatment

rates can be obtained, while keeping the overall sludge production characteristically low (76).

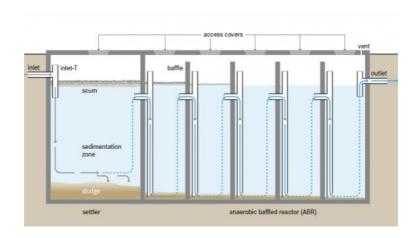


Figure 1.17: Schematic of an Anaerobic Baffled Reactor (77)

Every one to three years the ABR needs emptying, and the settled sludge needs to go through a treatment process in order to remove all the pathogens present within the ABR faecal sludge.

1.5.4 Human Faeces (HF)

Human faeces (HF) are a biomass which consists of a mixture of undigested fat, protein, water, polysaccharide, bacterial biomass, gut secretions, cell shedding and ash. HF can be converted into energy via thermochemical conversion processes such as gasification and combustion (78).

1.6 Processing Technologies

Below is a selection of some of the different technologies and approaches that are currently being used around the world to dry and treat faecal sludge.

1.6.1 Janicki Omni Processor

The Janicki Omni Processor is an efficient way to dry faecal sludge by combining three standard energy processes (Figure 1.18) (79):

- 1) Solid Fuel Combustion
- 2) Steam Power Generation
- 3) Water Treatment

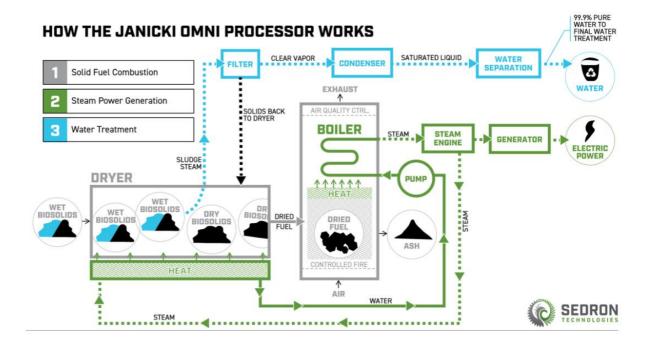


Figure 1.18: Schematic of the Janicki Omni Processor (79).

The first part of the drying system the solid fuel combustion, evaporates the moisture from the faecal sludge loaded into the system. The dried solid waste then continues into a fire where it is burned, reducing the solids to a dry fly ash which can then be used within the building industry.

At the same time steam power generation is added. The steam is created by heating water in a boiler in a controlled manner. The steam is then fed into a steam engine which runs the generator to produce electricity. This creates enough energy to run the whole Omni Processor and provides a surplus to the surrounding areas.

The last process is the water treatment. This takes the moisture that is leaving the faecal sludge in the first part of the drying system and sends it through a filtration system to purify the water and produces clean drinking water (79).

1.6.2 Latrine Dehydration Pasteurisation (LaDePa)

The LaDePa is a good demonstration of mid infrared drying (Figure 1.19). The process separates the detritus from the sludge by compressing the sludge in a screw compactor with lateral ports, through which the sludge is ejected. This is then deposited in a 25-40 mm thick layer of open pored matrix onto a porous, continuous steel belt.

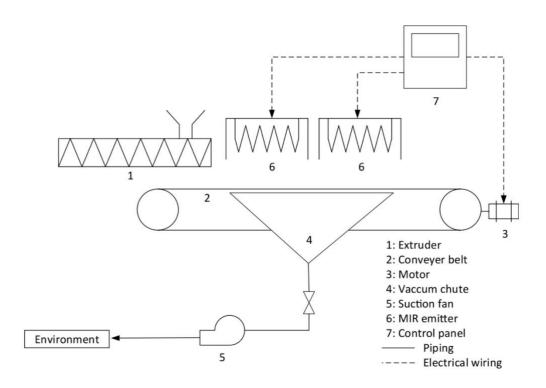


Figure 1.19: Schematic of LaDePa (80)

The drying section is composed of a porous steel belt, two MIR emitters, vacuum chutes, and a discharge hopper. The steel belt is driven by rollers while its tracking is regulated using compressed air. During operation, pellets produced by the extruder are deposited on the belt and then conveyed into the heating zone where they are exposed to thermal radiation emitted by two successive MIR emitters. The processed pellets are then discharged at the chute for collection. A vacuum under

the belt, created by a blower, induces an airflow in the heating zone to remove the evaporated moisture and enhance the drying process. A damper valve is placed in the pipe connecting the vacuum changer and the blower to regulate the airflow through the machine.

After pre-drying, using the waste heat from the internal combustion engine of the drive plant, the sludge on the belt, is conveyed through Particle System Separation (PSS) patented Parceps Dryer where it is subjected to pasteurisation, this also provides sufficient drying to take the sludge through the 'sticky' phase making handling simple. PSS's Parceps Dryer technology uses Medium Wave Infrared Radiation and a vacuum to draw air through a porous material or one with an open matrix (81,82).

1.6.3 Black Soldier Flies

One positive solution for waste processing is the larvae of *Hermetia illucnes* more commonly known as Black Soldier Fly Larvae (83). They consume large quantities of organic matter in a short period of time, reducing pathogens and converting excreta into organic fertilisers. This enables them to be used as part of simple engineered systems to reduce organic waste in low and middle income countries (84,85). The adult flies are neither a nuisance species or a mechanical vector of disease as they do not need to feed and can survive on fat stores from their larval stage (83). Black soldier fly larvae offer several advantages as a perspective waste processing system. These facilities can be developed and operated relatively cheaply due to low building and maintenance costs. They are also independent from a power supply and more adapted to the economic potential of developing countries (84). The larvae can reach maturity in 2-4 weeks depending on the temperature and food available, they are able to survive for up to 10 months in a state of quiescence. One of the main reasons that black fly larvae are suitable for FSM, is that they are able to extend their life cycles under environmentally stressful conditions (85).

1.6.4 Vermistabilisation

Vermistabilisation is the process that uses earthworms to stabilize organic matter such as sludge. This process has been used for waste stabilization for many years

particularly in the Philippines, China, and other southeast Asian countries. It has also been implemented in the UK, the Netherlands and Italy, not only to stabilize wastes but also to produce an organic end-product that can be used for horticultural purposes (86). Vermicomposting has become an alternative to conventional aerobic composting as it is not only rapid, easily controllable, cost effective, energy saving and zero discharge process, it also accomplishes the most efficient recycling of organics and nutrients (87). During this process, the appropriate species of earth worms are introduced to the organic matter. The worms derive their nourishment from the microorganisms that are involved in the waste decomposition and from the decomposing organic matter. The worms maintain aerobic conditions in the wastes, ingest solids, convert a portion of the organics to worm biomass and respiration products, and expel partially stabilised matter as discrete particles. Earthworms will only work in aerobic conditions. In anaerobic sludges they cannot exist and will not have a beneficial effect until aerobic conditions occur (86). Vermicomposting works in contrast to conventional aerobic composting, resulting in a homogeneous product with better quality in terms of desirable aesthetics, reduced levels of contaminants and more soluble and available plant nutrients. It has been found that Vermicomposting has caused a significant reduction in pH, volatile solids, specific oxygen uptake rates, total organic carbon, carbon/nitrogen ratios and pathogen contents. It also produces a substantial increase in electrical conductivity, total nitrogen and total phosphorus levels, when compared to compost (87).

1.6.5 Deep Row Entrenchment

The method of deep row entrenchment could help improve soil fertility where sludge acts as a slow-release fertiliser. This involves burying sludge in a ditch 200,000 mm long, 600 mm wide and 1,200 to 1,500 mm deep, with rows spaced 2.4 to 3 m between centres. In Australia, surface application of sludge achieved a 30% increase in the growth rates of existing pine plantations, and incorporation into the soil prior to planting improved tree height by up to 50% after 5 years. It can benefit the agroforestry sector as it increases the growth rate of trees and helps in recovering waste land such as mine spoils (88,89). Deep row entrenchment

overcomes the problems associated with the stabilisation of sludges, while also providing benefits to non-edible crops and to soil. The potential risk to the environment and public health can be effectively managed by providing periodic monitoring of the groundwater and soil (88).

1.6.6 Faecal Sludge Co-Composting

Applying faecal sludge to land is considered an attractive alternative compared to other popular forms of disposal. The direct soil incorporation of untreated faecal sludge is not necessarily beneficial to the local agriculture. Faecal sludge has a high pathogen content and releases an offensive odour which can cause major health concerns for farmers, farm workers and in some cases the consumer.

Stabilisation of the faecal sludge is a highly important process in order to allow it to be used safely as a compost, and to eliminate odours. Stabilisation of faecal sludge allows the nutrients within the faecal sludge to become readily available for plant use and prevents the sludge incorporated into the soil from being phytotoxic to plant growth (90).

Faecal sludge is often co-composted with other organic wastes in order to help improve its abilities as a composter. In Ghana, faecal sludge has been co-composted with mark waste, domestic waste, sawdust and municipal solid waste (91,92).

Although it has been found that it is more desirable to co-compost with market waste, than with household waste at a ratio of 2:1 (92).

Elsewhere faecal sludge has been used indirectly as part of sewage sludge and has been composted with other organic materials such as sawdust and oil palm wastes (93,94). One of the major problems with using a co-compost mixture of both faecal sludge and sewage sludge is the nutrient loss during the aerobic composting process. It has been reported that up to 50 % of the nutrient content of the initial materials could be lost due to ammonia volatilization (95).

Scientific research confirmed that improper and excessive use of mineral fertiliser in its organic faction is responsible for soil degradation in its organic faction, resulting in systematically decreasing crop yields. In order to counteract this tendency ecological cultivation methods are applied which includes human urine application, especially that collected from the dry toilet systems (96).

1.6.7 Fuel Blending

Fuel blending is a process that combines two or more materials to produce a finished product with superior quality. It is a widely used approach in biomass combustion, particularly for feedstocks with high moisture and ash contents or a low calorific value. Direct combustion of biomass and coal is progressively deployed in coal fired plants particularly in Germany and the Netherlands. Agricultural waste, municipal solid waste and energy crops are also increasingly applied in boilers and power plants with a 10-25 % blending ratio of fossil fuel or similar biomass material to reduce emissions and fossil fuel consumption or to utilise waste resources.

Fuel blending is a widely used approach in biomass combustion, particularly for feedstocks with low calorific value and high moisture content. In on-site sanitation technologies, fuel blending is proposed as a pre-treatment requirement to reduce moisture levels and improve the physiochemical properties of raw faeces prior to drying. As raw human faeces contain as much as 77 +/- wt.% moisture (97) and to limit the moisture entering the energy conversion unit, the residual solids are projected to undergo a pre-treatment process that involves partial drying and palletisation. These processes can consume a significant part of the energy contained and recovered from the faecal material if not adequately managed. Raw human faeces possess peculiar viscous, "sticky"" characteristics, varying waterbound levels and compositional non-uniformity from un-chewed and undigested foods that make sample homogeneity, handling and use difficult. For instance, the "lumpy" food particles in the residual solids cause blockages and the sticky characteristics promote adherence to mechanical equipment (98), which makes extrusion and pumping challenging. Upgrading the physiochemical properties of the faecal matter is needed prior to drying and palletisation, a process that can be improved by fuel blending (97).

It is thought that a fuel pre-treatment process that involves a blend with materials such as wood dust can make raw faeces more suitable for thermo-chemical conversion and for use in on-site sanitation technologies. This can enhance sustained fuel ignition and flame propagation, improving process efficiency and the continuous use operation of the energy conversion unit. It can accelerate the

product development and diffusion of heat through the material, by minimising the life-cycle energy requirement from drying and simplifying the fuel pre-treatment processes. This would accelerate the product development and diffusion with a link to existing user behaviour.

1.7 Thesis Scope

This thesis will investigate the way in which 4 different types of faecal sludge dry. By using STA-FTIR and UV-Vis-NIR, the drying rates, energy demands and the materials ability to dry using the suns radiation will the analysed. The chapters are broken down as follows:

Chapter 2: Methodology

The methods will be outlined in detail along with the instrumentation used for each experiment.

Chapter 3: Method Development

Prior to the work taking place, no methods had been developed using either the UV-Vis-NIR, or the STA-FTIR so containment and analytical procedures were developed to produce reliable and repeatable data sets.

Chapter 4: Results & Discussion 1 - STA-FTIR Analysis

This chapter will investigate how faecal sludge dries under differing conditions. It will investigate the drying rates, energy demand and evolved gases for all 4 types of faecal sludge.

Chapter 5: Results & Discussion 2 – UV-Vis-NIR Analysis

Using UV-Vis-NIR analysis, the interaction between the suns radiation and faecal sludge can be determined. Understanding how the deep the sun's rays penetrate into the faecal sludge can help to predict the drying capabilities of the different types of faecal sludge.

Chapter 6: Results & Discussion 3 Comparison of simulant sludge with real faecal sludge

Often simulant faecal sludge is used when developing new toilet systems and drying technologies. This chapter is going to investigate whether a simulant is an appropriate substitution for these developments by comparing data collected with faecal sludge.

1.8 Aims and Objectives

This landscape study was carried out to better understand faecal sludge drying and identify the relevant areas of investigation before identifying the gaps that sanitation practitioners are facing.

The objectives set following extensive research of the literature available on the drying of both simulant and faecal sludge include:

- Develop new experimental methods to analyse the material properties of faecal material.
- Identify whether simulant sludge is an appropriate alternative to faecal sludge when determining the best way is which to dry and it and developing new and improved drying techniques.
- Determine the ideal temperature for drying and thermal degradation.
- Characterise the effects of temperature as a function of thermal decomposition.
- Characterise the modification of its material properties during the drying process (thermal properties, radiative properties, calorific value).
- Identify and quantify the boundness of moisture within both simulant sludge and faecal matter.
- Estimate the energy demand of the total drying process and determine the most appropriate drying programmes for different types of faecal material.
- Identify the similarities and differences between the different types of faecal material analysed.
- Analyse the exhaust gas composition during drying.

Chapter 2 Methodology

This chapter will outline the methodologies used to capture the data found within this thesis. It will lay out the procedures followed, and the conditions used, along with the specific pieces of instrumentation used for each set of experimental work.

In order to understand the fundamental material drying properties of faecal sludge, both Ultraviolet-Visible-Near InfraRed (UV-Vis-NIR) spectroscopy and Simultaneous Thermal Analysis coupled with Fourier Transform InfraRed spectroscopy (STA-FTIR) were used to provide a broad picture of how the material dries.

There are two different analytical techniques that analysed both simulant and faecal sludge. This allowed a broad analysis to take place to draw conclusions on the way that faecal sludge dries both spectroscopically and thermally.

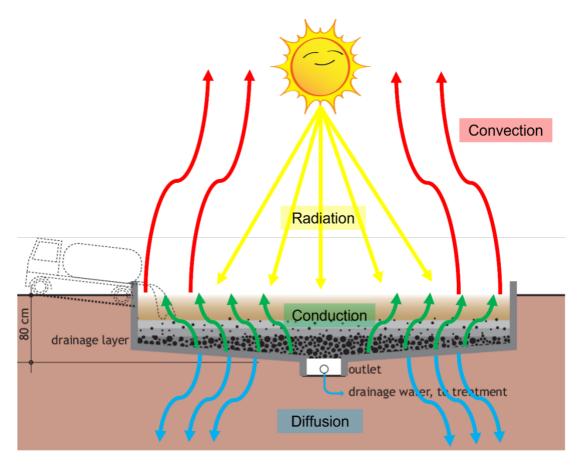


Figure 2.1: Diagram showing the mass transfer present when faecal sludge is dried using the suns radiation

Figure 2.1 shows the mass transfers present when faecal sludge is dried using the suns radiation, however the same mass transfer processes occur when using alternative heat sources. The moisture held within faecal sludge can leave the

sample in 2 main ways. Around 80% of the moisture found within faecal sludge is lost through diffusion, and the other 20% is lost as conduction and convection (10). The conduction process occurs when heat is applied in the form of the suns radiation to the faecal sludge causing the heat energy to be transmitted through the material due to collisions between neighbouring atoms or molecules. As the material heats up the water molecules begin to vaporise and are lost from the material as convection.

These three mass transfer processes occur simultaneously, however the rate of drying is dependent on the heat sources. This is why both UV-Vis-NIR and STA-FTIR analysis are needed to provide information on how the temperature effects the drying rate, and how the faecal sludge reacts with the suns radiation.

2.1 Techniques

2.1.1 UV-Vis-NIR Spectroscopy

UV-Vis-NIR spectroscopy is an analytical technique that can be used to characterise materials based on the wavelengths of light that they absorb. There are three classes of compound that can be spectroscopically analysed using a UV-Vis-NIR: organic compounds, inorganic compounds, and non-absorbing species (99).

UV-Vis-NIR spectroscopy studies the attenuation of an incident light beam after it is transmitted through or reflected off a sample. It works by allowing the molecule to absorb energy in the UV, visible or near-infrared part of the spectrum corresponding to the energy required for a possible electronic transition, a lone pair, or electrons in their ground state to get excited to higher energy states (100).

The quantitative analysis works due to the Beer-Lambert Law (Equation 1). This states that when monochromatic radiation is passed through a light absorbing medium, the decrease in the light intensity with thickness of the solution is proportional to the solution concentration (101).

$$A = \varepsilon. b. c$$
 Equation 1

Where A = absorbance, ε = molar absorptivity (mol cm L-1), b = path length (cm) and c = concentration

There are three main types of UV-Vis-NIR analysers which are all required for different areas of research posing the most difficult decision of which instrument to use:

1) Single Beam

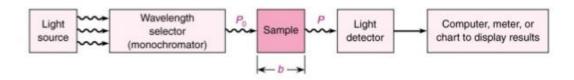


Figure 2.2: UV-Vis Single Beam spectrophotometer schematic (102)

This offers a low cost, simple design which can often be portable. It has a better signal to noise ratio (S/N) due to a higher signal and fewer parts but takes a long

time when scanning a large wavelength range. Overall, this instrument is best used for quantitative work (Figure 2.2).

2) Double Beam

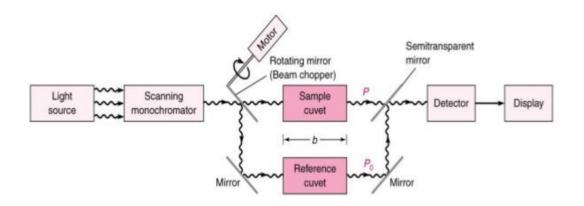


Figure 2.3: UV-Vis double beam spectrophotometer schematic (102)

The double beam allows compensation for source drift and short-term noise and can record an entire spectrum due to its scanning function (Figure 2.3).

3) Diode Array

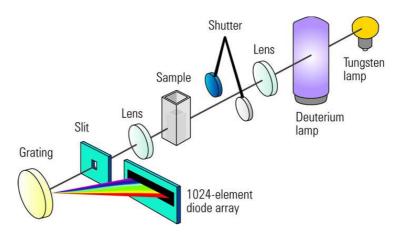


Figure 2.4: UV-Vis Diode Array spectrophotometer schematic (103)

This instrument has a much more rugged design and allows for very fast spectral acquisition. It is ideal if you are measuring a fast reaction where a slower scan time could miss something. It also allows for photobleaching, providing easy background subtraction (Figure 2.4).

UV-Vis-NIR was chosen as it allowed for the whole of the solar spectrum to be analysed simultaneously. As faecal sludge is regularly dried using the sun, analysing how it interacts with the whole of the solar spectrum was important as it provided an in site into how deep the radiation penetrated in the faecal sludge and how much of the suns radiation was absorbed into the sample.

The instrument chosen was the Perkin Elmer Lambda 750, like most high-end UV-Vis Spectrometers it is a double beam instrument allowing for precise measurements to be taken.

2.1.2 Simultaneous Thermal Analyser coupled with a Fourier-Transform InfraRed Spectroscopy (STA-FTIR)

The STA-FTIR is a hyphenated system combining the Perkin Elmer STA (6000) with the Perkin Elmer FTIR (Spectrum Two). The system works by placing the sample into the STA and heating it to release any volatile materials or combustion products. These volatiles are then transferred into the IR cell where the components can then be identified.

The STA blends together the analytical process and results that would be produced using both thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). It enables real-time measurements and analysis of a samples weight change and heat flow in one compact instrument.

The TGA technique measures the mass change (loss or gain) and the rate of weight change as a function of temperature, time, or atmosphere (Figure 2.5).

There are 4 reasons for the decrease in mass:

- 1) Decomposition breaking apart of chemical bonds
- 2) Evaporation Loss of volatiles with elevated temperature
- 3) Reduction interaction of sample to a reducing atmosphere
- 4) Desorption release of the absorbed substance for the surface

There are 2 different reasons for an increase in mass to be seen

- 1) Oxidation interaction of sample with an oxidising atmosphere
- 2) Absorption

Both the reasons for increases and a decreases in mass are kinetic processes meaning that there is a rate at which they occur (104).

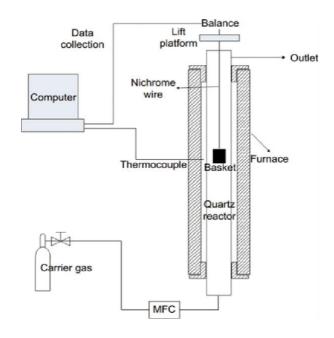


Figure 2.5: TGA schematic (105)

DSC measures the energy required to maintain an almost zero temperature difference between a sample and reference material subjected to the same hating program (Figure 2.6).

It is used to show the response when heating a sample. When a material undergoes a physical transformation, less or more heat (exo or endothermic) will need to flow to it compared to the reference sample (106).

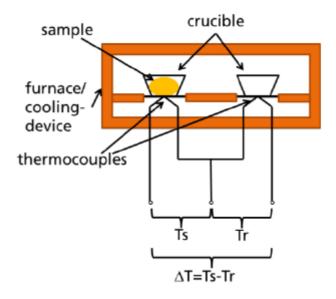


Figure 2.6: DSC schematic (106)

Both systems are combined within the STA to have one instrument that can produce both the mass loss and heat flow of a sample. Once the sample has been run through the STA, its evolved gasses then pass through the FTIR. This allows for the determination of the volatiles that are being released at different temperatures as the simulant sludge is dried.

FTIR is used to characterise materials based on their response to IR wavelengths. When organic molecules get excited by IR radiation, it causes them to vibrate. Light covering the whole frequency range, typically $5000 - 400 \, \mathrm{cm^{-1}}$, is split into two beams and when the beams are passed through the sample one beam is made to transverse a longer path than the other. Recombination of the two beams produces an interference pattern that is the sum of all the interference patterns created by each wavelength in the beam. By systematically changing the difference in the two paths, the interference patterns change to produce a detected signal varying with optical path difference (107).

When using infrared spectroscopy to analyse complex molecules or samples it is important to remember that they have many vibrational modes which involve the molecule. Some of the vibrations identified are associated with the vibrations of individual bonds or functional groups (Figure 2.7), while others are considered as vibrations of the whole molecule. These localised bond vibrations are either stretching, bending, rocking, twisting, or wagging, and can be very useful in identifying specific functional groups within an FTIR spectra.

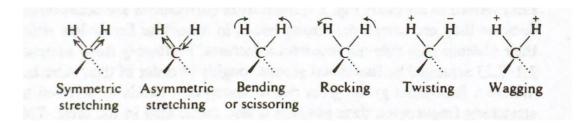


Figure 2.7: Potential bond vibrations present within a CH₂ molecule

Using this technique would allow for quantification of the volatiles being released at different temperatures, as well as a quick snapshot of which temperature changes result in the most variation of chemical compounds being released. The STA-FTIR was chosen as the technique to analyse the drying rate of faecal sludge as it allowed

for simultaneous collection of mass loss and heat flow data, while also analysing the evolved gasses.

2.2 Analysed Materials

2.2.1 Faecal Sludge

Faecal sludge was selected from four different sources to be analysed using the STA-FTIR and UV-Vis-NIR. This allowed for a more complete picture to be formed when drawing conclusions of how it dries and what happens during the drying process.

- Sample 1) Faecal sludge taken from Ventilated Improved Pit latrines (VIP) in the Bester informal settlement, located 25km north of Durban within the eThekwini municipality (Durban, South Africa)
- Sample 2) Faecal Sludge from Urine-Diverting Dry Toilets (UDDT) was collected from KwaMashu, located 20km north of Durban within the eThekwini municipality (Durban, South Africa)
- Sample 3) Faecal Sludge from an anaerobic baffled reactor (ABR) within a decentralised wastewater treatment system (DEWATS) at the Fraser's informal settlement, Durban within the eThekwini municipality (Durban, South Africa)
- Sample 4) Human Faeces (HF) from healthy donors at Cranfield University (United Kingdom)

The faecal sludge was initially sampled during pit emptying campaigns within the eThekwini municipality (Durban metropolis).

Sample 1) The VIP faecal sludge was collected from a vacuum truck once the pit had been emptied. To collect the sludge, water was flushed into the pit for liquefaction before being emptied by a vacuum truck. Due to the addition of water the VIP sludge had a collection moisture content of approximately 95% which was higher than expected. VIP latrines are emptied every 5 years independent of the volume of faecal sludge within the pit and are typically used by one or more families. These latrines can also contain other household trash (58).

Sample 2) The UDDTs are emptied every two years with faecal sludge being collected during manual pit emptying and found to have a moisture content of approximately 70% due to its semi-solid state. Although the collected material was

mainly excreta, there are high amounts of sand which is used as a cover material to help promote dry conditions by absorbing the moisture while also controlling odour and preventing insect infestation. The faecal sludge is accessed from a door at the back of the vault and collected from the top, middle and bottom using a shovel. These latrines will also often contain household trash consisting of anything from old clothes to paper to sanitary materials (99,100).

Sample 3) The ABR faecal sludge was collected during pit emptying from a vacuum truck and was measured to have a moisture content of approximately 88% confirming its very liquid state. ABR's accumulated scum and sludge are emptied every two to three years when the sludge-blankets have reached a height of around 1m. The faecal sludge analysed was collected from an ABR at Frasers informal settlement and was composed of a settling tank followed by six ABR compartments (109).

Sample 4) The fresh faeces were donated at Cranfield University, United Kingdom. The Ethics Committee from the institution granted permission to collect and use the faecal material (ethical clearance CURES/2310/2017). The moisture content of the collected human faeces was calculated to be 60%.

Three of the faecal sludge sources were located in South Africa, where employees of UKZN collected the samples. Each type of faecal sludge was collected within a knotted topped plastic bag within a bucket with a sealed lid, transported to the laboratory at UKZN and stored at 4°C in a cold room to minimise sample degradation. Due to university links these samples were transported to Cranfield University, however before the samples were transported to Cranfield University all faecal sludge was screened to remove any non-homogenous trash larger than 5mm and initial tests were carried out to calculate their general physiochemical properties including chemical oxygen demand (COD), orthophosphate content and pH (Table 2.1). These tests were carried out following the Standard Operating Procedures (SOP) created by the Pollution Research Group which initially were adapted from the Standard Methods for Examination of Water and Wastewater for the use with faecal sludge (110).

The other source was located at Cranfield University where the samples were collected and stored onsite. All samples analysed were then collected from Cranfield University in sealed small vials and UV-Vis slides and transported to Swansea University in a cool box. They were transferred to a fridge and stored at ~4°C (for up to 3 months) to stop any degradation of the faecal sludge. To ensure homogeneity all samples were mixed within their sample container before any smaller samples were taken for analysis.

Within this report the VIP, UDDT and ABR samples are differentiated from the faeces with the former being referred to as 'faecal sludge', and the latter 'fresh faeces'.

Table 2.1: Characteristics of the faecal sludge samples analysed at UKZN (109)

Sample Type	Moisture Content (% mass water/mass wet sample)	COD (mass O ₂ /mg wet sample)	Orthophosphate (mg P/mg wet sample)	рН
VIP	95	0.099	0.003	7.43
UDDT	70	0.409	0.005	8.13
ABR	88	0.094	0.0001	6.93

2.2.2 Simulant Sludge

To create and develop new analytical methods to understand the drying processes that take place when faecal sludge dries it was important to use a material that not only was easily accessible, but also had similar properties to faecal sludge.

A simulant faecal sludge recipe was selected from the University of KwaZulu-Natal. Their pollution research group had developed a recipe from testing three different simulant sludges and altering it until its physical properties were comparable to faecal sludge. The properties tested included, but were not limited to moisture content, chemical oxygen demand, pH, density and heat capacity (111).

The recipe found in Table 2.2 below is from the University of KwaZulu-Natal and was followed to create batches of simulant faecal sludge.

Table 2.2 : Simulant Sludge recipe taken from The University of KwaZulu-Natal – shaded column showing the average quantities used (111)

Ingredients	% Wet	Mass for 1 kg	Mass for 500 g	Mass for 250 g	% Dry Mass
Instant Yeast	7.3	72.80	36.40	18.20	32.49
Water	77.6	776.10	388.05	194.03	-
Psyllium Husks	2.4	24.30	12.15	6.08	10.84
Peanut oil	3.9	38.80	19.40	9.70	17.31
Miso Paste	2.4	24.30	12.15	6.08	10.84
Polyethylene Glycol (PEG)	2.7	27.20	12.15	6.08	12.14
Inorganic Calcium Phosphate	2.4	54.30	12.15	6.08	10.84
Cellulose (half cotton linters, half shredded tissue)	1.2	12.40	6.20	3.10	5.53
Total mass	100.00	1000.20	500.10	250.05	100.00

Due to the presence of instant yeast, new 250g batches were made up before each set of experiments as the material expanded, changed its viscosity, and even started to mould. This led to the quantities of each component slightly varying from batch to batch, but this did not alter the results collected from each set of analysis.

2.3 STA-FTIR Method

A heating programme was developed to create a similar one to what faecal sludge would experience during a traditional drying programme. The 5 different temperatures were chosen as they represented the different thermal conditions in which thermal dryers operate, while avoiding any pyrolyzing of the material. A Perkin Elmer STA (6000) was used to measure the mass change and the energy required within the heating program.

Table 2.3: Experimental conditions during the STA-FTIR experiments

Model of the instrument	Pekin Elmer STA 600 coupled with a Perkin Elmer Spectrum 100		
Start Temperature (°C)	30		
Heating Rate (°C/min)	10		
Final Temperature (°C)	55, 85, 105, 155 and 205		
Carrier Gas	Nitrogen		
Carrier Gas Flow Rate (ml/min)	30		
Mass of Sample (mg)	40 (± 1mg)		

The following programme was then run with X being the final temperature that all samples were heated to (Table 2.3).

- 1. Weigh sample at 40 mg ± 1 mg into ceramic sample pan
- 2. Hold sample at 30 $^{\circ}$ C for 1 minute (this ensured that the sample temperature starts at 30 $^{\circ}$ C)
- 3. Heat sample from 30 $^{\circ}$ C X $^{\circ}$ C with a ramp rate of 10 $^{\circ}$ C/minute
- 4. Hold at X °C for 40 minutes to allow a constant final mass to be reached (80 minutes when heating to 55 °C)

- 5. Heat from X $^{\circ}$ C 450 $^{\circ}$ C with a 50 $^{\circ}$ C ramp rate (to ensure that all pathogens have been removed)
- 6. Hold at 450 °C for 1 minute (to ensure that all pathogens have been removed)

Throughout the whole of the STA programme, the FTIR was collecting spectra to see how the volatiles released from the sample changed over time.

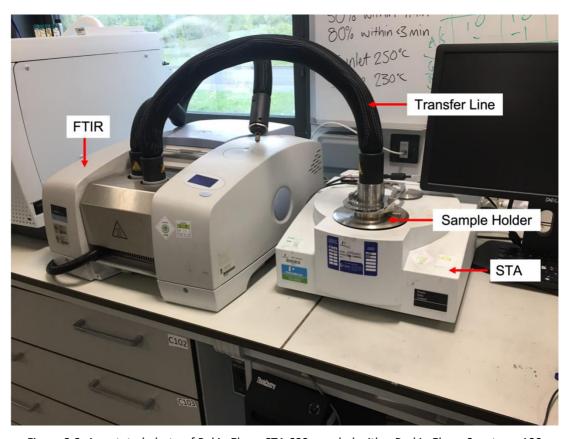


Figure 2.8: Annotated photo of Pekin Elmer STA 600 coupled with a Perkin Elmer Spectrum 100 apparatus

2.4 UV-Vis-NIR Method

To run faecal sludge samples through the UV-Vis-NIR they first needed to be contained within sealed slides using the following method.

- 1) 1mm clear acrylic sheets were cut using a laser cutter into 50 x 50 mm squares to create outer acrylic squares.
- 2) 1-, 2-, 3- and 4-mm acrylic sheets were cut into 50 x 50 mm squares using a laser cutter to create the spacer acrylic squares. This was to allow for 4 different thicknesses of sludge to be analysed to determine whether thickness influenced the sludges ability to dry.
- 3) A 200 mm diameter hole was cut with a laser cutter in the centre of each acrylic spacer.
- 4) Double sided tape was attached to each side or the spacer acrylic squares, and the hole was cut out of the middle.
- 5) Peel off one side of double-sided tape and attach the spacer square to 1 of the outer acrylic squares.
- 6) Fill the hole in the middle with faecal sludge.
- 7) Remove the final piece of double-sided tape and attach a second outer acrylic square.
- 8) Seal around the edges of the slide with a hot glue gun to form a hermetic system and prevent sample degradation (Figure 2.9).

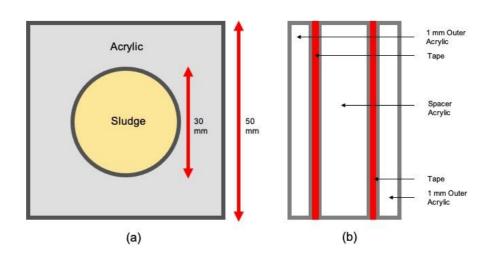


Figure 2.9: Schematic of slides produced to hold faecal and simulant sludge, (a) birds eye view, (b) cross-section (schematic not to scale)

All samples were run through a Perkin Elmer Lambda 750S instrument with a 60 mm integrating sphere. Before each analysis programme the instrument was calibrated to know that 100% transmission was the same as an empty sludge containing slide. The following parameters were followed:

- 1) Data collection range 2500-250 nm
- 2) Data collection interval 5.00 nm
- 3) Scan speed 1196.19 nm/minute
- 4) Slit width fixed at 4.00 nm
- 5) Lamp D2
- 6) Ordinate mode %R/%T

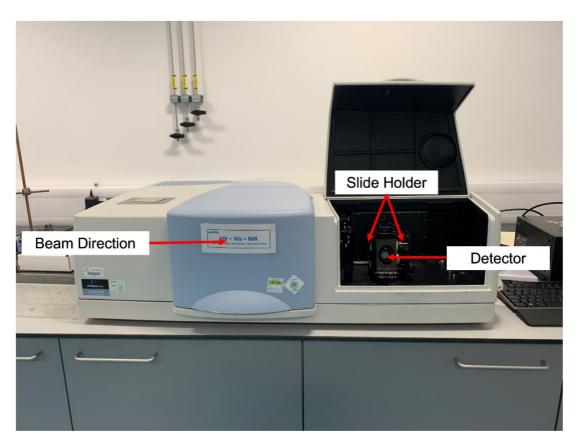


Figure 2.10: Annotated photo of Pekin Elmer Lambda 750S apparatus

Chapter 3 Method Development

This chapter is going to demonstrate how each of the analytical techniques found within the methodology chapter were developed. It will explain how the slides used within the UV-Vis-NIR analysis were created and justify why the chosen STA-FTIR methods were used.

The techniques used throughout this research had not been previously used within the sanitation field so before any faecal sludge analysis could be carried out, experimental methods needed to be developed. When creating new experimental procedures, it is vital to fully appreciate the scope of what is trying to be achieved.

3.1 UV-Vis-NIR Spectroscopy Method Development

Many factors were considered when developing an experimental method for analysing faecal sludge using the UV-Vis-NIR, with arguably the most important being safe containment of the faecal sludge during analysis. It was vital to remember that for these techniques to be replicated in other universities around the world they needed to be simple and inexpensive.

Due to the lack of accessibility of faecal sludge the recipe found in Table 2.2 was used to create an experimental method using UV-Vis-NIR Spectroscopy. This simulant sludge was used to create an appropriate sample containment system along with an appropriate analysis method.

All samples were run through a Perkin Elmer Lambda 750S instrument with a 60 mm integrating sphere. The following parameters were followed:

- 1. Data collection range 2500-250 nm
- 2. Data collection interval 5.00 nm
- 3. Scan speed 1196.19 nm/minute
- 4. Slit width fixed at 4.00 nm
- 5. Lamp D2
- 6. Ordinate mode %R/%T

The most traditional method for UV-Vis-NIR analysis is to use either a disposable plastic or a quartz cuvette. The quartz cuvettes are more expensive but offer a higher precision and unlike the plastic cuvette's they do not tend to absorb at the lower wavelengths. If using a solvent, they will not dissolve unlike the plastic curvette's. As this cuvette was just being used for method development, and no solvents were used in the production of the simulant sludge, the more cost effective disposable plastic cuvette was chosen for the UV-Vis-NIR analysis.

When analysing the results produced by the UV-Vis-NIR the instrument reported a lot of noise (Figure 3.1). This was due to the sample being too thick and opaque and not allowing the instrument beam to pass through the sample which produced a lot of background scattered noise.

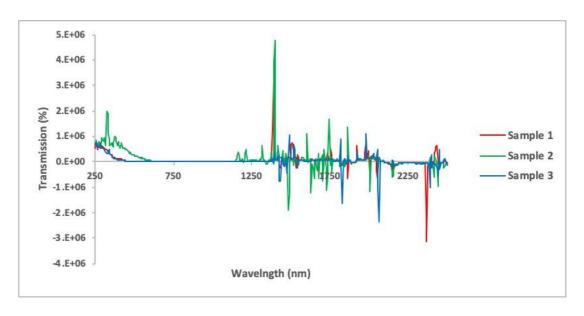


Figure 3.1: UV-Vis-NIR data produced when analysing Simulant Sludge in a Cuvette

To try and combat this problem, it was hypothesised that the samples needed to be thinner in order to allow the instrument beam to pass through simulant sludge.

To try and combat this issue, many different methods of encapsulating the simulant sludge in a slide form were tried.

The two important aspects of the slides that needed to be controlled were the thickness of the slide to ensure that there was uniformity and consistency across all the slides and the size of the hole that the simulant sludge sits in. This needed to be large enough that the UV beam would pass through the sample and not the surrounding slide. It was decided that the optimum slide diameter would be 50 mm² with a 30 mm diameter hole.

The first method attempted used a 2mm double-sided mounting foam tape to control the thickness. This was sandwiched between two sheets of 1mm acrylic to encase the sludge. The slides were then made up using a new batch of simulant sludge and left-over night. When they were re-examined in the morning that the sludge had leaked out of the sides of the slide leaving air pockets (Figure 3.2) and rendering the attempt a failure.

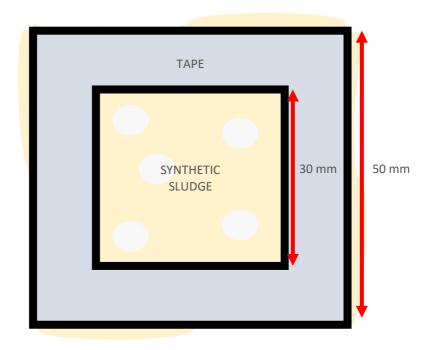


Figure 3.2: Slide production using double sided foam tape, showing slide leaking

UV-Vis-NIR spectra were recorded using these slides (Figure 3.2) with an empty slide used to record the methods base line. Even though the shapes of the UV-Vis-NIR spectra stayed constant the intensities of the spectra varied dramatically across the 3 slides analysed (Figure 3.3). This was due to the slides leaking causing a variation in the thickness of the simulant sludge as well as the formation of air pockets leading to variations within the spectra produced.

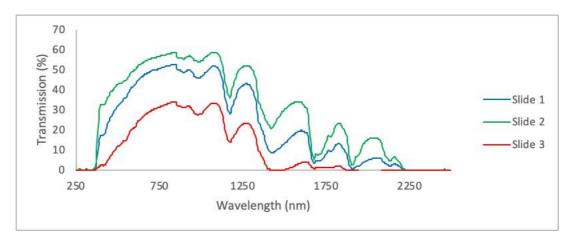


Figure 3.3: Graph produced for a 2 mm thick slide producing using 1 mm acrylic and double-sided foam tape as a spacer

Tape was placed around the edges of the slide to try and seal them and prevent the slides from leaking. This did improve the quality of the data produced with two of

the slides now having the same intensities and one with a higher intensity (Figure 3.4). Unfortunately, after just one hour the slides started to leak, and again air pockets started to form.

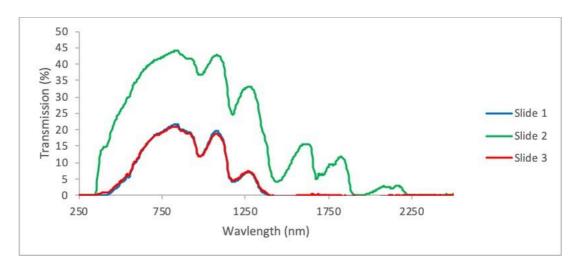


Figure 3.4: UV-Vis-NIR data collected when the slides were sealed with tape around the perimeter

The next method used to stop the simulant sludge leaking from the sides of the slides was to seal them with a hot glue gun (Figure 3.5). Each of the 3 slides recorded an almost identical transmission % reading, which is what would be expected when running 3 identical samples.

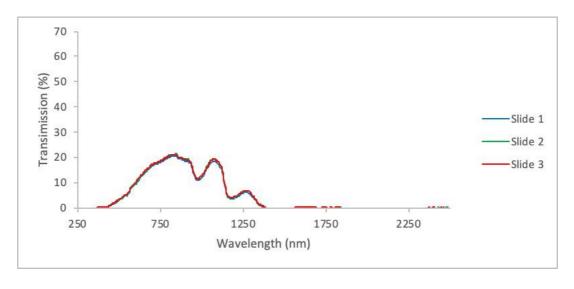


Figure 3.5: Data produced using double sided tape and a hot glue gun to seal the slide using the UV-Vis-NIR

To validate this method, the same slides were run 7 days later (Figure 3.6). On initial inspection they had not leaked but holes had again appeared within the slides. The two hypotheses for this were either that the liquid from the simulant sludge had been absorbed into the foam tape, or that the yeast within the simulant sludge had reacted leaving holes in its place. It was concluded most likely that the liquid from the simulant sludge had been absorbed into the foam tape.

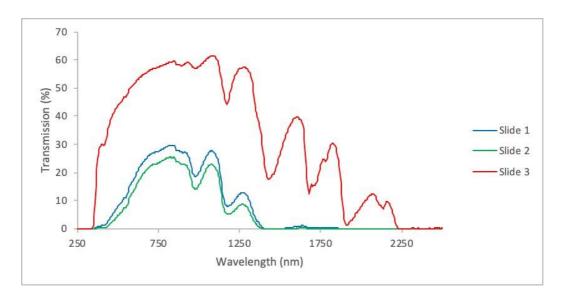


Figure 3.6: Data produced using double sided tape and a hot glue gun to seal the slide using the UV-Vis-NIR after 7 days

The next containment method needed to continue to use the glue to seal the edges of the slides but change the foam tape as this was proving to be the problem.

Acrylic sheets were used to create the space between the two 1 mm outer acrylic sheets. 4 different thicknesses were chosen – 1mm, 2mm, 3mm and 4mm – and a 30mm diameter hole was cut into the middle of each one. The sheets of acrylic were attached with double sided tape, before being sealed with hot glue around the edges to prevent any sample degradation (Figure 3.7). These were left over night to ensure that no sample leaked out of the slides.

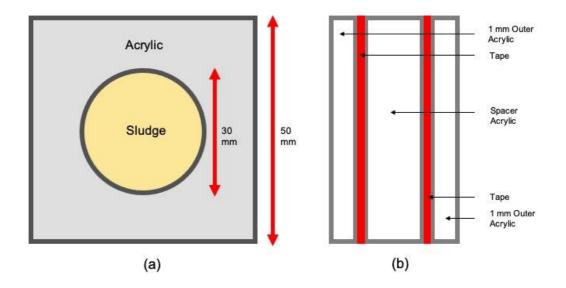


Figure 3.7: Schematic of slides produced to hold faecal and simulant sludge. a) birds eye view, b) cross-section (this schematic is not to scale)

Without the foam tape to allow the expansion of the simulant sludge it had leached between the acrylic sheets and leaked. It was determined that the yeast within the simulant sludge was causing it to expand and that it would be left out of the next batch of simulant sludge produced. The same recipe as found in Table 2.2 was used but with the exclusion of the yeast.

Three slides were made up as previously and sealed with a hot glue gun (slides 1 - 3), with another made up without the hot glue gun seal (slide x). This was to determine if the glue seal was vital to the longevity of the slides. These were left overnight to ensure that there were no obvious signs of sample leakage before UV-Vis-NIR analysis was carried out.

The following day the samples were inspected, and no obvious signs of sample degradation were noticed. The samples were then run through the UV-Vis-NIR and produced relatively consistent intensities across all the slides (Figure 3.8).

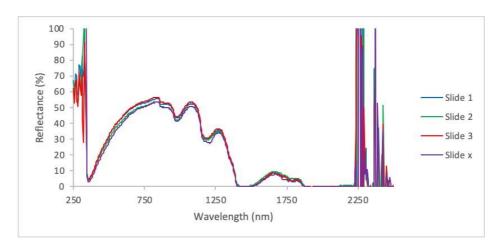


Figure 3.8: Data produced from a slide containing simulant sludge with no yeast using the UV-Vis-NIR. Slides 1-3 are sealed with hot glue and slide X is not sealed with hot glue after 1 day of being produced.

The same slides were then run through the same UV-Vis-NIR programme twelve days later to confirm that the slides were still intact (Figure 3.9). Following initial inspections of the sample slides it could be seen that there had been a small amount of leaking in slide x but slides 1-3 still seemed to be fully sealed.

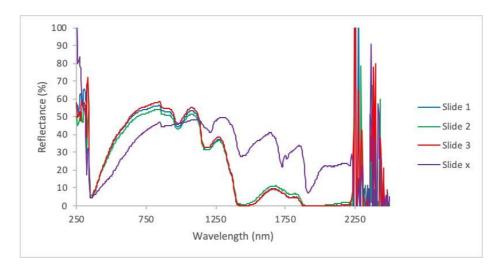


Figure 3.9: Data produced from a slide containing simulant sludge with no yeast using the UV-Vis-NIR. Slides 1-3 are sealed with hot glue and slide X is not sealed with hot glue after 12 days of being produced.

As could be seen by looking at Figure 3.9, the three slides that were sealed with a hot glue gun were still at consistent intensities however, the unsealed slide was noticeably different. This indicated that the slides needed to be sealed with a hot

glue gun to preserve the simulant sludge. This also proved that the yeast within the simulant sludge was causing the sludge to expand.

Now that a successful encapsulation method had been established, all three faecal sludge types along with the fresh faeces were collected from Cranfield University. They were returned to Swansea University and stored at 4 $^{\circ}$ C to prevent sample degradation (Figure 3.10).



	1	2	3	4
a	1mm	2mm	3mm	4mm
	VIP	VIP	VIP	VIP
b	1mm	2mm	3mm	4mm
	UDDT	UDDT	UDDT	UDDT
С	1mm	2mm	3mm	4mm
	ABR	ABR	ABR	ABR
d	1mm	2mm	3mm	4mm
	HF	HF	HF	HF

Figure 3.10: UV-Vis-NIR Slides collected from Cranfield University with table indicating faecal sludge type and thickness

3.2 STA-FTIR Method Development

The simulant sludge produced from the recipe developed by the University of KwaZulu-Natal (Table 2.2) was run through a Perkin Elmer Pyris 1 Thermal Gravimetric Analyser (TGA) using the following programme (Figure 3.11):

- 1) Set system to have a nitrogen gas flow of 30 ml/minute
- 2) Weigh between 10 and 20 mg of simulant sludge into a sample pan
- 3) Heat from 30 150 °C at a ramp rate of 5 °C/minute
- 4) Hold at 150 °C for 0.5 minutes
- 5) Heat from 150 450 °C at 20 °C/minute

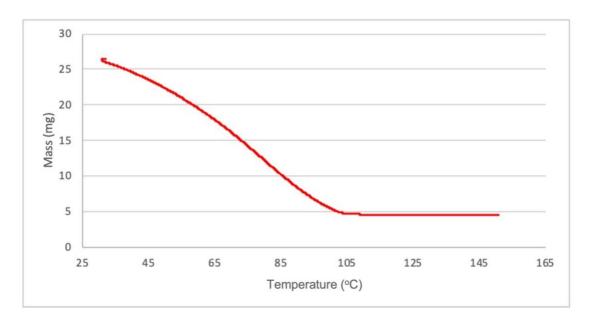


Figure 3.11: TGA data collected using 26.520 mg of simulant sludge

Looking at Figure 3.11 it was decided that there was no need to vary the ramp rate whilst heating.

The simulant sludge was then run through a Perkin Elmer STA 6000 using the following programme which allowed the heat flow curve to be analysed:

- 1) Set the system to have a nitrogen gas flow of 20 ml/minute
- 2) Fill ceramic sample pan 2/3^{rds} full of simulant sludge
- 3) Heat from 30 450 °C at a ramp rate of 10 °C/minute
- 4) Hold at 450 °C for 5 minutes.

An FTIR spectrum was also recorded during this STA run and analysed.

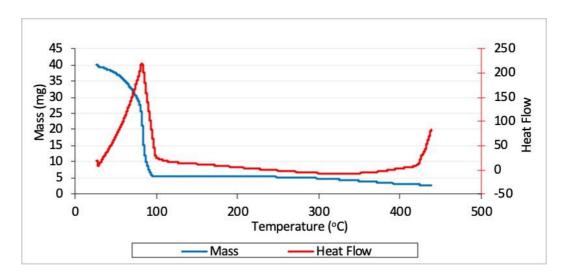


Figure 3.12: STA curve produced when analysing simulant sludge using a 10 $^{\circ}$ C ramp rate from 30 - 450 $^{\circ}$ C

The STA thermograph produced showed a lot of detail in the areas necessary and proved that by 450 °C most of the moisture had been removed from the sample (Figure 3.12).

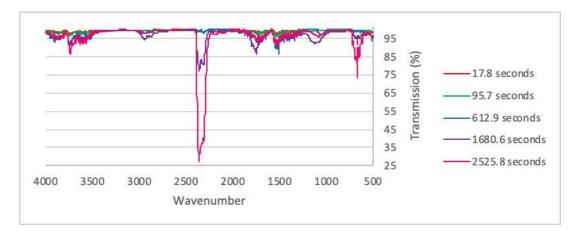


Figure 3.13: FTIR spectrum produced when analysing simulant sludge form 30 - 450 °C with a ramp rate of 10 °C. The FTIR spectrums were collected at intervals throughout the STA heating programme.

The FTIR spectrum produced using the heating programme was easy to read and the peaks could be clearly distinguished (Figure 3.13). The data peaks produced were as expected, and therefore this method was kept in consideration for the final method.

Further ramp rates were tried to achieve the most accurate STA curve. All sample pans were filled 2/3^{rds} full of simulant sludge with a constant nitrogen flow rate of 30 ml/minute.

Trail 1:

- 1) Heat from 30 450 °C at 45 °C/minutes
- 2) Hold at 450 °C for 5 minutes

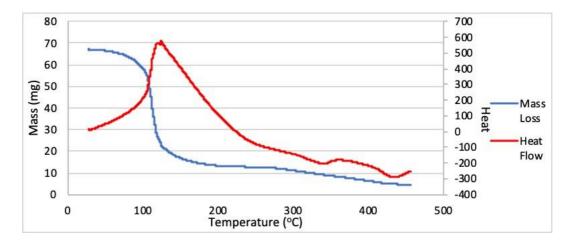


Figure 3.14: STA curve produced using 67.377 mg of simulant sludge with a ramp rate of 45 °C

Trail 2:

- 1) Heat from 30 450 °C at 8 °C/minutes
- 2) Hold at 450 °C for 3 minutes

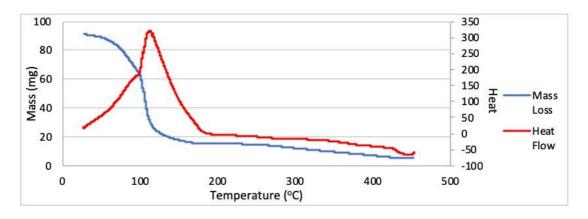


Figure 3.15: STA curve produced using 135.319 mg of simulant sludge with a ramp rate of 8 $\,^{\circ}\text{C/minute}$

Trail 3:

- 3) Heat from 30 450 °C at 50 °C/minutes
- 4) Hold at 450 °C for 3 minutes

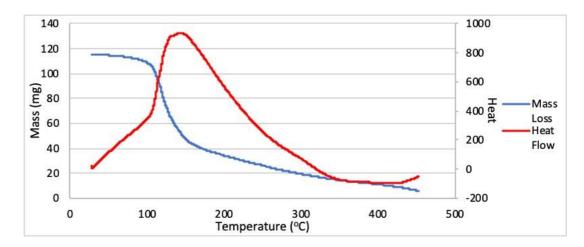


Figure 3.16: STA curve produced using 135.319 mg of simulant sludge with a ramp rate of 50 $\,^{\circ}\text{C/minute}$

Trail 4:

- 3) Heat from 30 260 °C at 5 °C/minutes
- 4) Hold at 260 °C for 1 minute

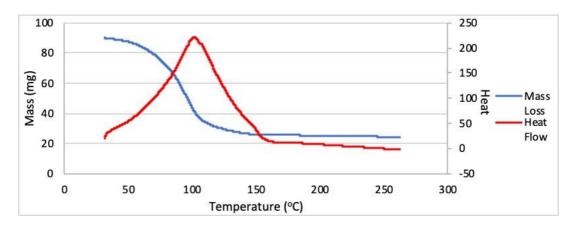


Figure 3.17: STA curve produced using 135.319 mg of simulant sludge with a ramp rate of 5 °C

Trail 5:

- 1) Heat from 30 550 °C at 8 °C/minutes
- 2) Hold at 550 °C for 5 minutes

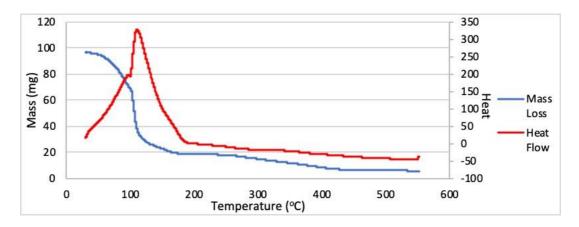


Figure 3.18: STA curve produced using 135.319 mg of simulant sludge with a ramp rate of 8 °C

Analysing Figure 3.14, Figure 3.15, Figure 3.16, Figure 3.17 and Figure 3.18 there is a positive correlation between the temperature and the highest point of heat flow, with a 5 °C/minute ramp rate having the lowest temperature at the highest heat flow, and the highest ramp rate (50 °C/minute) having the highest temperature when the sample reaches its peak heat flow.

The same relationship can be seen when analysing the mass loss curve. At the lower ramp rates (5 °C/minute - Figure 3.17) there is a more gradual end to the curve when reaching its end mass, whereas when comparing it to having a ramp rate of 50 °C/minute it has a much steeper gradient towards the end of the curve as the sample reaches its end mass. This is because at slower ramp rates there is a much higher resolution of the thermographs. This allows for more thermal events to be identified leading to a better understanding of how the faecal sludge will dry on a material level. When the sample is heated to quickly with too high a ramp rate it can cause the thermal events to overlap along with alterations to the drying process due to there being less time to allow for mass transport or mass diffusion within the sample. It was important to not only look at the shape of the thermograph, but also understand why the ramp rates needed to be as quick or slow as they were.

Following on from the data collection a discussion was held between collaborators both in the UK and South Africa to determine the most effective way to analyse faecal sludge using STA-FTIR analysis.

When analysing the faecal sludge there are 5 different temperatures that it needs to reach – 55, 85, 105, 155 and 205 °C from an initial temperature of 30 °C. This was to allow the drying properties to be analysed across a large range of temperature programmes to represent the different temperatures that faecal sludge is currently being heated to within faecal sludge drying processes across the world.

Originally a 100°C a minute ramp rate was suggested as both collaborators were only interested in what happened when it reached its end temperature and not what happened between 30°C and reaching the end temperature. It was pointed out that important thermal events could be identified if the samples were heated at a slower ramp rate. Although it would be interesting to see what would happen when the faecal sludge was held at a specific temperature, it was important for this research to track any thermal events that presented themselves during the heating programme as that could indicated that different drying methods were needed.

It was suggested that if they only needed to know what happened to the faecal sludge at a set temperature it may be simpler to use an alternative method to just measure the mass loss at a set temperature for example an oven rig.

To demonstrate this, 3 different ramp rates were chosen (100°C, 50°C and 10°C) to show not only the differences in the thermal events that could be identified, but also the variation in the FTIR graphs produced.

After collating all the data together, it was presented to the collaborators to strengthen the original argument for slowing the ramp rate. It was explained that at the high ramp rates not only does the instrument not have time to identify any of the important thermal events happening, but also no FTIR peaks appeared. It was concluded that this was because either there was not enough time to allow the volatiles to move through the transfer line to the FTIR or that they were being burnt away by the temperatures that were being reached very quickly during the rapid ramp rate.

It was finally agreed that the ramp rate should be 10 °C as this would allow the collection of FTIR data at a consistent ramp rate to be used across all heat programmes, while also allowing the thermal events and drying patterns to be identified.

The following STA-FTIR method was used as it was the most similar heating programme when compared to a traditional faecal sludge drying programme.

Table 3.1: Experimental conditions during the STA-FTIR experiments

Model of the instrument	Pekin Elmer STA 600 coupled with a Perkin Elmer Spectrum 100
Start Temperature (°C)	30
Heating Rate (°C/min)	10
Final Temperature (°C)	55, 85, 105, 155 and 205
Carrier Gas	Nitrogen
Carrier Gas Flow Rate (ml/min)	30
Mass of Sample (mg)	40 (± 1mg)

The following programme was then run with X being the final temperature that all samples were heated too (Table 2.3).

- 1. Weigh sample at 40 mg ± 1 mg into ceramic sample pan
- 2. Hold sample at 30 °C for 1 minute (this ensured that the sample temperature starts at 30 °C)
- 3. Heat sample from 30 °C X °C with a ramp rate of 10 °C/minute
- 4. Hold at X °C for 40 minutes to allow a constant final mass to be reached (80 minutes when heating to 55 °C)
- 5. Heat from X $^{\circ}$ C 450 $^{\circ}$ C with a 50 $^{\circ}$ C ramp rate (to ensure that all pathogens have been removed)
- 6. Hold at 450 °C for 1 minute (to ensure that all pathogens have been removed)

Throughout the whole of the STA programme, the FTIR was collecting spectra to see how the volatiles released from the sample changed over time.

This was the chosen method to analyse all 4 types of faecal sludge once they had been collected from Cranfield University.

Faecal Sludge samples for STA-FTIR analysis were collected from Cranfield in small vials. This was due to the STA metal lid not forming a complete seal around the sample pan causing moisture to be released from the sample. Due to such a small mass ~40mg being used, so it was decided that any loss in moisture would have a detrimental effect on the analysis and therefore appropriate instruction were given on how to handle the samples once they were returned to Swansea University.

Chapter 4 Results & Discussion 1 STA-FTIR Analysis

Chapter 4 will investigate how each type of faecal sludge analysed dried under differing conditions. Using the STA-FTIR, analytical data was collected to allow for a detailed analysis to be undertaken to provide final moisture contents, drying rates and the energy demand needed to reach set moisture contents for each type of faecal sludge.

Simultaneous thermal analysis was carried out to help form predictions on how faecal sludge dries under differing conditions. It allowed conclusions to be drawn regarding drying properties including optimal drying temperatures, rate of drying, energy consumption along with several other properties.

Drying is a complex process involving simultaneous heat and mass transfer. Due to the physical properties of solids being prone to change during drying, predicting how a material will dry from theoretical principles is often impossible (112).

It is important to fully understand the way in which faecal sludge dries for a few reasons, arguably the most important being cost. Drying is a largely cost intensive process, especially when large amounts of water are being removed (113). It is often important to see if there is a pre-treatment step that can be implemented to remove any moisture by mechanical means such as expression, pressure filtration or by centrifugal means. An issue with this when removing the moisture from faecal sludge is that it will not remove all the pathogens present.

During the drying process wet solids can be grouped into the two following categories according to their drying behaviour.

- 1) Granular or crystalline solids that hold moisture in open pores between particles. During the drying process, the solid is unaffected by the removal of the moisture such that the selection of drying conditions and rate of drying are not critical to the final properties and appearance of the dried product. Materials found within this category can be dried quickly and can achieve very low moisture contents.
- 2) Fibrous, amorphous, and gel-like materials that dissolve moisture or trap moisture in fibres or very fine pores. These materials are significantly affected by moisture removal, often shrinking when dried and swelling when

wetted. With this class of materials, drying in the later stages can be slow. If the surface is caused to dry too rapidly, moisture and temperature gradients can cause checking, warping, case hardening and/or cracking. This means that the selection of drying conditions is a critical factor. Drying to low moisture contents is only possible when using a gas of low humidity.

One of the largest problems faced when drying faecal sludge is its variability in components and therefore structure. While it could be assumed that it would be found within category 1 due to the inconsistency of materials added to toilets such as pit latrines, the faecal sludge which is recovered is therefore also inconsistent leading to a mix in the drying category. This makes it harder to identify the best drying method for faecal sludge (10).

Within Faecal Sludge Management, the drying mechanism removes both water and other volatile compounds from the faecal sludge to produce a solid product ready for reuse (113). To be able to determine how low a moisture content each faecal sludge can reach all mass readings from the STA were converted into moisture content percentages.

Thermal analysis of faecal sludge is an evolving area of research, which is proving to be invaluable to many developers within the faecal sludge management sector. It is allowing more informed decisions to be made when creating containment and treatment processes. When developing a new drying technique, it is important understand how much initial energy is needed to dry the faecal sludge to reach the appropriate moisture content required before it can be used within the treatment system. This can then indicate whether it is necessary to reduce the moisture content to that level, or whether it is worth reducing the amount of energy needed by altering the system to work with a higher initial moisture content.

There is currently limited data available when it comes to understanding how faecal sludge dries. This has led to numerous drying techniques being developed without the information needed to allow it to work as efficiently as possible, often leading to processes using more energy than necessary. This can increase start-up costs and limit the amount of energy left over which can then be used within the local community.

Table 4.1 shows some of the most popular drying technologies currently available or being developed for use with faecal sludge and faeces drying. Most of the technologies rely on convective and contact drying, with only a few centred around radiative drying such as infrared, microwave and solar drying. This proves that it is even more important to fully understand how faecal sludge dries at a material level so that new technologies can be developed with as much information as possible.

Table 4.1: List of faecal sludge and drying technologies (114)

Type of Drying	Technology	Application	Place in the Process	Energy Source	Source
Convective Drying	Belt Dryer	Faecal sludge treatment plant from Tide Technocrats	Drying before a pyrolysis unit for biochar production	Heat from the combustion of the pyrolysis fumes	Tide-Technocrats, 2016 (115)
	Vertical Multi-Tray Dryer	Reinvented "Firelight" toilet from Janicki Industries	Drying before a combustion system	Heat from faecal sludge combustion	SuSana, 2015 (116)
	Rotary Dryer	Faecal sludge treatment plant from Pivot	Final treatment (reuse of the product as biofuel)	Combustion of paperboard	Pivot, 2016 (117)
Contact Drying	Hot surface wall screw conveyer	Faecal sludge treatment plant, "Omniprocessor", operated from Janicki Industries	Drying before a combustion system	Heat from faecal sludge combustion	Villarreal, 2015 (118)
	Heated rotary plate	Reinvented "A Better Toilet" from Research Triangle Institute	Drying before a combustion system	Heat from faecal sludge combustion	RTI, 2013 (119)
Convective, Contact, Radiative Drying	Drying in the top of a fixed bed with a smouldering front at the bottom	Reinvented "Sanitation NoW" from Toronto University	Drying before a smouldering system	Heat from faecal sludge smouldering	Yermán, 2016 (120)
Convective and Radiative	LaDePa machine (convective pre-drying stage, followed by an infrared blet dryer stage)	Treatment of faecal sludge from VIP latrines in eThewini municipality	Final treatment (reuse of the product in agriculture)	Diesel generator providing the hot air and electricity	Harrison and Wilson, 2012 (121); Mirara, 2017 (122)
Radiative	Microwave Dryer	Treatment of faecal sludge in emergency cases	Final Treatment	Microwave radiation generated using electricity	Mawioo et al. 2017 (123)
Solar	Greenhouse Dryer	Faecal sludge treatment plant from Pivot	Pre-drying before the rotary dryer (see above)	Solar Energy	Pivot, 2016 (117)

4.1 Drying Kinetics

Analysing the Moisture Content curves produced, it is easy to see that there is variation between the four different samples analysed (Figure 4.1 and Figure 4.2). The moisture contents were calculated using equations 2 & 3.

moisture
$$(mg) = mass \ (mg) \times initial \ mositure \ contnet$$
 Equation 2

Moisture contnet $(\%wt) = \frac{moisture \ (mg)}{initial \ mass \ (mg)} \times 100$ Equation 3

Analysis of the general trends present when comparing the four different types of faecal material has shown that VIP sludge has the largest moisture content loss, with HF having the smallest moisture content loss as can be seen in the example below showing how the 4 different types of samples dry at 55°C (Figure 4.1).

There is a consistent varied drying rate across each of the samples and temperature programmes, with HF having the shallowest drying curve, and the sludge from the VIP having the steepest drying curves independent of the temperature programme. Figure 4.3 demonstrates a trend forming with the rate of moisture loss constantly increasing until it reaches 155°C, at which point it starts to decrease across all sample types.

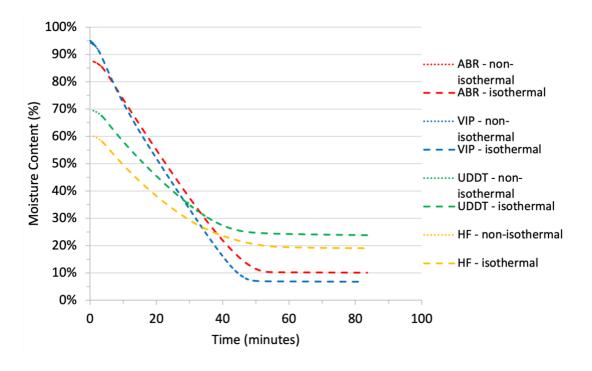


Figure 4.1: Moisture content curves produced when heating samples to 55°C at 10°C/minute and holding them for 80 minutes

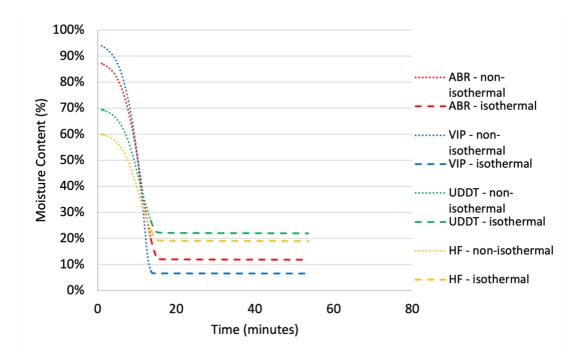


Figure 4.2: Moisture content curves produced when heating samples to 155°C at 10°C/minute and holding them for 40 minutes

Urine appears to play a large role in a faecal sludges ability to dry quickly and to a low moisture content due to both its pH and nutrient content. To correctly draw comparisons between the types of sludge and the way they dry, for this section I will only be using ABR, UDDT and VIP sludge as they were all collected in South Africa under similar conditions and from communities with similar diets. pH is a measurement that is often taken when analysing faecal sludges as it is essential for understanding the water chemistries of the samples (precipitation, corrosion control, neutralisation etc), however it hasn't previously been used to understand the drying properties present (10). Urine has an average pH within the range 4.5 – 7.8 making it slightly acidic (124), which can be seen when looking at the pH's of faecal sludge with VIP and ABR sludge having a much lower than UDDT (Table 4.2). As you decrease the volume of the urine in the faecal sludge, it causes the pH of the sludge to increase in turn making it more difficult to dry. VIP sludge content varies dependent on the users feeding habits and anal cleansing material used (65). Also when being emptied water is added to liquify the sample to ensure that all of the contents of the pit are emptied by vacuum truck. The ABR sludge will contain a limited level of urine due to its pre-treatment step, with UDDT sludge having no

urine present as is the main point of its collection method. This helps to explain why VIP sludge dries the quickest and easiest whereas UDDT sludge dries the slowest (

Table 4.3 and

Table 4.4).

Table 4.2: Faecal Sludge Types analysed with their pH and initial moisture content (%) (109,125)

Type of Faecal Sludge	рН	Initial Moisture Content (%)
VIP	7.43	95
UDDT	8.13	70
ABR	6.93	88
HF	6.6	60

When investigating how long each type of faecal sludge takes to reach a constant moisture content, the higher the temperature the sample is held at, the closer together the end time at which each type of faecal sludge reaches its constant moisture content.

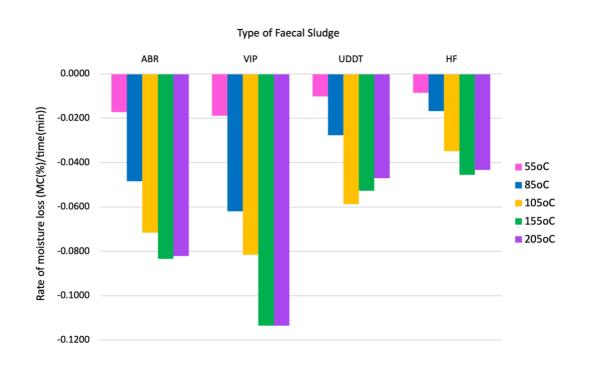


Figure 4.3: Rates of moisture loss of each type of faecal sludge using each temperature programme

Figure 4.4 to Figure 4.7 allow comparisons to be drawn about the different temperature programmes. Figure 4.7 shows that at 55 °C and 85 °C the HF dried slower than any of the other faecal sludge samples. Among the faecal sludge samples UDDT sludge (Figure 4.6) dried slower than the VIP and ABR samples. Above 100 °C they all exhibited a much closer drying pattern; however, the human faeces still dried the slowest.

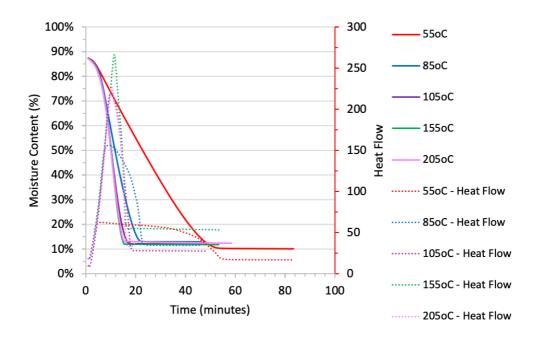


Figure 4.4: Moisture content and Heat flow curves produced for ABR faecal sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

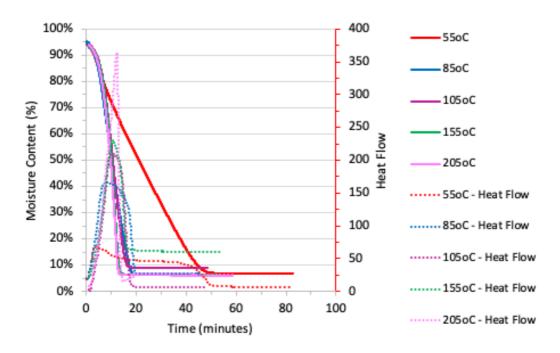


Figure 4.5: Moisture content and Heat flow curves produced for VIP faecal sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

The times in which the samples took to dry also varied depending on the sample type (

Table 4.3).

Table 4.3: Length of time needed for each type of faecal material to dry

Sample Type	Time Range for Sample to Dry (minute)
VIP	13 – 49
UDDT	14 - 64
ABR	15 – 56
HF	16 – 61

It can also be seen that at the higher temperatures the drying times become more comparable across the faecal matter type, as can be seen from

Table 4.4 HF are always the last to reach a constant moisture content.

The Nitrogen and Phosphorus content present within faecal sludge can have a major impact on its ability to dry. It is known that urine contains a high volume of nutrients which is why it is often removed during the collection of the faecal sludge as in the case of UDDT sludge as no pre-treatment is needed to use it as a fertiliser. The down side to this however is that phosphorus has a direct link on a materials dewaterability (126). When the phosphorus content decreases, so does the dewaterability due to increased flocculation of the bound water making it much more difficult to remove (127). This further helps to explain why VIP sludge dries the quickest, easiest and to the lowest moisture content. This is an important discovery in understanding how faecal sludge dries on a material level as depending

on its final usage, removing the urine before its drying process in some cases may be counterproductive.

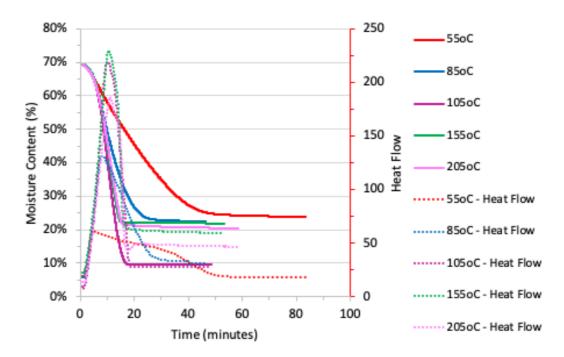


Figure 4.6: Moisture content and Heat flow curves produced for UDDT faecal sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

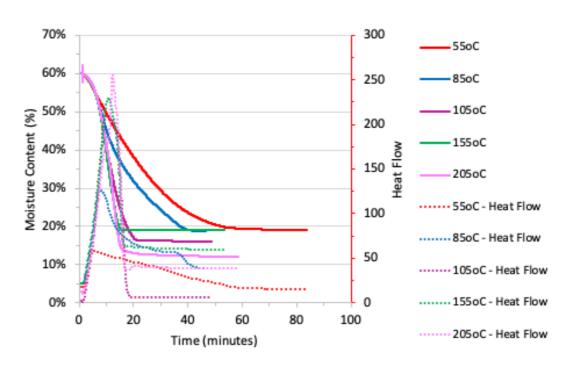


Figure 4.7: Moisture content and Heat flow curves produced for HF faecal sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

Table 4.4: Which faecal sludge types reach their final moisture contents first and last

Temperature	First to Constant Moisture Content	Last to Constant Moisture Content
55 °C	UDDT	HF
85 °C	VIP	HF
105 °C	VIP	HF
155 °C	VIP	HF
205 °C	VIP	HF

When investigating a moisture content curves (Figure 4.4 to Figure 4.7), there are traditionally 3 stages present, the induction, the constant rate and the falling rate period. When considering these 3 stages, all the samples analysed behave in a similar way at each temperature programme (Figure 4.8).

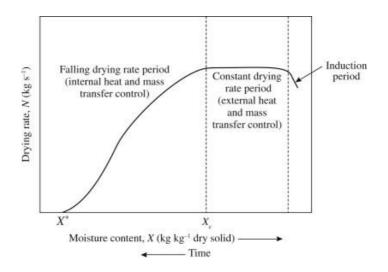


Figure 4.8: Drying rate curve for constant drying conditions (112)

There is an induction period found within each of the curves analysed (Figure 4.4 to Figure 4.7). None of the curves analysed exhibited a constant drying rate period before the falling rate period commenced. The falling rate period was present until the final moisture content was reached, where a final drop can be seen indicating the rate at which the final moisture content was reached (113). The falling rate period shows the time between the first dry area on the materials surface, until the equilibrium moisture content is reached. This is reached when the vapour pressure over the solid is equal to the partial pressure of the vapour in the atmosphere. This equilibrium moisture condition is independent of the drying rate as it is a material property. The equilibrium moisture content is an important value in drying as it represents the limiting moisture content for given conditions of humidity and temperature (128).

The absence of a constant rate period before the falling rate period suggests that there is a limited layer of free surface water on the material that can be easily removed by dewatering. It shows that the initial moisture content of the samples is below the 'critical moisture content'.

This shows that there is a thin boundary layer for drying that favours evaporation from within the material, rather than from the surface. Here, the migration of water to the surface is typically achieved by capillary action and by progressive evaporation-condensation processes that are accompanied by a temperature gradient within the material. The multiple falling rates with no intermittent

constant rate characterises the drying of faecal sludge as a partially wet, partially dry process (78).

Analysing Figure 4.3, the differences between the rate curves produced for each type of faecal sludge can be linked to the varying compactness of the samples. Due to the level of variety between the samples it suggests that the process which removes the moisture within the samples from the interior to the surface could occur via a different phenomenon. This is due to the level of compactness within the sample impacting the moisture transport and diffusion process within the material, leading to an impedance in the migration of water from the inside to the surface of the sample (78).

Figure 4.4 to Figure 4.7 not only shows the different ways that the types of faecal sludge dries, but it also allows the heat flow to be analysed. Variation can be seen in both the shape and intensities of each of the heat flow curves. You can see that VIP faecal sludge has the most intense heat flow curve, while HF have a noticeably lower peak intensity. The trail of the peaks also differs, with VIP and ABR faecal sludge having a much quicker drop off, whereas UDDT and HF have a much shallower drop off. From this it can be beneficial to interpret each temperature programme of a specific faecal sludge type to see how the heat flow curve produced can vary.

The graphs show that when drying ABR sludge at 155°C it produces the largest heat flow peak, while there is then a large drop before the 105 and 205°C peaks can be seen. 55°C has the lowest heat flow peak which would be expected, while also having the broadest peak.

By reducing the temperature, the faecal sludge is heated to, it often allows more thermal events to be identified. When heating each sample type to 55°C an extra drop in heat flow can be seen as the faecal sludge approaches its final moisture content. Although energy will be needed to reach the samples minimum moisture content, as it gets closer the moisture is easier to remove indicating that by this point most of the tightly bound water has been removed.

4.2 Heat Flow

Figure 4.4 to Figure 4.7 not only show the different ways that faecal sludge dries, but also allows the heat flow to be analysed. Variation can be seen in both the shape and intensities of each of the heat flow curves. You can see that VIP faecal sludge has the most intense heat flow curve, while UDDT sludge has noticeably lower peak intensities. By looking at just the heat flow curves, it is hard to get full picture of exactly how much energy is needed to dry each type of faecal sludge. To overcome this, the cumulative heat flow for each curve was calculated to be able to directly compare each temperature programme and faecal sludge type.

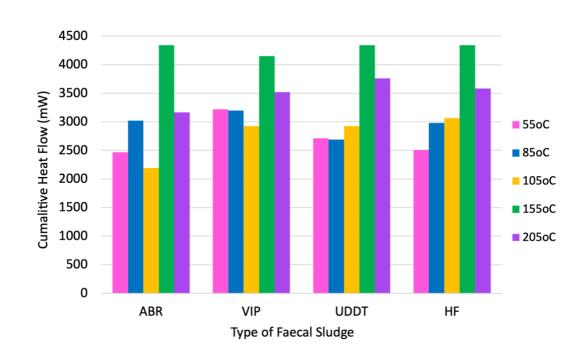


Figure 4.9: Cumulative heat flow for each type of faecal sludge at each temperature programme for the whole temperature programme

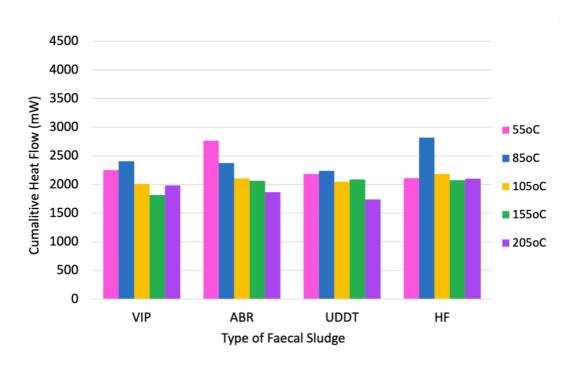


Figure 4.10: Cumulative Heat flow for each type of faecal sludge at each temperature programme until a final moisture content is reached where VIP = $^{\sim}5\%$, ABR = $^{\sim}12\%$, UDDT = $^{\sim}22\%$ and HF = $^{\sim}15\%$

Figure 4.10 displays how much heat flow is needed to reach its final moisture content with every faecal sludge type showing that less energy is needed when heating past 100°C. This is what would be expected since water has a boiling point of 100°C. Each type of faecal sludge uses a similar amount of energy to remove the moisture at each temperature programme with both ABR and UDDT sludge use the least energy when heated at 205°C, while VIP and HF use the least at 155°C. This demonstrates that whilst heating at higher temperatures does decrease the energy demand on the drying system, these temperatures are often hard to reach and require specialist equipment, when heating at lower temperatures such as 55°C these can be reached using technologies such as solar drying which once implemented are virtually free.

This creates an interesting balance between deciding if heating at a higher temperature for a shorter amount of time at a lower energy cost but a higher initial cost to set up the drying system is better than using a lower temperature programme for a longer amount of time but using a virtually free drying system. When using a lower temperature system, it is important to consider the impact on the removal of pathogens. At 55°C it takes around a day of direct heat to reach the

safety zone with respect to pathogen removal, whereas if heating at 205°C it takes only minutes of direct heating. While Figure 4.10 shows the total heat flow needed to reach its final moisture content, Figure 4.9 shows how much heat flow was needed throughout the whole of the drying programme. This demonstrates how much more energy is needed when holding a sample at its final moisture content, leading to wasted energy and time. While using higher temperatures less energy is needed to reach the final moisture contents, when held at these high temperatures a much larger amount of energy is required than at the lower temperatures. If high temperatures are to be used to dry faecal sludge quickly and cheaply it is vital to know exactly how long it takes to reach the final moisture contents otherwise a large amount of energy will be wasted keeping the system at these high temperatures. At lower temperatures, this is far less important as the variation between the total heat flow when the final moisture content is reached, and the end of the heating programme is much smaller leading to much less energy wasted and therefore limited costs.

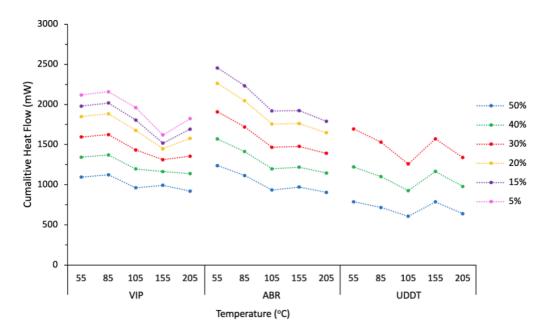


Figure 4.11: The cumulative heat flow required to reach set moisture contents (%) for each type of faecal sludge and temperature programme when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

By analysing Figure 4.11 while the total energy needed to reach a samples final moisture content is comparable across sample types, if you look at the energy

needed to reach specific moisture contents a very different picture can be seen. This is due to the varying initial moisture contents across the different types of faecal sludge (Table 4.2).

When interpreting the energy demands for each type of sludge to reach a specific moisture content it is important to remember that each started at a different initial moisture contents (Table 4.2). Although UDDT requires the least amount of energy to reach a 50% moisture content, it has only been reduced by 20%, whereas VIP sludge has needed to remove more than double the amount of moisture but has only used a fraction more energy to do so. Figure 4.11 also shows that while the trends within each type of faecal sludge are the same, independent of the moisture content it is trying to reach, the variations between each temperature programme increases as the moisture content reached decreases. This shows that if the sludge only needs to reach a moisture content of around 40-50%, then the temperature programme isn't as important, but if the VIP sludge needs reducing to a moisture content of 10% it is important to note that if heated at 155°C it uses over 500mW less energy.

This is a vital comparison to draw as often faecal sludge treatment processes require the sludge to be at specific moisture content before their treatment process will work. Understanding the different energy demands that each type of faecal sludge requires to reach specific moisture contents is of paramount importance when choosing the most compatible treatment process.

4.3 Energy Kinetics

Within FSM heat flow curves (mW) are often converted into energy (kJ/kg) curves to allow the total amount of energy needed to reduce the moisture contents of each type of faecal sludge to the required volume (Equation 4).

$$Heat of drying = \frac{\delta time(s) \times \delta heat flow(mW)}{\delta mass(mg)}$$
 Equation 4

Energy kinetics indicates the energy input required to dry the faecal matter. Traditionally the heat of drying is usually approximated to the latent heat of water vaporisation, however when analysing the energy kinetics of faecal sludge these assumptions are not always correct. Within faecal matter the moisture is tightly bound within the solid structures, meaning that supplementary energy would have to be required to break these bonds in addition to the traditional water-water hydrogen bonds. When drying faecal sludge, the mass of the sample is constantly changing with the loss of different volatile compounds which is discussed further on in this chapter. By calculating the latent heat assumptions are made that the mass or volume of the sample will be kept constant, however within faecal sludge there are far too many variables to be able to make this claim. By converting to energy (kJ/kg) assumptions are being made that are unfounded as it is not a pure water sample. It would give a close approximation given the volume of water found within faecal sludge, but the impurities will cause suppression of the removal of water. Faecal sludge is a very complex material with water in lots of different forms, and by comparing it to the latent heat of water vaporisation it is being measured as a dynamic system. Eventually they would all reach the same moisture content at one point, there will always be different masses across each temperature programme and faecal sludge type making direct comparisons very difficult. Comparing the energy demands using cumulative heat flow is a much simpler and more appropriate method to use.

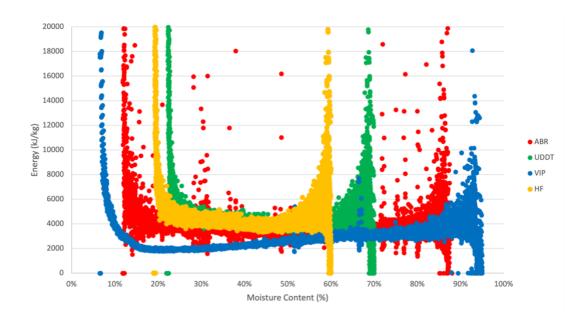


Figure 4.12: Heat of drying profiles for each type of faecal matter when heated to 155°C at 10° C/minute before being held for 40 minutes

As can be seen in Figure 4.12, the VIP faecal sludge requires the least amount of energy to reach its final moisture content. This is not what was expected because it reaches the lowest moisture content out of any of the faecal sludge types analysed and does not agree with the cumulative heat flow curves found in Figure 4.11. The heat of drying varies between 2000 and 5000 kJ/kg across the faecal matter samples analysed. This data has been compared to data collected at the University of KwaZulu-Natal (UKZN) in Durban, South Africa and can be seen to be comparable (Table 4.5). By analysing the amount of energy required to dry faecal material it can help to indicate which type of faecal sludge is right for which treatment processes.

Table 4.5: Average Energy readings collected at UKZN using a TGA-DTA (114)

Drying Temperature (°C)	Energy (kJ/kg)		
	ABR	UDDT	VIP
50	4000 ± 500	3000 ± 0	2600 ± 200
100	4800 ± 400	5700 ± 600	4100 ± 500
150	2400 ± 300	6100 ± 100	3700 ± 100
200	4900 ± 600	5600 ± 0	4300 ± 900

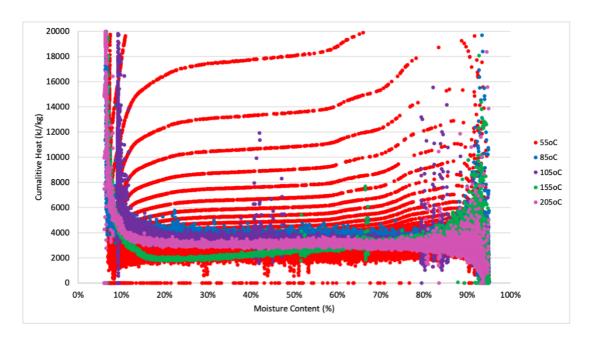


Figure 4.13: Heat of Drying of VIP sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

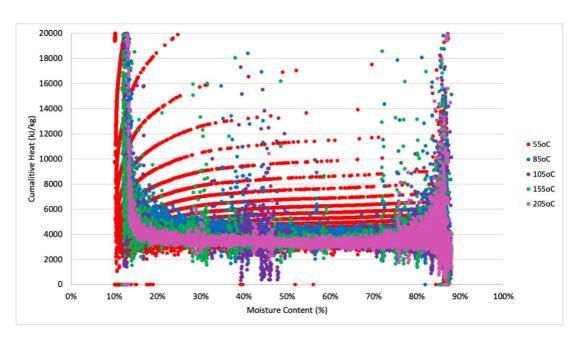


Figure 4.14: Heat of Drying of ABR sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

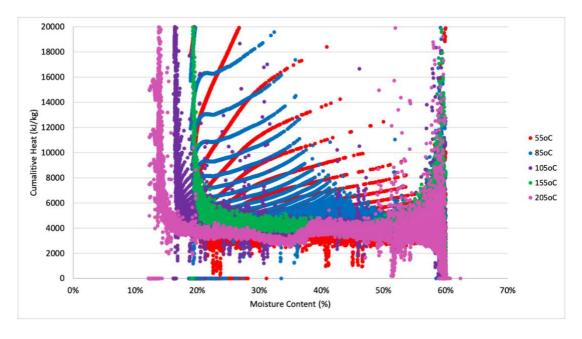


Figure 4.15: Heat of Drying of HF sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

Human faeces require the largest amount of energy to reach its final moisture content and in comparison, to the VIP sludge, has the lowest initial moisture content. It did not have any extra water added to the sample during collection, confirming the link between the amount of energy needed to dry the faecal material, and how much of the water is bound into the solids.

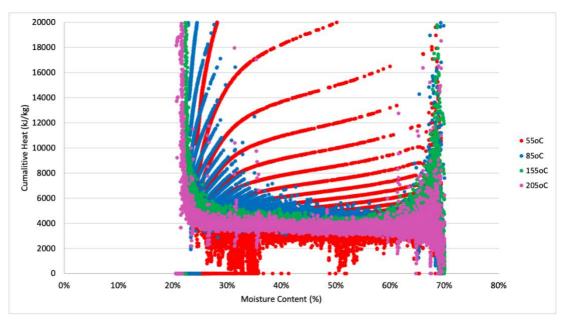


Figure 4.16: Heat of Drying of UDDT sludge when heated to a set temperature at 10°C/minute before being held for 40 minutes (80 minutes when heated to 55°C)

By analysing the different types of faecal sludge, the energy output for each one can be identified. The latent heat of water vaporisation is usually approximated to 2260 kJ/kg but it can be seen in Figure 4.13 to Figure 4.16 that the amount of energy required is noticeably higher than this. The most similar energy profile to what would usually be approximated was found to be the VIP sludge. VIP sludge has an extremely high initial moisture content due to water being added to the sample upon retrieval. This could explain why the VIP sludge has a similar latent heat of water vaporisation value to what is approximated (Figure 4.13), however a variation of between 2000-3000 kJ/kg is not a close enough comparison to draw any conclusions, and confirms that calculating the energy consumption using this method is not the most appropriate for the drying system analysed.

4.4 Drying Emissions

Faecal material is known for its emission of unpleasant odours which are caused by volatile organic or inorganic compounds. During drying the effect of heat provokes a substantial increase in the volatilisation of the olfactory compounds which causes these to leave within the evaporated moisture in the exhaust air stream. During the drying process these olfactory are released in varying concentrations causing the bad odours to intensify. Aside from the odours, these olfaction's can also contain compounds that can potentially be harmful to the environment and human health. If these are identified the exhaust fumes would need to be contained and collected for safe disposal.

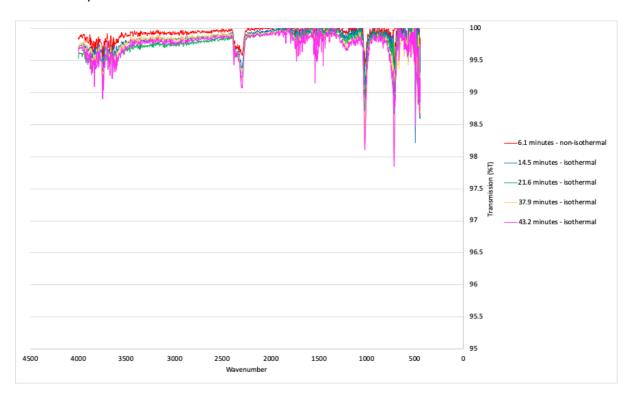


Figure 4.17: FTIR spectra produced when ABR faecal sludge is heated to 85 °C at 10°C/minute before being held for 40 minutes

Table 4.6: Functional Groups present when analysing faecal sludge using a Perkin Elmer FTIR

Functional Group	Band Frequency (cm ⁻¹)	
H ₂ O (Vapour)	4000 – 3400	
CO ₂	2340 – 2360	
CH₂/CH₃ deformations	1470 – 1430	
CH ₂ rocking	~720	
NH ₂	1650 - 1560	

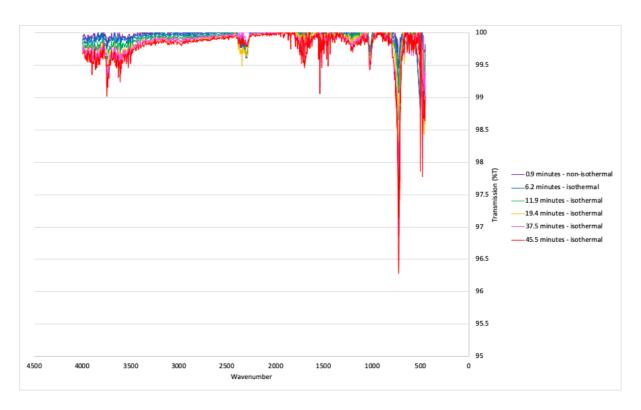


Figure 4.18: FTIR spectra produced when UDDT faecal sludge is heated 85 $^{\circ}$ C at 10 $^{\circ}$ C/minute before being held for 40 minutes

When analysing the FTIR spectra there is some variation across the samples analysed, however they all exhibit the same peaks (Figure 4.17 and Figure 4.18). This is also demonstrated within the Book Addendum to the Methods of Faecal Sludge Analysis (129) and the Final Technical Report submitted to the Bill & Melinda

Gates Foundation (114). Arguably the most important peak present is the water vapour peak. This is present in every sample analysed and typically increases as the heating programme progresses until the higher temperatures are reached when it starts to decrease in intensity during the heating programme. The CO_2 peak increases across all samples excluding the UDDT sludge at 85° C. UDDT faecal sludge sees an increase in peak intensity up to a point where this starts to decrease. This trend can be seen across all UDDT spectra's, independent of the temperature that the samples are heated too. This could lead to the conclusion that there is not as much CO_2 present, or it is not as tightly bound within the makeup of UDDT faecal sludge.

4.5 Summary

Using the STA-FTIR it was possible to identify the different drying rates found within each type of faecal sludge. The VIP consistently dried the quickest and with the lowest energy demand at each temperature used, whereas the UDDT sludge had the highest energy demand and the slowest drying rate. It was also identified that the removal of urine hindered the drying process due to altering the faecal sludges pH and nutrient contents within faecal sludge.

Chapter 5 Results & Discussion 2 UV-Vis-Analysis

Chapter 5 will understand the way in which faecal sludge interacts with the suns radiation. By undertaking UV-Vis-NIR analysis it is possible to determine the amount of suns radiation that is absorbed and reflected off the surface of the faecal sludge.

Chapter 4 investigated how faecal sludge dried when heated to different temperatures (55, 85, 105, 155 and 205°C), but this chapter is going to identify whether using solar is an efficient drying method and if so, what the best region of the solar spectrum to dry faecal sludge is using UV-Vis-NIR spectroscopy.

Spectroscopy is one of the most widely used Analytical techniques which can be used for a wide range of analysis. For the analysis of Faecal Sludge, UV-Vis-NIR was chosen allowing data from the whole solar spectrum to be collected at the same time.

It is important to understand how faecal sludge dries within the solar spectrum as drying beds are currently one of the most widely used drying system within the developing world. Drying beds are used within faecal sludge management systems as they have a low capital cost, low energy consumption, minimal to no chemical consumption, there is virtually no operator skill or attention needed and they allow for a large variability in the type of faecal sludge used (130). They are also popular within developing nations, as these countries typically have access to large areas of land and warmer climates than other areas of the world making it a perfect drying system. By fully understanding how different types of faecal sludge dry using this common method it can help direct how they use their drying beds.

Solar Thermal Drying is another technique that is being investigated as a potential for faecal sludge drying. It is being considered as a more cost-effective solution, as even though there is a higher capital cost, there are lower drying times, they are more thermally efficient and there is the possibility of automatization.

Understanding how faecal sludge dries using this heat source is vital to understand how to best utilise the technique within FSM systems.

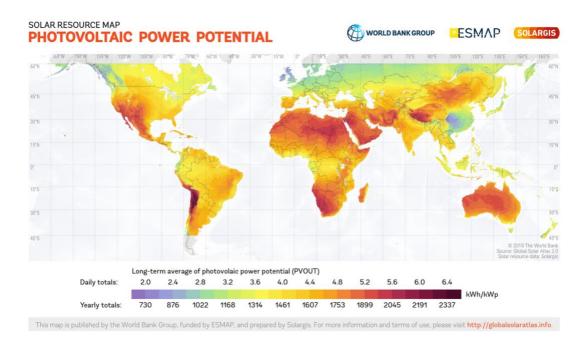


Figure 5.1: Solar Map of the World as of 2019

Many developing countries facing a lack of sanitation options are situated within the solar belt with the highest solar irradiances (Figure 5.1). Understanding which parts of the solar spectrum interact with the earth and the faecal sludge is important when discussing the longevity of these drying systems. The solar spectrum consists of an average of 44.7% visible radiation, 6.6% ultraviolet radiation and 48.7% infrared radiation (131). While these values are good indicators to the radiation found within the solar spectrum, it is important to note that the solar spectrum does change throughout the day and with location so these values cannot be assumed precise (132). By analysing the whole of the solar spectrum it is possible to determine which regions provide the most beneficial drying conditions to help influence newly developed technologies. The analysis found within this chapter was included within the Addendum to the Methods of Faecal Sludge

Analysis which aimed to provide data and knowledge about the behaviour of faecal sludge, which in turn is expected to contribute to the development of guidelines of best practices (129).

5.1 Total Transmission

To collect total transmission data from the 4 different types of faecal material analysed, the slides needed to be positioned in-front of the integrating sphere (Figure 5.2). This allows the instruments detector to calculate the amount of light that is able to transmit both as direct and diffuse transmission (133) through the faecal sludge containing slide at each of the measured wavelengths.

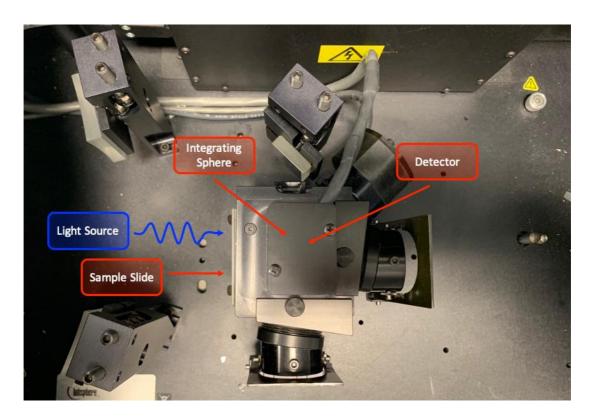


Figure 5.2: UV-Vis-NIR instrument set-up for collecting transmission data

Analysing the transmission data produced by the UV-Vis-NIR shows how much light can pass through the faecal sludge at different wavelengths and thicknesses, which indicates how much light is reflected from the surface.

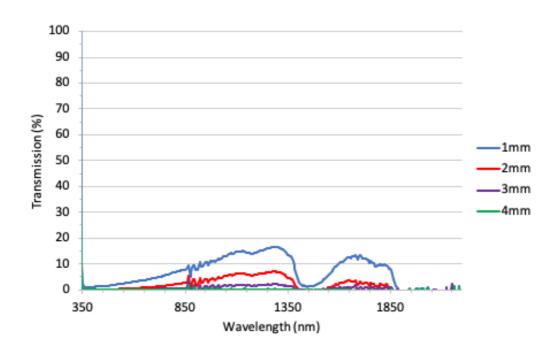


Figure 5.3: Transmission spectra produced by the UV-Vis-NIR for VIP faecal sludge

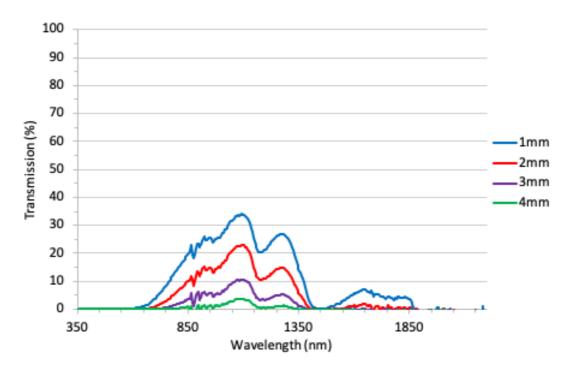


Figure 5.4: Transmission spectra produced by the UV-Vis-NIR for Human Faeces $\,$

Figure 5.3 and Figure 5.4 show a low level of transmission in the NIR region around 1400-1500 nm, and again above 1900 nm in both VIP sludge and HF indicating that there is a high level of absorbance within these regions. At all wavelengths, the thicker the slide becomes the lower the percentage transmission. By 3 to 4 mm in VIP sludge there is no significant transmission indicating that all available light has

been absorbed or reflected from the surface. A low level of transmission would be expected due to the appearance of the HF however, the VIP sludge has a higher transmission peak than predicted due to its dark appearance (Figure 3.10). It was assumed that it would behave more like UDDT and ABR sludge as they all appear dark in colour (Figure 3.10). Human faeces has a much lighter coloured appearance indicating that less light would be absorbed, which correlates with what was observed using the UV-Vis-NIR (Figure 5.4). Both the UDDT and ABR faecal sludge showed no significant transmission at any wavelength indicating that most of the available light had been absorbed at all thicknesses measured (Figure 5.5).

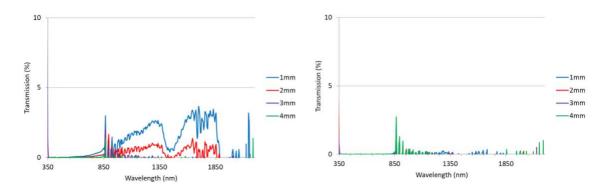


Figure 5.5: Transmission spectra produced by the UV-Vis-NIR for (a) ABR and (b) UDDT sludge

The way that light passes through a sample is not only due to its perceived colour but also its material properties. Although each of the samples were held within acrylic slides, there was still variation present within the sludge. Although VIP sludge is one of the darkest faecal sludges analysed, it has the highest moisture content (95%) meaning it is a much less opaque sample and therefore allows light to pass through more easily. As can be seen in Figure 3.10, each of the slides had a uniform colour across each slide thickness proving that the difference in spectra at each thickness is only dependent on the depth of penetration and not the colour of the sludge. This helps to explain why it had a noticeably higher transmission rate than ABR and UDDT sludge given its similarities in appearance.

5.2 Diffuse & Specular Reflectance

Reflection from a surface is comprised of both specular and diffuse components which are both important when discussing the ability for faecal sludge to dry under differing radiative conditions.

The specular reflection is a mirror-like reflection, whereas the diffuse reflection arises from multiple surface reflections on a rough surface (134). By analysing the specular reflectance of faecal sludge, it is possible to determine how smooth the samples are, whereas the diffuse reflection shows the samples roughness.

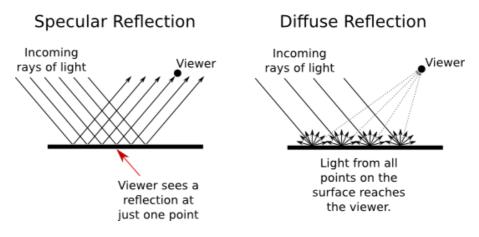


Figure 5.6: Image showing how light reflects off both smooth and rough surfaces

To collect diffuse reflectance data from the 4 different types of faecal sludge analysed, the slides needed to be positioned at the back of the integrating sphere parallel with the white beam blocker (Figure 5.7). This allows the instruments detector to calculate the amount of incident light that is reflected in all different directions off the faecal sludge containing slide at each of the measured wavelengths (133).

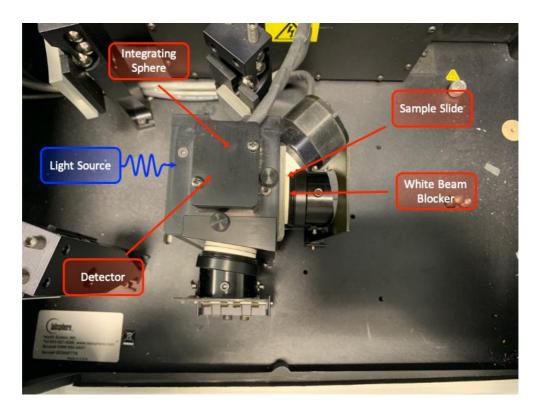


Figure 5.7: UV-Vis-NIR instrument set-up for collecting diffuse reflectance data

The Specular reflectance percentages were calculated mathematically (Equation 5). Specular reflectance refers to the part of the incident beam reflected at the same angle as the angle of incidence (133).

Specular Reflectance = Total Reflectance - Diffuse Reflectance Equation 5

By understanding both the diffuse and specular spectra's, conclusions can be drawn about each type of faecal sludge's ability to dry along with its surface roughness.

A surface built from a non-absorbing powder such as plaster, or a polycrystalline material such as white marble reflect light diffusely with great efficiency. This leads to them having a very low absorbance and makes them an unfit substance for drying. Most substances produce both diffuse and specular reflectance, however matt substances reflect light so diffusely that no specular reflectance is seen (Figure 5.8) (135).

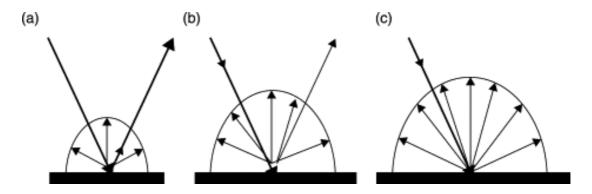


Figure 5.8: Reflection from different types of surface a) gloss b) semi-gloss and c) matt (135)

Diffuse reflection from solids is not always due to the surface roughness, most of the light reflected is contributed by scattering light beneath the surface. For example, if you took white marble and polished it, it would never turn into a mirror leading to both diffuse and specular reflectance.

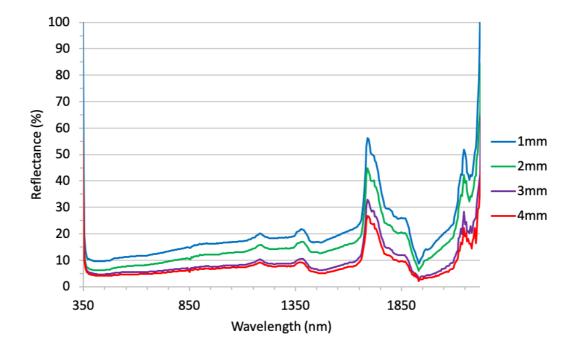


Figure 5.9: Diffuse Reflectance spectra produced by the UV-Vis-NIR for VIP sludge

The diffuse reflectance graphs for VIP sludge allows an assumption to made about the roughness of its surface. Its high percentage reflectance shows that VIP sludge has a rough surface due to its non-homogeneous texture leading to having a relatively high surface area. Figure 5.9 shows a high diffuse reflectance percentage within the near infrared region indicating that it would dry the slowest using this heat source.

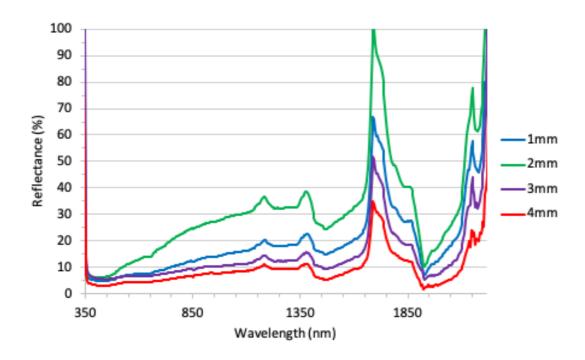


Figure 5.10: Diffuse Reflectance spectra produced by the UV-Vis-NIR for ABR sludge

Analysing the ABR diffuse reflectance graph the 2mm peak is noticeably higher than that of the 1mm. This does not fit the usual pattern seen and is thought to be due to air pockets forming within the sample slides. Like VIP sludge, the diffuse reflectance percentage peaks can be seen within the NIR region of the spectra, however they are all at a higher intensity than VIP showing that there would be less absorbance within this area and would be much harder to dry using this heat source.

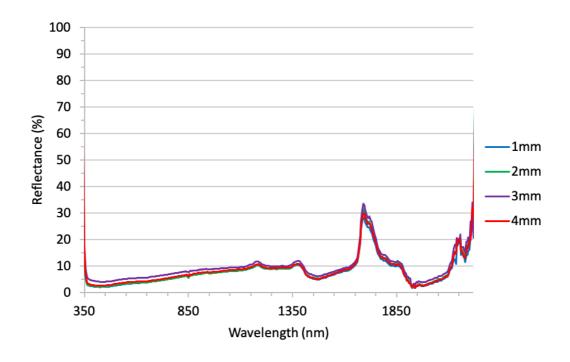


Figure 5.11: Diffuse Reflectance spectra produced by the UV-Vis-NIR for UDDT sludge

Both UDDT and ABR faecal sludge produced similar transmission spectra's (Figure 5.5) showing no significant transmission indicating that all available light was either absorbed or reflected. Analysing the diffuse reflectance graphs there is a noticeable difference between the two spectra's (Figure 5.10 and Figure 5.11), the specular reflectance graphs show little variation (Figure 5.12). This indicates that ABR sludge has a rougher consistency than the UDDT sludge causing more light to be reflected of the surface at differing angles to the angle of incidence. As the sample is rougher it will have a larger surface area which could lead to it drying at a quicker rate than UDDT sludge (Figure 3.10).

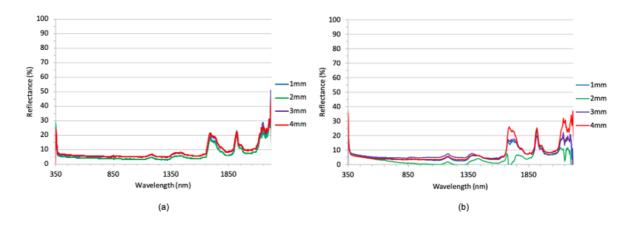


Figure 5.12: Specular Reflectance spectra produced by the UV-Vis-NIR for (a) UDDT & (b) ABR sludge

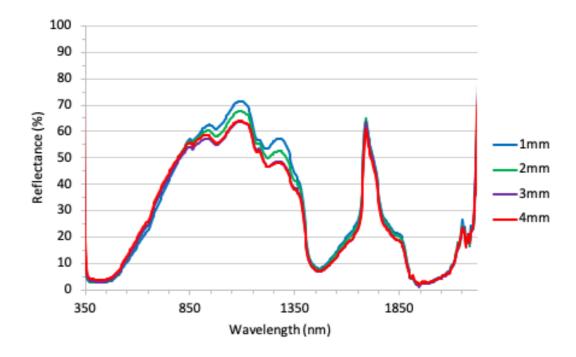


Figure 5.13: Diffuse Reflectance spectra produced by the UV-Vis-NIR for human faeces

5.3 Total Reflectance

To collect total reflectance data for the 4 different types of faecal material analysed, the slides needed to be positioned at the back of the integrating sphere, at an 8-degree angle with the white beam blocker (Figure 5.7). This allows the instruments detector to calculate both the diffuse and specular reflectance of the faecal sludge containing slide at each of the measured wavelengths (133).

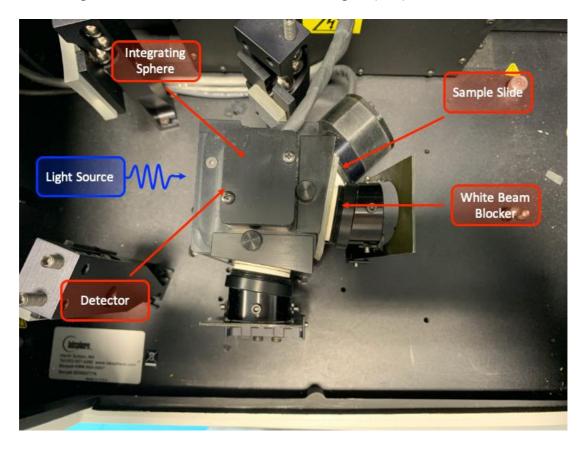


Figure 5.14: UV-Vis-NIR instrument set-up for collecting total reflectance data

Converting total reflectance into total solar reflectance by applying a solar rating (ASTM E903) allows the reflectance data to be calibrated with respect to the energy available at each wavelength from the Sun radiating onto the Earth surface. The total solar reflectance can be calculated using equation 6.

$$\%TSR = \frac{\int (\%R \times I \times \delta \lambda)}{\int I \times \delta \lambda}$$
 Equation 6

Where:

%R = Total Reflectance %

I = Solar Irradiance

 $\delta\lambda$ = wavelength interval of integration

Total solar reflectance measurements are often used in the paint industry to help determine how much of the sun's radiation hits the surface of the material and reflects off and how much of it was absorbed into the material. Total solar reflectance is always expressed as a percentage with white coatings having a total solar reflectance value of approximately 75% and black coatings having a total solar reflectance value of about 3.5% (43). This process can be mirrored into the FSM field when drying faecal sludge using the Sun in drying beds. When using these drying methods, it is important to understand how much of the sun's radiation is being reflected off the material and how much is being absorbed. From this data combined with the transmission data it is also possible to hypothesise how deep the radiation will penetrate, in turn leading to a fuller understanding on how faecal sludge dries in the Sun, and how often if at all it will need turning or raking to ensure that it fully dries.

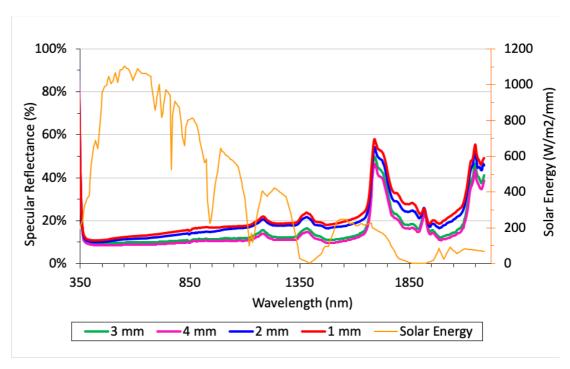


Figure 5.15: Total Solar Reflectance spectra with a ASTM E903 solar weighting applied produced by the UV-Vis-NIR for VIP sludge

Figure 5.15 shows the total solar reflectance spectra for VIP sludge. As expected for VIP sludge, there is a low reflectance in the visible and near-infrared region (~10 and 15% respectively) showing that it is highly absorbing in this region due to its

dark colour. The average total solar reflectance of ~13% confirms that using sunlight as a drying method would be a good choice (Figure 5.15 and Figure 5.16).

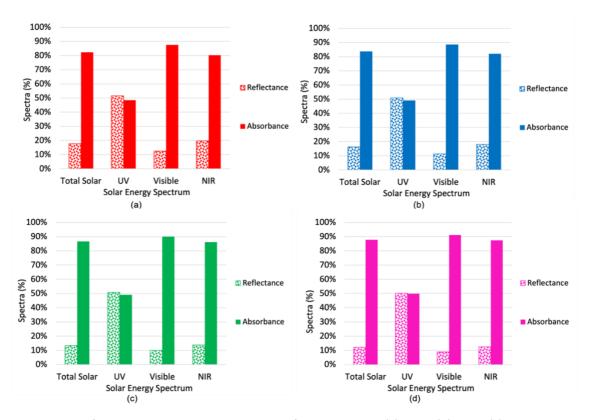


Figure 5.16: Reflectance & Absorbance percentages for VIP sludge at (a) 1mm, (b) 2mm, (c) 3mm and (d) 4mm

When analysing Figure 5.16, a direct comparison can be drawn between the materials ability to absorb and reflect at different wavelengths of the electromagnetic spectrum.

The higher the absorbance seen, the easier and quicker the faecal sludge will dry at a specific wavelength. The absorbance is very high (~87%) across each sample thickness. Within the UV part of the electromagnetic spectrum the reflectance and absorbance are almost identical, indicating that if drying programmes were to be developed focusing on a specific region of the electromagnetic spectrum, the UV region would not be appropriate. Instead using either the visible or NIR regions of the electromagnetic spectrum would allow for a much quicker and more reliable drying source.

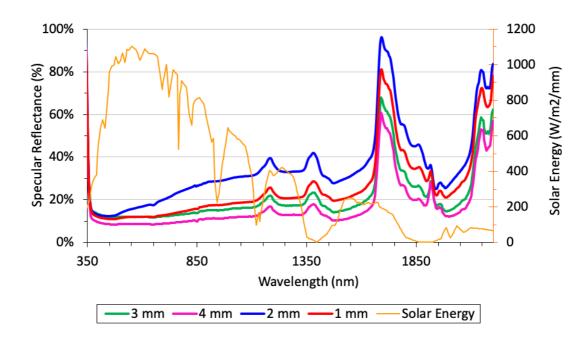


Figure 5.17: Total Solar Reflectance spectra with a ASTM E903 solar weighting applied produced by the UV-Vis-NIR for ABR sludge

ABR faecal sludge has a low reflectance in the visible (~12%) and near infra-red regions (~20%) showing that it is highly absorbing in this region, again as expected due to its dark colour. The average solar total solar reflectance shows that using sunlight as a drying method is a good choice (Figure 5.17 and Figure 5.18).

As with VIP sludge, the absorbance within the whole of the solar spectrum is high (~85%), indicating that drying using this source would be reliable (Figure 5.18). However, unlike with VIP sludge the absorbance values for the UV region of the electromagnetic spectrum are lower than the reflectance values for ABR sludge. While the total solar values show that drying in the open air is a good system, using UV radiation as a drying source should be avoided.

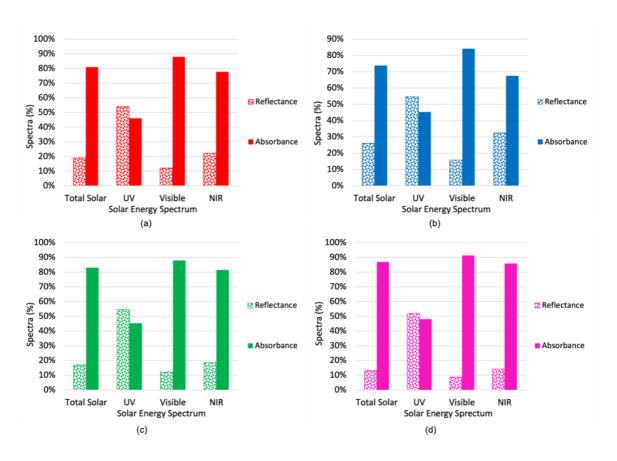


Figure 5.18: Reflectance & Absorbance percentages for ABR sludge at (a) 1mm, (b) 2mm, (c) 3mm and (d) 4mm

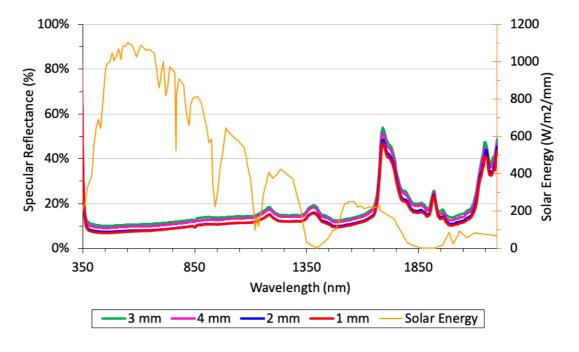


Figure 5.19: Total Solar Reflectance spectra with a ASTM E903 solar weighting applied produced by the UV-Vis-NIR for UDDT sludge

Figure 5.19 shows a low reflectance in both the visible (8 %) and near infra-red (14 %) regions indicating that it is highly absorbing in these areas. Also, its total solar reflectance percentage (13 %) is also low showing that using sunlight as a drying method is a good choice (Figure 5.19 and Figure 5.20).

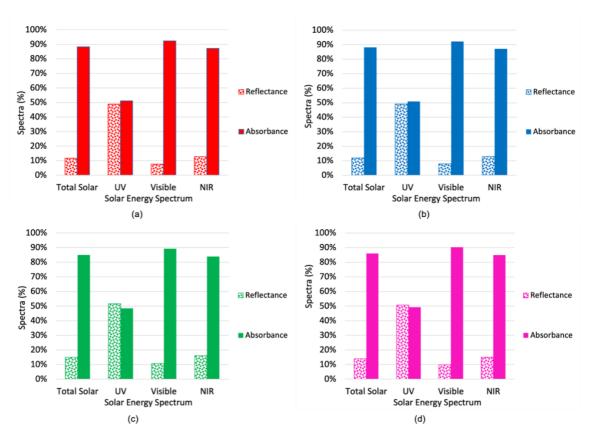


Figure 5.20: Reflectance & Absorbance percentages for UDDT sludge at (a) 1mm, (b) 2mm, (c) 3mm and (d) 4mm

Figure 5.20 shows variation from both VIP and ABR sludge. At both 1 and 2mm the absorbance values are slightly higher in the UV region, whereas at 3 and 4mm the reflectance values become higher (Figure 5.20). This indicates that the UV's ability to penetrate the faecal sludge decreases at these thicknesses.

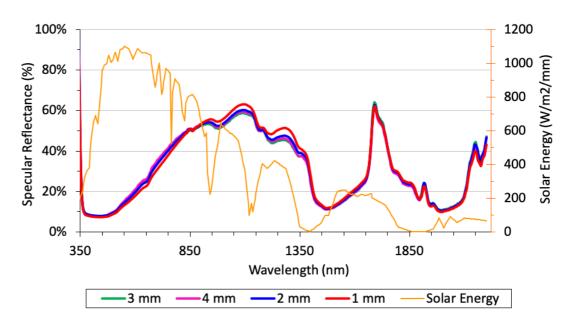


Figure 5.21: Total Solar Reflectance spectra with a ASTM E903 solar weighting applied produced by the UV-Vis-NIR for Human Faeces

Human Faeces has a low reflectance in the visible region (~17%) showing that it is highly absorbing at these wavelengths. In comparison it has a relatively high reflectance in both the UV (~33%) and near infra-red (~45%) regions meaning that less light is absorbed within these regions, which is what would be expected due to it being lighter in colour. Solar drying would still be an appropriate drying method when looking at the average total solar drying reflectance value (~33%) (Figure 5.21 and Figure 5.22).

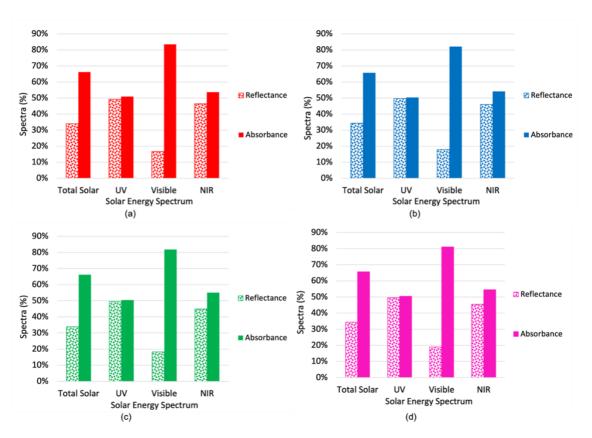


Figure 5.22: Reflectance & Absorbance percentages for HF sludge at (a) 1mm, (b) 2mm, (c) 3mm and (d) 4mm

While Figure 5.22 shows that human faeces has a relatively high reflectance percentage, it also has a very high transmission percentage (Figure 5.4). This shows that while a large proportion of the radiation is reflected away from the sample, what is absorbed is transmitted through the material with relative ease. This indicates that using sunlight to dry human faeces is an excellent drying source.

5.4 How does sample thickness affect the ability to reflect and absorb light

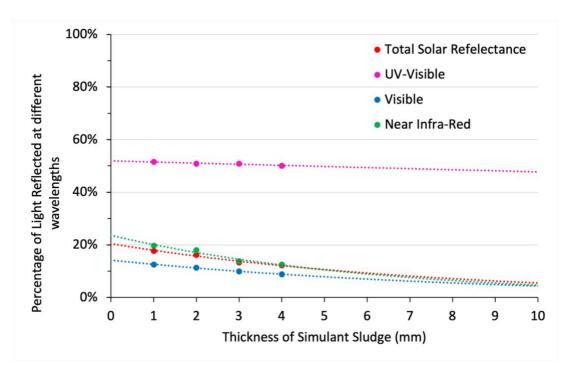


Figure 5.23: How the thickness of faecal sludge affects its ability to reflect light for VIP sludge

The reflectance percentages consistently decrease for VIP sludge. Once the thickness of faecal sludge reaches approximately 10mm the light will not be able to penetrate all the way through the faecal sludge causing crusting. This means that the sludge will need to be turned or raked on a regular basis, as drying at <3 mm thickness is not plausible on a large scale (Figure 5.23).

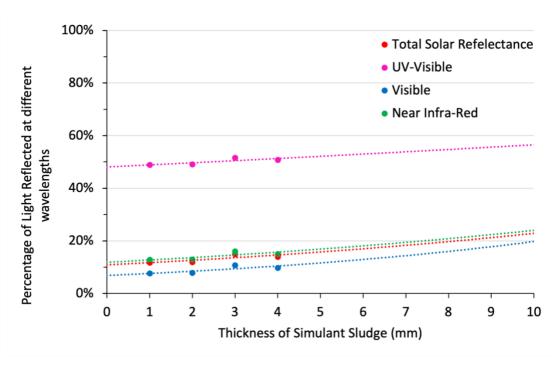


Figure 5.24: How the thickness of faecal sludge affects its ability to reflect light for UDDT sludge.

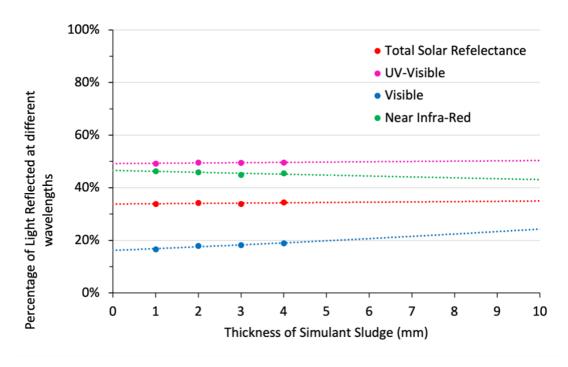


Figure 5.25: How the thickness of faecal sludge affects its ability to reflect light for human faeces.

When investigating how the thickness of the UDDT sludge and HF affects its ability to reflect and absorb light, the reflectance percentages stay consistent, demonstrating that the thickness of the slide does not have any significant impact on the penetration depth. (Figure 5.24)

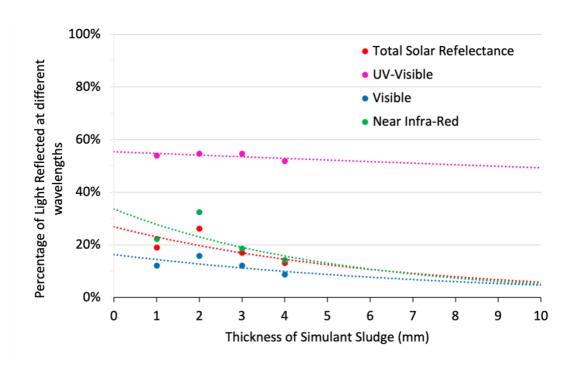


Figure 5.26: How the thickness of faecal sludge affects its ability to reflect light for ABR sludge

The reflectance percentages consistently decrease when analysing the ABR faecal sludge, indicating that once the thickness reaches approximately 10mm the light will not be able to penetrate all of the way through. This causes crusting and like VIP sludge it will need to be turned or raked on a regular basis (Figure 5.26).

5.5 Summary

Using the UV-Vis-NIR it was possible to identify whether solar thermal drying is an appropriate drying technique for faecal sludge, and whether there were any limitations. While all 4 types of faecal sludge absorbed more than 60% of the total suns radiation, with VIP and UDDT absorbing more than 80%, it was found that the sun wasn't able to penetrate more than 10mm into the faecal sludge which would in turn create crusting and mean that the sludge beds needed to be turned on a regular basis.

Chapter 6
Results & Discussion 3
Comparison of simulant sludge with real faecal sludge

Chapter 6 will compare the drying properties found within faecal sludge and simulant sludge to establish whether there are any similarities or differences between them. This will help to argue whether or not the simulant sludge can be used as an alternative to faecal sludge when developing new faecal sludge drying techniques.

When developing new drying techniques, gaining access to faecal sludge can pose a problem for several different reasons. This has meant that simulant sludge is sometimes used as an alternative to faecal sludge when developing new drying techniques, as well as new toilet designs. This has started an important discussion into whether this is an appropriate alternative and if they are similar enough in their material properties to allow simulant sludge to be used as an alternative to faecal sludge to develop these new systems. This chapter is going to investigate the similarities and differences that occur when understanding how each of the materials dry. Chapters 4 & 5 investigated some of the fundamental material properties of faecal sludge with noticeable differences observed between the different types of faecal sludge. This indicated that there would also be differences present between faecal sludge and simulant sludge at a material level. When the type of simulant sludge used within this research was developed it was chosen due to it having similar physio-chemical and rheological properties to faecal sludge (111). This made it an appropriate substitute for faecal sludge when used in a demonstrating capacity to prove a simple concept, such as at the Reinvent the Toilet challenge in India 2014. It is unknown whether it is appropriate to use as a substitute for proving chemical processes that are taking places within the reinvented toilets and newly developed drying technologies.

To identify whether simulant sludge should be used as an alternative to faecal sludge both materials were run through the simple UV-Vis-NIR and STA-FTIR methodology (Chapter 2) to draw direct comparisons. The same simulant sludge recipe was used as was used during the method development along with the three types of faecal sludge (Chapter 3).

6.1 UV-Vis Analysis

By running both the simulant and faecal sludge through the same UV-Vis-NIR programmes and collecting both %T and %R data allows direct comparisons to be drawn with respect to each type of faecal sludge.

6.1.1 Total Transmission

Figure 6.1 to Figure 6.4 show the transmission spectra for each of the 3 types of faecal sludge (VIP, UDDT and ABR) as well as the simulant sludge (111). As explained in Chapter 5 the transmission spectra allows the amount of light transmitted through the sample to be identified.

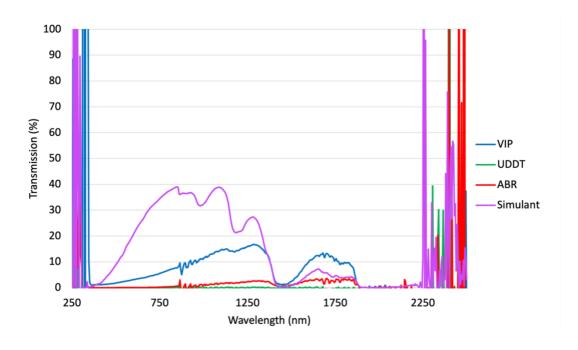


Figure 6.1: Transmission spectra produced by the UV-Vis-NIR when analysing 1mm samples

While the simulant sludge spectra produced appears to have a similar shape to the faecal sludge samples analysed, it does vary within the whole of the visible region and the end of the NIR region. Almost half of the energy emitted by the sun is in the form of visible radiation (44.7%) covering a relatively small wavelength range of 400 – 750 nm (131). Within this region of the transmission spectra's, Figure 6.1 to Figure 6.4 show that across all thicknesses there is a much larger transmission present within the simulant sludge than the faecal sludge.

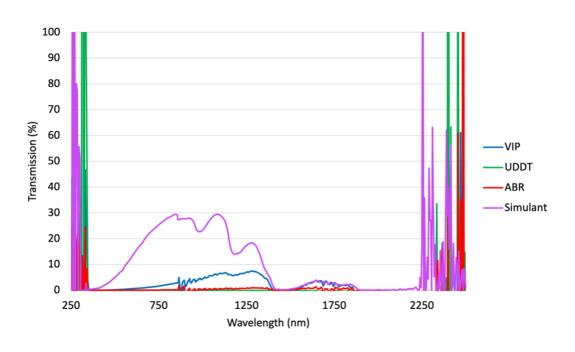


Figure 6.2: Transmission spectra produced by the UV-Vis-NIR when analysing 2mm samples

At 1mm (Figure 6.1) there is variation within the NIR region of the spectra. While there is still the large drop at about 1450 nm related to the vibrational overtone of an OH stretch within the high water content within the samples, it doesn't increase at the same rate as the faecal sludge. The same trend can be seen with a 2mm thick sample (Figure 6.2), however at this point the ABR and UDDT spectra are minimal showing that no real light is transmitted through the samples by this thickness.

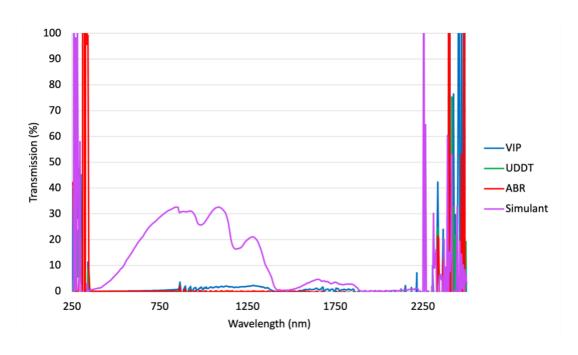


Figure 6.3: Transmission spectra produced by the UV-Vis-NIR when analysing 3mm samples

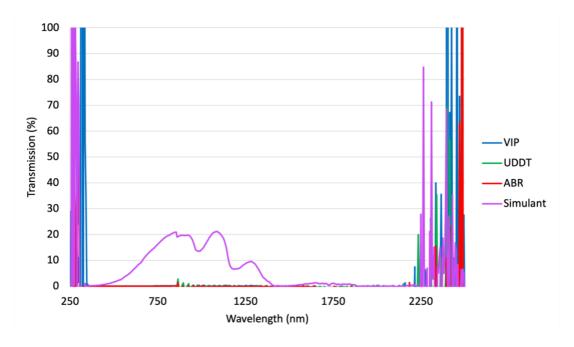


Figure 6.4: Transmission spectra produced by the UV-Vis-NIR when analysing 4mm samples

Figure 6.3 and Figure 6.4 show how the simulant and faecal sludge transmitted radiation at 3 and 4 mm thick respectively. They demonstrate that while the faecal sludge spectra are almost a flat line, the simulant sludge curve still has a noticeable transmission within the visible region and towards the beginning of the NIR region (450 - 1450 nm).

Comparing the differences between the simulant sludge and the faecal sludge shows that they transmit the suns radiation very differently. While at the lower thicknesses there are some similarities between the simulant sludge and the VIP sludge, once a thickness of 3mm is reached there is no comparison between them. This shows that if simulant sludge was used to create and develop dying technologies that focused on the sun's radiation, when these technologies were then used with faecal sludge, the way in which they would interact with the radiation from the solar spectrum would be vastly different. This would have a huge impact the efficiency and effectiveness of these new drying systems.

6.1.2 Diffuse Reflectance

By comparing the diffuse reflectance spectra it is possible to see how much light has been reflected off the rough surface of the simulant and faecal sludge (135).

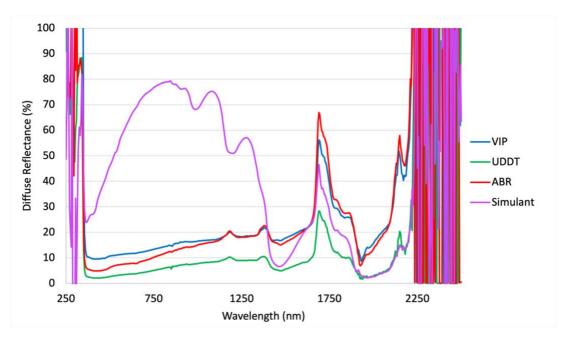


Figure 6.5: Diffuse Reflectance spectra produced by the UV-Vis-NIR when analysing 1mm samples

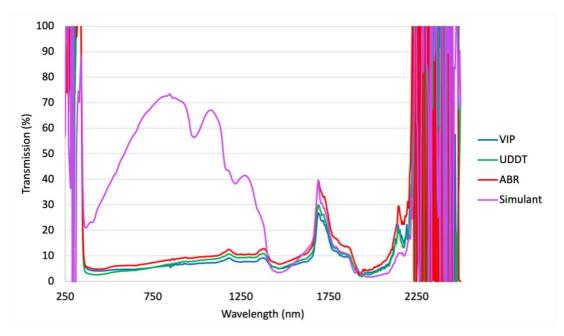


Figure 6.6: Diffuse Reflectance spectra produced by the UV-Vis-NIR when analysing 4mm samples

Figure 6.5 and Figure 6.6 show that when analysing the light that is reflected off the surface of the sample there is a lot of similarity within the NIR region from 1600 – 2200 nm with the spectra showing an almost identical distribution across all samples analysed, including the simulant sludge. When analysing the visible part of the spectra, as with the transmission spectra it can be seen that there is a huge difference in the distribution. Not only is there increased reflectance within the simulant sludge, but there is also a completely different shape to the curve. This shows again that the simulant sludge interacts with the radiation from the solar spectrum in a completely different way than each of the different types of faecal sludge. The simulant sludge is not an appropriate alternative when developing new drying techniques that use solar irradiance.

6.2 STA Analysis

6.2.1 Drying Kinetics

After investigating how simulant and faecal sludge reacts with the solar spectrum, it was important to understand how the drying rate was affected by using a simulant sludge rather than faecal sludge.

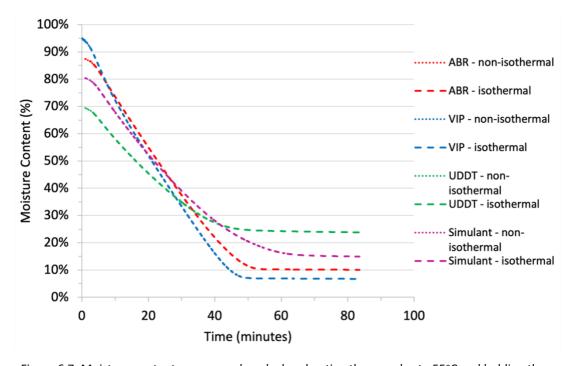


Figure 6.7: Moisture content curves produced when heating the samples to 55°C and holding them for 80 minutes

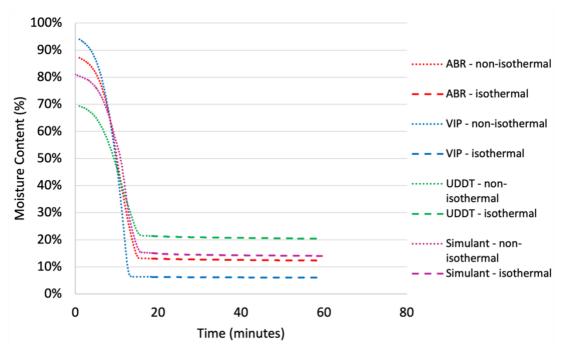


Figure 6.8: Moisture content curves produced when heating the samples to 205°C and holding them for 40 minutes

Chapter 4 investigated how the different types of faecal sludge acted when drying at different temperatures. Figure 6.7 and Figure 6.8 show how simulant sludge dries in comparison to VIP, UDDT and ABR sludge at 55°C and 205°C respectively. The simulant sludge follows a similar trend to the faecal sludge with the higher the initial moisture content, the lower the final moisture content reached. Independent of the temperature programme, the simulant sludge has a shallower drying curve than VIP sludge, but a steeper curve than the UDDT and ABR sludge. At 55°C the simulant sludge takes the longest to reach its final moisture content almost 10 minutes after the ABR sludge. This indicates that something within the material makeup of the simulant sludge causes the moisture to become trapped.

While there are not any similarities in the way that simulant and faecal sludge dry, it is important to note that there are also no major similarities between the different types of faecal sludge. However, as it falls within the range of faecal sludge types analysed it could be used within development stages of new drying technologies that use heat as a drying source. The developer would need to be aware that it was just an approximation, and that the system would need to be adjusted depending on the type of faecal sludge used.

6.2.2 Heat Flow

By understanding the heat flow demands needed to remove moisture from simulant sludge, it can be determined whether it is a suitable alternative to faecal sludge when trying to understand the energy demand needed to run new drying systems. While there are similarities across each of the samples analysed there are also major differences present when comparing simulant to faecal sludge.

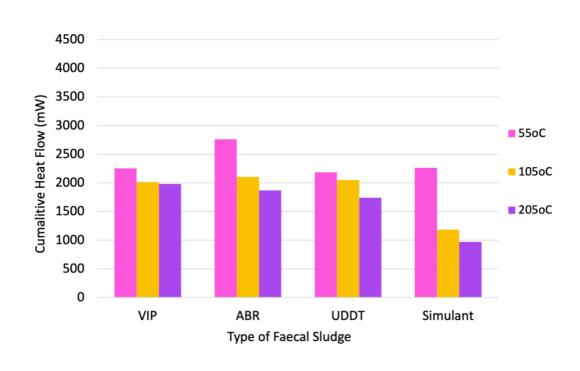


Figure 6.9: Cumulative heat flow curve for simulant, VIP, UDDT and ABR sludge at set temperature programmes until a constant moisture content was reached

Figure 6.9 shows that while heating to 55°C, there are similarities between the faecal and simulant sludge as soon as the simulant sludge is heated to 105°C and higher there is a noticeable difference in the energy demand needed to remove the moisture. This indicates that while the amount of time needed to remove the moisture is similar (Figure 6.8), the amount of energy needed to achieve this is much lower. This is as expected as with the faecal sludge, when heating above 100°C the energy needed to remove the moisture would be lower due to water having a boiling point of 100°C. The fact that the simulant sludge has such a lower energy demand requirement indicates that there is something present within faecal sludge that causes the water to be more tightly bound meaning that more energy is needed to remove the moisture. This leads to the understanding that using a

simulant to predict the energy demand necessary to remove faecal sludge would not produce accurate results especially when heating to the higher temperatures.

6.3 Can Simulant Sludge be used as an alternative to Faecal Sludge?

Combining the data produced using both the UV-Vis-NIR and STA-FTIR shows that while there are some similarities between faecal sludge and simulant sludge, the differences with respect to its drying properties are huge. Previous studies have shown that they have similar rheological properties (111), and after analysing the mass loss curves (Figure 6.7 and Figure 6.8), although simulant sludge shows some variation when compared to faecal sludge the trends present are comparable.

At low temperatures, the energy demand for drying simulant sludge is comparable, however as the temperature increased the variation also increased. Although the energy demand needed to dry simulant sludge follows the same downward trend as the temperature increases when compared to faecal sludge, it does so at a much faster rate. This indicates that while it could be used as an alternative to test low heat drying technologies, once temperatures of over 100° C are used the energy demand is so much lower that direct comparisons cannot be drawn.

When investigating how the solar spectrum radiation would interact with both the faecal and simulant sludge huge differences were observed. While there were vast differences when analysing all thicknesses, the variation became more obvious when examining the thicker samples analysed. Independent of the type of faecal sludge analysed, once a thickness of >2 mm was reached there was no significant transmission through the sample at any wavelength within the solar spectrum, whereas there was still a significant transmission at 4 mm when analysing the simulant sludge spectra.

The diffuse reflectance spectra's show that while there is similar reflectance within the NIR region of the solar spectrum, the visible region demonstrates a high variability. As this area represents 44.7% of the total solar radiation a large variability indicates that using a simulant sludge to replicate the drying properties of faecal sludge would produce inaccurate results leading to inefficient drying technologies being developed.

Chapter 7 Conclusion & Future Work

7.1 Conclusion

Understanding the way in which faecal sludge dries is more important now than ever before. This research has identified circumstances where simulant sludges can be used as an alternative to faecal sludge effectively, discovered new trends within the drying processes, and determined the most effective ways to dry different types of faecal sludge.

Before any analysis could be carried out on faecal sludge, new containment methods needed to be developed to allow for safe transport and analysis. By creating a new containment method for capturing and analysing faecal sludge, it has allowed for UV-Vis-NIR analysis to be carried out on a material which, if using traditional analysis methods would not have been possible.

Understanding how each type of faecal sludge interacted with the radiation from the solar spectrum is vital to fully understand how it will dry within the simplest of drying systems.

The way that solar irradiance was able to transmit through the faecal sludge varied greatly. While the VIP sludge allowed between 5 and 35% transmission depending on the thickness of the sample, the UDDT and ABR sludge showed no significant transmission within any part of the solar spectrum. When the same sample were analysed to understand how the solar radiation would reflect off the faecal sludge a more rounded understanding was found. The reflectance spectra produced shows a high reflectance within the NIR region of the solar spectrum, indicating that drying using this heat source alone would be difficult. If using the whole of the solar spectrum every faecal sludge type has a low reflectance and therefore a high absorbance capability proving that using solar energy to dry faecal sludge is an excellent drying process.

By analysing the faecal sludge using the UV-Vis-NIR it was also possible to identify how deep the radiation penetrated at different thickness, it is also important to remember that depending on the drying system being used there are also other drying processes in play. If you were to use a drying bed with VIP sludge, while the suns radiation would penetrate to approximately 10mm, there would also be a conduction process occurring leading to a quicker drying process. When these

processes occur simultaneously it can cause issues with crust formation which slows down the mass transport of moisture from deeper within the drying bed meaning that the sludge would need turning more frequently.

This research has established what happens when wet faecal sludge interacts with radiation from the solar spectrum but didn't research how these interactions change as the faecal sludge dries.

Understanding the link between pH, nutrient content and the type of faecal sludge has allowed conclusions to be drawn as to why VIP sludge dries quicker and easier than UDDT and ABR sludge. It has proved that removing the urine during the initial collection of faecal sludge to be used as a fertiliser is not always the most appropriate pre-treatment step dependent on the end goal.

The cumulative heat flow showed that to reduce each type of faecal sludge to set moisture contents, the faecal sludge dried quicker and with a lower energy demand using hotter temperature programmes. However, unless the exact end point is known, the amount of energy wasted when heating to higher moisture contents is much larger than at lower moisture contents. When developing a new drying programme, it is also important to note not only the energy demands needed to dry the faecal sludge, along with the time it takes, but also how easy would it be to heat the sludge to these high temperatures. Solar energy can be used when heating at low temperatures which is a low-cost option, however more complex technologies would need to be developed to dry at elevated temperatures leading to more costly process.

Understanding the energy demands and drying patterns for each temperature programme and faecal sludge type can make the drying process easier, quicker, and less expensive. Simple processing can enable on site drying, allowing systems to be set up much closer to the faecal sludge emptying sites in turn meaning that less energy will be needed to transport the faecal sludge as the water will have already been removed.

This research has concluded that VIP sludge dries most efficiently at 105°C, while UDDT and ABR sludge dries best at 155°C on a material level (40mg).

After investigating the drying emissions present within faecal sludge, it was seen that while each of the samples had a similar emission progression for water, the UDDT sludge had varied peak intensities when exploring the CO₂ peak. This suggests that while each of the faecal sludge samples release water in similar ways, the UDDT sludge has less CO₂ present and therefore the longer the heating process occurs, the less CO₂ is released.

This UV-Vis-NIR method was used to help determine how similar faecal sludge is to simulant sludge when using radiation produced by the whole of the solar spectrum. Investigating the UV Spectra produced for transmission and diffuse reflectance, it could be seen that while the simulant acted similarly to the faecal sludge within the near infrared region, within the visible vision there were vast differences. This was due to the simulant sludge being much lighter in colour than the faecal sludge. Faecal sludge is a mixture of both solid and liquid wastes that accumulate in onsite sanitation systems that's characteristics vary widely based on the climate, toilet type, diet etc, whereas simulant sludge is a laboratory made material with consistent properties, which doesn't have any of the same variables or major components. Differences were also observed when comparing simulant sludge to faecal sludge in terms of their energy demands. While they had similar energy demand when heating at low temperatures (55°C), simulant sludge had a much lower energy demand at 105°C and above.

This shows that although there are some similarities between simulant sludge and faecal sludge in terms of their physical properties, there are several major differences in the way that they dry. This proves that although simulant sludge can be useful in some contexts to start the development of testing and drying processes, simulant sludge cannot be relied on to provide accurate estimations for how a drying system will work.

Taking into consideration all the above points, it can be claimed that although there are some similarities between different types of faecal sludge, creating assumptions that they are all similar in the way that they will dry is incorrect. By understanding how the specific type of faecal sludge dries can help prevent an overuse of energy, allow precise moisture contents to be reached for further processing steps and

ensure that the correct drying systems are used to allow for quick and easy moisture removal.

7.2 Future Work

There are currently ongoing projects continuing the research from this project. A current EngD project is investigating the drying processes which take place when drying at a much larger scale. This will help to validate the results from this research, by understanding how the drying process differs on a larger scale. Investigating the evolved gases on a larger scale will allow a more detailed analysis of the organics being released at different times throughout the drying process, while also creating a more detailed understanding about the exact organic compounds that were present within the different types of faecal sludge. This would be required to establish trends with respect to the drying conditions and feedstock leading to better knowledge in understanding how diet can affect the chemical make-up of the faecal sludge, and in turn further validate why different types of faecal sludge dry at different rates and to different moisture contents.

Further work is required to understand how the sun interacts with the faecal sludge during the drying process. This will improve the understanding into how the material changes during the faecal sludge drying process, and how this will impact its interaction with radiation from the whole of the solar spectrum.

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