Understanding Bound Water Content and Water Binding Strength in Faecal Sludge from On-site Sanitation Technologies and Human Faeces

Report to the
Water Research Commission

by

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EXECUTIVE SUMMARY

Dehydration allows reducing the volume and mass of the faecal waste considerably and can cause the deactivation of pathogens. There is a need to improve the actual dehydration methods (dewatering and drying) and develop innovative technologies that are adapted to the faecal material and socioeconomic context.

The aim of this project is to characterize the moisture boundness in faecal sludge, i.e. how moisture can be found in the sludge structure matrix and its interactions with the solid material. The understanding of moisture boundness will be greatly beneficial to improve the dewatering and thermal drying processes. The investigation is led by the WASH R&D (Water, Sanitation and Hygiene Research and Development) Centre, University of KwaZulu-Natal (UKZN). The first part of the project consists in the analysis of the results from previous investigations that give an indication of the moisture boundness in faecal materials and the compilation of the extracted information to formulate the first explanations of moisture boundness in the faecal materials. The second part of the project is to generate a new set of data through experiments that will allow to obtain a more detailed and insightful representation of moisture boundness based on a multi-dimensional study. This work has been done by conducting experiments following different approaches to characterize moisture boundness, including the determination of the sorption isotherms and the hydraulic properties. For this study, faecal sludge from different on-site sanitation facilities within the eThekwini municipality (Durban metropolis, South Africa) have been used.

The limit of bound-unbound moisture varies between 50 to 70% moisture content (MC), depending on the analytical technique employed and type of faecal sludge. This limit could indicate the extent to which the sludge could be dewatered by a passive or moderate intensity method leading to the removal of unbound moisture. Capillary moisture is the more abundant type of bound moisture. It can be found approximately between around 70 to 20% MC. It is responsible of the lumpy consistency and high stickiness behaviour exhibited by faecal sludge in this MC range (observed by a peak of the cohesion and adhesion forces). Capillary moisture can removed by dewatering until a certain extent, but it will probably require high intensity mechanical force for this. Thermal drying is required for the complete removal of the capillary moisture.

Below 30% MC, the remaining moisture is mostly in the form of vicinal moisture (adsorbed at the surface of the solid particles by poly-layers) and internal moisture (being part of the chemical structure of the solid material and biological bodies). The removal of this type of moisture can only be achieved by thermal drying, but this will lead to an exponential increase of the energy consumption (because the energy to remove the vicinal and internal moisture is significantly higher than the latent heat of water vaporization). Nonetheless, drying could be stopped below 30% MC, and might not require the removal of the vicinal and internal moisture. Indeed, the sludge has the form of a granular solid at this point and could be considered safe in terms of pathogens since the remaining moisture is too bound for most of the microbes development and survival. Below 10% MC, the residual moisture can be found as a monolayer adsorbed at the surface of particles and as vicinal moisture. High amount of thermal energy would be required to reduce the MC to this level. Increasing the material temperature would reduce slightly the fraction of vicinal and internal moisture.

In general, faecal sludge drying requires a considerably higher thermal energy input than the latent heat of water vaporization because of the moisture boundness. In order to lead to energy savings, drying could be stopped at around 30% MC where the sludge could be considered safe, i.e. after removing the unbound and capillary moisture. The removal of the unbound and capillary moisture leads to significant changes in the physicochemical and mechanical properties of the faecal material, by converting it into a granular solid from a slurry or viscoelastic consistency and passing through an intermediary sticky lumpy phase. These changes must be considered in the design of dewatering and thermal drying units. The moisture boundness between the different types of faecal sludge differ slightly, but a more marked difference was observed with respect to fresh faeces. The average fresh faeces (type III and IV in the Bristol Stool Chart) could be more difficult to dewater and dry than faecal sludge due to a higher moisture boundness.

Once the missing work was achieved, this will allow to differentiate the different types of capillary moisture based on the capillaries characteristics and to understand how the physiochemical and structural properties of faecal sludge can explain the moisture boundness.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS & ABBREVIATIONS

ABR	Anaerobic Baffle Reactor
BMGF	Bill and Melinda Gates Foundation
BW	Black water
CMC	Critical moisture content
COD	Chemical Oxygen Demand
DSC	Differential Scanning Calorimetry
EC	Electrical Conductivity
EMC	Equilibrium moisture content
EPS	Extracellular Polymeric Substances
FS	Faecal sludge
GW	Greywater
MC	Moisture content
MSI	Moisture sorption isotherms
NMR	Nuclear Magnetic Resonance
OSS	Onsite sanitation
PAUWES	Pan African University – Water and Energy Sciences
PRIS	Postgraduate Research Innovation Symposium
PSD	Particle Size Distribution
R&D	Research & Development
RH	Relative humidity
ST	Septic tank
SWRC	Soil water retention curves
TGA	Thermogravimetric Analysis
TS	Total Solids
UDDT	Urine Diverting Dehydrating Toilet
UKZN	University of KwaZulu-Natal
VIP	Ventilated Improved Pit
VS	Volatile Solids
WASH	Water Sanitation & Hygiene
WRC	Water Research Commission

1 INTRODUCTION

1.1 BACKGROUND

Faecal sludge (Human waste accumulated in onsite sanitation facilities) is a wet material due to the inherent high moisture content of fresh faeces, and the eventual addition of urine, flush water and grey water into the toilet, as well as the infiltration of rainwater. Dehydration allows reducing the volume and mass of the faecal waste considerably and can cause the deactivation of microorganisms, including pathogens. Two dehydration methods exist, namely mechanical dewatering and drying. Mechanical dewatering leads to the removal of moisture from the sludge in liquid form using gravity (e.g. settling, natural percolation) or by applying a mechanical force (e.g. centrifugation, compression, filtration). Drying implies the removal of moisture in vapour form through evaporation, which can be done through natural drying (i.e. by relying on the natural evaporation of the moisture when exposed to open-air) or thermal drying (i.e. by applying heat). Thermal dryers can be categorized according to the method employed to provide the heat, which can be done by contact with a hot gas or hot surface, or by exposing the material to infrared, microwave or solar radiation, or by collecting solar thermal energy. Thermal drying can be an efficient way to disinfect faecal sludge as the generated hot temperatures can destroy the pathogen cells.

Nonetheless, the implementation of efficient dewatering and drying technologies has been challenging. Typically, drying beds are used to dehydrate faecal sludge, combining natural evaporation of moisture at the surface of the sludge bed, and leachate percolation and drainage at the bottom. This type of technology is low-cost and simple, but exhibits poor performance. More sophisticated dewatering and drying technologies have been attempted to be implemented, but many times they fail or are abandoned due to the technical difficulties and subsequent high operating costs. There is a need to improve the actual dehydration methods and develop innovative technologies that are adapted to the faecal material and socioeconomic context. Furthermore, the concept of toilets with in-situ treatment has been developed. This type of toilets would be composed of a frontend for user interface and backend for the treatment of the excreta. One of the major difficulties in the development of these toilets has been to down-scale dewatering and drying technologies in a viable way for the backend.

Research & Development (R&D) is required to tackle the technological gaps related to the dewatering and drying of faecal sludge. Currently, there is a critical lack of understanding of faecal sludge dewatering and drying processes, leading to the wrong selection or inappropriate use of technologies that are usually transferred from other applications. One of the major factors to comprehend corresponds to how the moisture is bonded to the dry-bone structure. In literature, it is well recognized that moisture can be found as unbound and bound through physical, chemical and biological interactions in moist materials. The different ways that the moisture is bound inside the material influences the thermodynamic and kinetic aspects of the drying process, as well as the dewaterability, hydraulic, physical, chemical, thermal and mechanical properties of the faecal material.

1.2 AIM AND OBJECTIVES

The aim of this project is to characterize the moisture boundness in faecal sludge, i.e. how moisture can be found in the sludge structure matrix and its interactions with the solid material. This project will bring useful information to better understand the structure of faecal sludge and the role of moisture into the properties of the material (chemical, physical, physiochemical mechanical, hydraulic, biological, thermal and hydraulic properties). This information will enable to better understand and predict the behaviour of faecal sludge in

treatment processes and convey systems. In particular, the understanding of moisture boundness will be greatly beneficial to the dewatering and thermal drying process to identify the limits of these processes, find technological solutions to overcome these limitations and improve the dewatering and drying technologies.
The objectives of the project are as follow:

- Reconciliation of different approaches to determine boundness moisture;
- Determination of the proportion of unbound moisture and the different types of bound moisture within the faecal materials
- Identification of the different types of interaction that bound moisture can undergo within the faecal materials.
- Correlation of the moisture boundness to the properties of the materials.

1.3 SCOPE OF THE RESEARCH

The investigation from this research project is led by the Water Sanitation & Hygiene (WASH) Research & Development (R&D) Centre, University of KwaZulu-Natal (UKZN), in Durban, South Africa. The first part of the project consists in the analysis of the results from previous investigations that give an indication of the moisture boundness in faecal materials and the compilation of the extracted information to formulate the first explanations of moisture boundness in the faecal materials. The second part of the project is to generate a new set of data through experiments that will allow to obtain a more detailed and insightful representation of moisture boundness based on a multi-dimensional study. This work has been done by conducting experiments following different approaches to characterize moisture boundness. The experimental work from this project is led by a PhD student as part of his thesis. He is assisted by a MSc student to characterise the thermodynamic aspects of moisture boundness through the sorption isotherm approach and interns to study the hydraulic properties of faecal sludge with the support from the Centre for Water Resources Research. The sorption isotherms and hydraulic properties can give an indication of the level of moisture boundness in faecal sludge. The methodology approach of this project is illustrated in Figure 1.1.

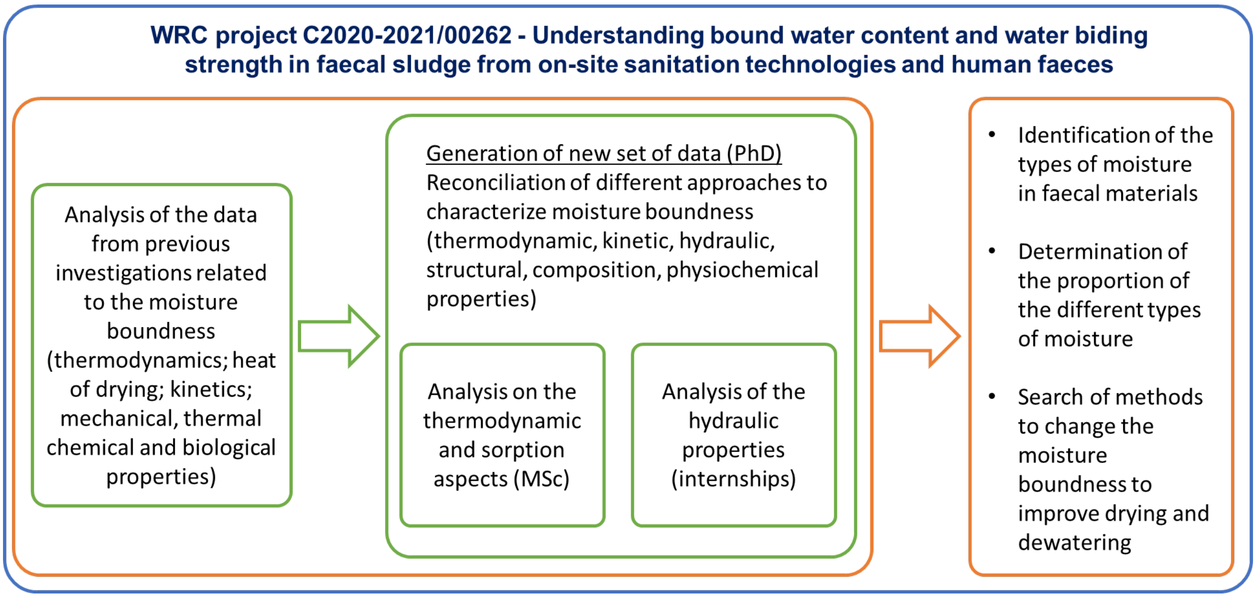


Figure 1.1. Summary of the project methodology

For this study, faecal sludge from different on-site sanitation facilities within the eThekwini municipality (Durban metropolis, South Africa) have been used, including faecal sludge from ventilated improved pit latrines, urine diversion dry toilets and septic tank. The experiments from this investigation were carried out at the laboratory of the WASH R&D Centre, as well as in the experimental facilities of other institutions when special equipment was required.

1.4 PREVIOUS DELIVERABLES

Deliverable 1 describes the general concepts of the project, provides a short literature review about moisture boundness and presents the general experimental methodology to follow during the project.
Deliverables 2 provides the updated strategy of the project, summarises the findings from previous investigation's related to moisture boundness, and presents the concepts and experimental plan of the experimental work to carry out.
Deliverable 3 presents the preliminary results that were obtained.
Deliverable 4 provides the final draft final report.

The previous Deliverables can be accessed from the Supplementary Material section.

1.5 OUTLINE OF THE REPORT

Section 1 (present one) depicts the general concepts of the project, including the background, aim, specific objectives and scope of the research project.

Section 2 describes the moisture boundness information that was deduced from the analysis of the experimental results from past investigations.

Section 3 shows the experimental and modelling sorption isotherm work performed for the characterization of moisture boundness for different types of faecal sludge (MSc research project).

Section 4 presents the experimental work conducted for a fine characterization of moisture boundness (PhD research project). It is important to mention that the experimental work related to this part of the project has not finished yet and experiments are currently in course, as well as the data treatment and analysis of the results. Therefore, this section is not yet finalised and presents only partial conclusions, which are enough to provide a general representation of moisture boundness in faecal sludge but not at finer detail level.

Section 5 discusses the outcomes from this project and the way forward.

Section 6 (Appendix) presents the preparation of experimental setup for the hydraulic tests and preliminary results. Indeed, the experimental work to characterise the hydraulic properties of faecal sludge is still under preparation. Due to several challenges (load shedding, difficulties for material procurement, problems during the experimental setup preparation), this part of the work could not be performed in the project timeline. However, it will be still carried out since it will provide further valuable data and a deeper understanding about the moisture boundness characteristics in faecal sludge. It is expected the publication of a paper for a peer-review journal with this work.

IMPORTANT: The outcomes from the missing work (i.e. hydraulic tests and finer characterization of faecal sludge) will be presented in the thesis of the PhD students and papers published in peer-review journals, and this report will be updated if time allows it. It is expected that these documents will be ready by the end of 2023 or beginning of 2024. The documents could be accessed online from the link displayed in the Supporting Documents section.

2 SUMMARY OF FINDINGS FROM PREVIOUS INVESTIGATIONS

The WASH R&D Centre has conducted several investigations related to faecal sludge drying characterization and understanding. These investigations provided an important set of data of different types, which can be analysed to get information about moisture boundness. This section describes the data obtained in past investigations and analyses it to get information about the characteristics of moisture boundness in faecal materials.

2.1 INTRODUCTION

Various investigations have been conducted at the WASH R&D Centre related to faecal sludge drying from 2014 onwards, mostly with funds from the BMGF and WRC. The characterization of moisture boundness was not the main focus of these investigations, however they generated valuable information that gives an indication of moisture boundness and its effects on the faecal material. The type of information related to moisture boundness depended on the experiment, and it was related to kinetic and thermodynamic data of the process, as well as the properties of the material (mechanical, thermal, physiochemical, biological). The data was obtained through different types of experiments for faecal sludge collected from different types of onsite-sanitation facilities in the eThekwini municipality, namely from ventilated improved pit (VIP) latrines and urine diversion dry toilets (UDDT). The studies also included sludge collected from the anaerobic baffled reactor (ABR) from a decentralised wastewater treatments system and fresh faeces collected from healthy donors at UKZN.

Table 2.1 gives the summary of the investigations at the WASH R&D Centre that provide information for the characterization of moisture boundness. The results from the different studies are compiled in this Chapter in order to give an initial overview of the state of moisture in faecal materials.

Table 2.1.Summary of the investigations conducted at the WASH R&D Centre related to the moisture boundness in faecal materials

Investigation	Funding source	Parameters measured related to moisture boundness	Reference
Convective drying of faecal sludge	BMGF	Drying kinetics	(Makununika, 2017)
LaDePa process for the treatment of faecal sludge	WRC	Thermal properties, nutrient content	(Septien *et al.*, 2020)
Drying behaviour of faecal matter	BMGF	Sorption isotherms, heat of drying, drying kinetics, dewaterability, thermal properties	(Getahun *et al.*, 2020; Naidoo *et al.*, 2020; Septien, 2020)
Faecal sludge solar thermal drying	BMGF/WRC	Drying kinetics, sorption isotherms	(Mawejje, 2021)
Aging of fresh faeces	BMGF	Sorption isotherms, dewaterability, thermal properties, rheological properties	(Chatema, 2021)
Stickiness of faecal sludge	BMGF	Cohesive / adhesive properties, plastic properties, rheological properties, sorption isotherms	(Mupinga, 2021)

2.2 THERMODYNAMIC PROPERTIES

In some of the investigations, the moisture boundness of faecal sludge and fresh faeces was deduced from thermodynamic data, i.e. sorption isotherms and heat of drying.

2.2.1 Sorption isotherms

The sorption isotherms gives the relationship of the moisture content (MC) at the thermodynamic equilibrium as a function of the relative humidity (RH). Once the thermodynamic equilibrium is reached, the water activity of the moisture in the sludge and the vapour water in the air are equal, and the system cannot consequently further evolve. Because the water activity of the vapour water in the air is the same than the RH, the sorption isotherms can be expressed as MC as a function of the water activity of the moisture within the sludge.

The thermodynamic activity is a term referring to the chemical potential of a substance within a chemical system. More specifically in the case of water activity, it can measure the level of interactions of the water molecules with its surrounding. In other words, it can indicate the level of moisture boundness. When the water activity is equal to 1 (maximum value), it can be considered that the moisture is unbound. At values lower than 1, the moisture boundness strength increases as the water activity decreases. A value of 0 means an infinitely bound moisture that could be never removed.

Therefore, the moisture boundness can be determined in a sorption isotherm curve and different types of moisture within the sludge could be identified, as it can be seen in Figure 2.1 According to this graph, the segment A-D represents the amount of bound moisture, with the segments A-B and B-C when the moisture is mostly found as adsorbed to the sludge solid particles as monolayer or multilayer respectively, and the segment C-D when most of the moisture is capillary. At MC above D, the moisture is unbound.

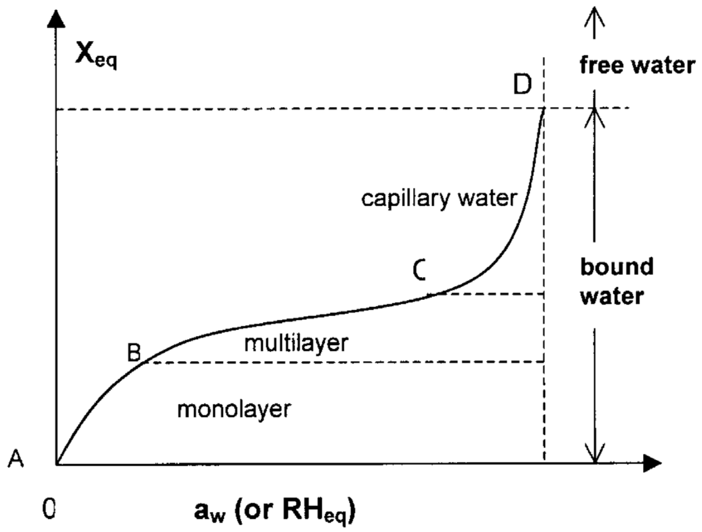


Figure 2.1. Classical sorption isotherm curve for moist materials (Vaxelaire, 2001)

In past investigations, the sorption isotherms of different types of faecal materials were determined experimentally through the method of the saturated salt solutions or the direct measurement of the water activity in an analyser. Figure 2.2. Sorption isotherms for 3 types of faecal sludge (Getahun *et al.*, 2020) illustrates an example of sorption isotherms that were obtained for VIP, UDDT and ABR sludge. A similar pattern can be observed for the three types of feedstocks and it follows the shape from the theoretical sorption isotherm from. The water activity is approximately equal to 1 at MC above 70%wt, which marks the transition between unbound (or free) and bound moisture (point D). Below this point, the water activity decreased smoothly until arriving to the point C at around 20%wt, from which the water activity dropped as the MC was lower. Hence, according to the representation from Figure 2.1, moisture is unbound above 70%wt MC, capillary between 70 and 20%wt MC and adsorbed below 15%wt MC (multilayer adsorption between 20 and 5%wt MC, and monolayer adsorption below 5%wt).

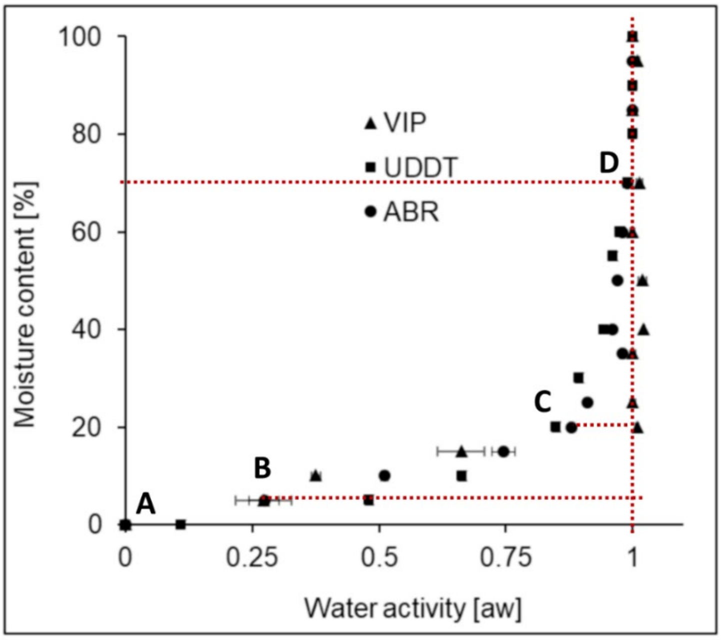


Figure 2.2. Sorption isotherms for 3 types of faecal sludge (Getahun *et al.*, 2020)

After applying the same methodology to analyse the sorption isotherms from the other investigations than in Figure 2.1, the different regions of type of moisture were identified and the results were summarized in Table 2.2. In summary, the moisture was found as unbound for MC above 70%wt, capillary between 70 to 20% MC and adsorbed (chemical, physical and/or biological) below 20% MC

Table 2.2. Summary of the moisture type boundaries deduced from the sorption isotherms data from investigations conducted at the WASH R&D Centre

Investigation	Type of FS	Unbound moisture	Capillary moisture	Bound moisture (physical, chemical, biological)
Drying behaviour of faecal matter	VIP	< 75%wt	75 to 10-20%wt	> 10-20%wt
	UDDT	< 75%wt	75 to 10-20%wt	> 10-20%wt
	ABR	< 75%wt	70 to 10-20%wt	> 10-20%wt
	Faeces	< 80%wt	80 to 10-20%wt	> 10-20%wt
Faecal sludge solar thermal drying	VIP	< 70%wt	70 to 30%wt	> 30%
	UDDT	< 70%wt	70 to 30%wt	> 30%
Aging of fresh faeces	Faeces	Not observed	80 to 20-30%wt	> 20-30%
Stickiness of faecal sludge	VIP	< 70%wt	70 to 20-30%wt	> 20-30%
	UDDT	< 70%wt	70 to 20-30%wt	> 20-30%

2.2.2 Heat of drying

Moisture boundness can also be evaluated through the measurement of the heat of drying and comparison to the latent heat of water vaporization (2 230 kJ/kg). A null or small difference in these values will mean that the moisture is mainly unbound, whereas a significant difference could reflect high moisture boundness strength. The heat of drying was measured at different samples and temperatures through thermal analysis instruments (differential thermal analysis and differential scanning calorimetry). The results obtained with UDDT sludge can be seen in Figure 2.3.

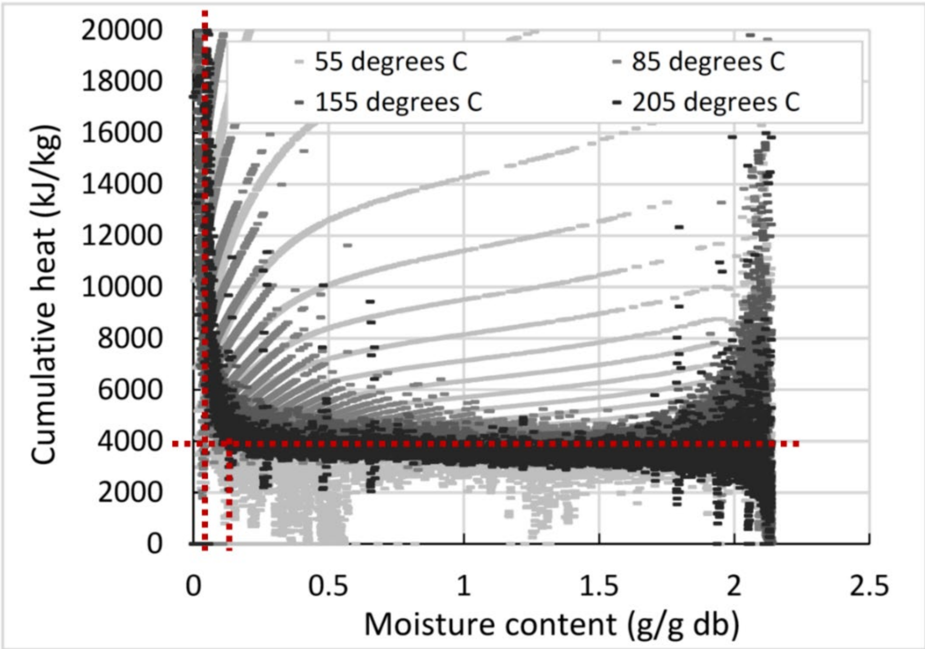


Figure 2.3. Evolution of the heat of drying as a function of MC for UDDT faecal sludge (Septien, 2020)

The heat of drying remained fairly constant at 4 000 kJ/kg during the transformation until achieving a MC of 0.2 g/g_{db} (i.e. 15%wt) below which it increased drastically almost as a vertical asymptote. It can be noted that the heat of drying was significantly higher than the latent heat of water vaporization, suggesting a certain level of moisture boundness. The drastic increase of the heat of drying below 15%wt MC could reflect that only moisture tightly bound to the solid matrix remained (for example: moisture chemically and biological bound).
A similar trend was obtained for the ABR and VIP sludge, and fresh faeces. However, for the VIP and ABR samples, the heat of drying was closer to the latent heat of water vaporization, which could be attributed to their higher initial moisture and therefore higher amount of unbound moisture.

2.3 KINETIC PROPERTIES

According to the drying kinetic theory, the drying rate can be divided into 3 major phases:

- Constant rate period when drying occurs at the surface of the material, which is saturated in moisture.
- First falling rate period when drying still occurs at the surface of the material but it is not anymore saturated in moisture, causing a decline in the drying rate.
- Second falling rate period when the surface of the material is completely dried and a drying front moves inside the material, leading to a further slowing down of the process,

It is considered that constant rate period ends when the movement of moisture within the sludge cannot longer ensure the replacement of the moisture evaporated at the surface. It is evident that the duration of the constant rate period depends on a certain extent of the amount of unbound moisture in the material, as this type of moisture is the one that can move with more freedom within the sludge. It would be considered that, at operating conditions leading to low drying rates, the end of the constant rate period corresponds to the depletion of the unbound moisture, considering that the movement of the other types of sludge is too limited to keep the surface of the material saturated in moisture.

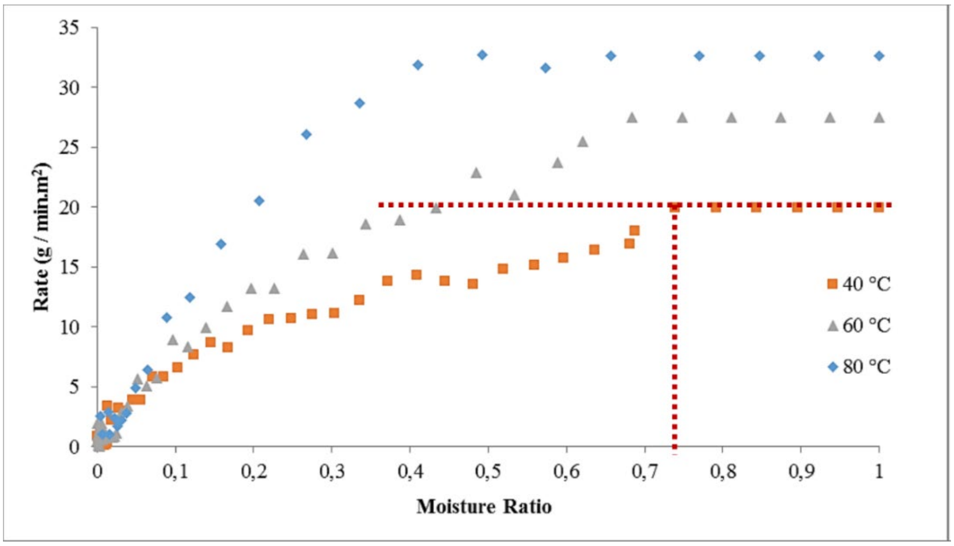


Figure 2.4. Drying rate curves during the convective drying of VIP sludge (Makununika, 2017)

The approach described above was employed in the kinetic data from previous investigations in order to determine the amount of unbound and bound moisture. For this, only the data obtained from tests with a slow drying rate was considered. An example of this can be observed in Figure 2.4, where only the drying experiment at 40°C was deemed enough slow for the analysis. The transition between the constant and falling rate period occurred at a moisture ratio of 0.75 approximately, i.e. 60% MC, marking the transition between unbound and bound moisture.

The rest of the results is presented in Table 2.3. According to the drying kinetic method, the moisture is unbound above 60-70% MC, and bound below this range.

Table 2.3. Summary of the moisture type boundaries deduced from the drying kinetics data from investigations conducted at the WASH R&D Centre

Investigation	Type of FS	Unbound moisture	Bound moisture (capillary, physical, chemical, biological)
Convective drying of faecal sludge	VIP	< 60%wt	> 60%wt
Drying behaviour of faecal matter	UDDT	< 60%wt	> 60%wt
	ABR	< 75%wt	> 75%wt
Faecal sludge solar thermal drying	VIP	< 60%wt	> 60%wt

2.4 MECHANICAL PROPERTIES

The mechanical properties of the sludge depends on how the moisture and solid matrix are assembled and interact together. Therefore, according to the mechanical properties that the material exhibits at the macroscopic level, it is possible to get information on the moisture boundness and the nature of the interactions of moisture with the solid constituents of the sludge.

2.4.1 Rheological properties

It has been already demonstrated that faecal sludge and fresh faeces are a shear thinning material whose viscosity decreases by increasing the shear rate (Woolley *et al.*, 2014; S Septien *et al.*, 2018). By increasing the MC for this type of material, it becomes less viscous and thereby it can flow more easily. In contrast, a reduction of the MC leads to a more viscous material which shows a greater resistance to flow. Below a given MC, the material can lose its viscoelastic properties and in consequence its ability to flow. Assuming that the sludge can flow thanks to the unbound moisture that transports the solid particles and aggregates as it moves, the loss of its ability to flow could reflect that no unbound moisture is anymore available.

During experiments in the rheometer by varying the MC of the sludge, it was noticed that the sludge was not able to flow below a given MC, which could probably mark the end of the unbound moisture as explained above. As seen in Table 2.4, this happened around 70% MC.

Table 2.4. Summary of the moisture type boundaries deduced from the rheology data from investigations conducted at the WASH R&D Centre

Investigation	Type of FS	Unbound moisture	Bound moisture (capillary, physical, chemical, biological)
Aging of fresh faeces	Faeces	< 65%wt	> 65%wt
Stickiness of faecal sludge	VIP	< 70%wt	> 70%wt
	UDDT	< 70%wt	> 70%wt

2.4.2 Stickiness

Stickiness is defined as the mixture of the cohesive and adhesive forces inside the material. Cohesiveness refers to the force between the constituents of the material while adhesiveness corresponds to the force between the material and an external surface. In the case of sludge, cohesiveness and adhesiveness depends on the amount of moisture in the material, affecting its stickiness as it can be seen in Figure 2.5. At high MC, the stickiness is at its minimum value probably because of the high amount of unbound moisture acting as a lubricant. However, as unbound moisture is removed from the matrix, the flocs and particles approach, leading to the formation of interstices and capillary where moisture is trapped and is unable to move. The immobilized moisture acts as a glue keeping tightly together the sludge constituents and creates a strong attachment of the bulk material with any external surface with affinity to water, leading to the subsequent increase of the cohesiveness and adhesiveness in the so-called sticky region. After shrinkage of the capillaries during drying, the adsorbed moisture on the flocs and particles surface becomes in contact, creating dense clusters of solid material, leading to the maximum cohesiveness and adhesiveness at the sticky peak. After this, the cohesive and adhesive forces decrease as the capillary moisture is removed until the material stops to be sticky.

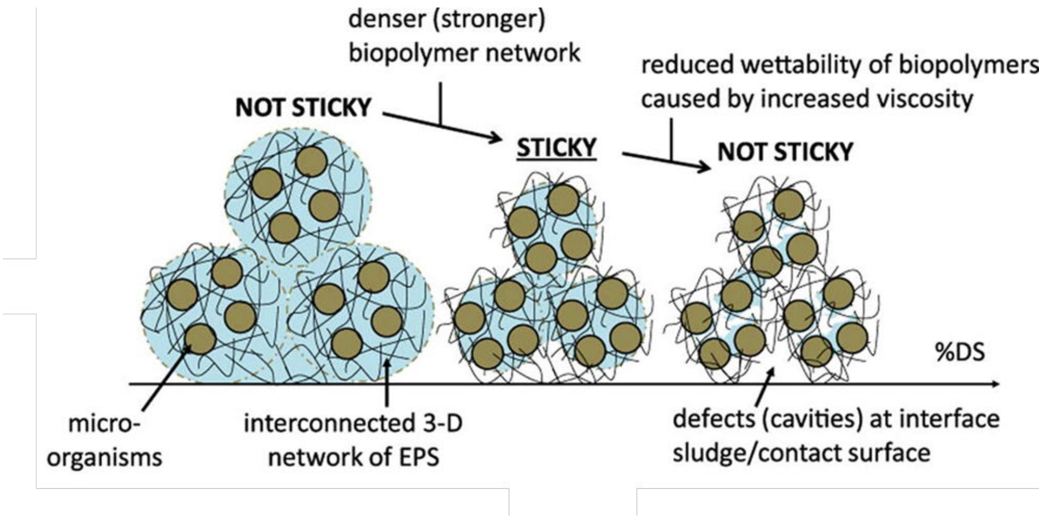


Figure 2.5. Relationship of stickiness and MC (adapted from (Peeters *et al.*, 2013))

Based on the above observations, the types of moisture in faecal material can be identified by the determination of the cohesive and adhesive forces profile as a function of MC, as it was done in Figure 2.6 for UDDT sludge using a texture analyser apparatus. In this case, the sticky region ranged between 70 to 30% MC, with the stickiness peak between 50 to 60% MC. This result suggests that capillary moisture was mostly found between 70 to 30% MC. The unbound moisture was completely removed below 70%wt MC. Below 30% MC, the remaining moisture was probably tightly bound to the solid probably through physical, chemical and/or biological interactions.

A similar result was obtained for VIP sludge (Table 2.5).

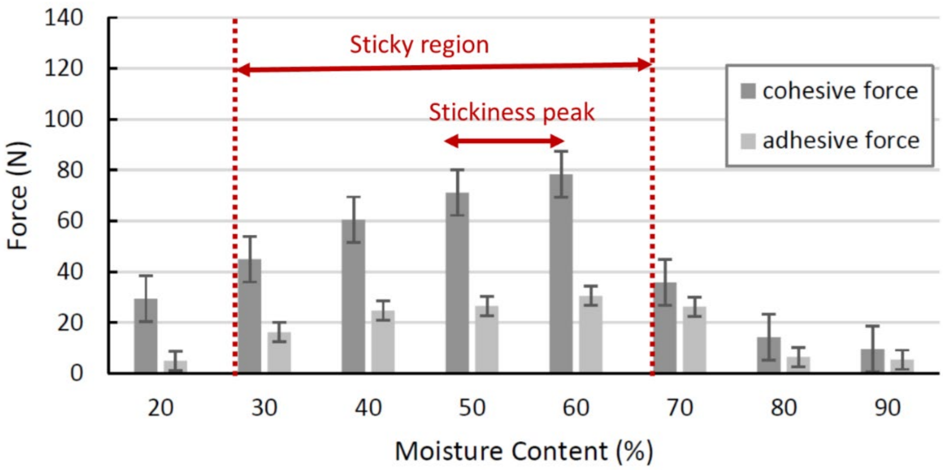


Figure 2.6. Adhesive and cohesive forces measured for VIP sludge at different MC (Mupinga, 2021)

Table 2.5. Summary of the moisture type boundaries deduced from the stickiness data from investigations conducted at the WASH R&D Centre

Investigation	Type of FS	Unbound moisture	Capillary moisture	Bound moisture (physical, chemical, biological)
Stickiness of faecal sludge	VIP	< 70%wt	70 to 50-60%wt	> 30-40%wt
	UDDT	< 70%wt	70 to 50%wt	> 30-40%wt

2.4.3 Dewaterability

Experiments in centrifuge were performed in order to assess the dewaterability of various types of faecal sludge and fresh faeces. Based on the assumption that the removed water after consists mainly of unbound moisture, this method could give an estimation of the amount of unbound and bound moisture in the faecal materials. The results from this analysis in Table 2.6 indicates that the limit of unbound and bound moisture lies in the range 60 to 75%wt MC. Fresh faeces could not be dewatered, denoting that they might not contain unbound moisture.

Table 2.6. Summary of the moisture type boundaries deduced from the stickiness data from investigations conducted at the WASH R&D Centre

Investigation	Type of FS	Unbound moisture	Bound moisture (capillary, physical, chemical, biological)
Drying behaviour of faecal matter	VIP	< 70-60%wt	> 70-60%wt
	UDDT	< 70%wt	> 70%wt
	ABR	< 75%wt	> 75%wt
	Faeces	Not observed	> 80%wt
Aging of fresh faeces	Faeces	Not observed	> 80%wt

2.4.4 Morphological changes

During the drying of faecal sludge, the samples were photographed at different MC and the evolution of their aspect was recorded, as it can be observed in Figure 2.7 for UDDT sludge. Between 90 to 80%wt MC, the sludge exhibited a liquid consistency (corresponding to unbound moisture), which changed to a more pasty and lumpy from 70 to 40%wt MC (corresponding to capillary and surface moisture). Finally, below 30%-20% moisture, the sludge started to have the look of a dried solid (corresponding to strongly bound moisture).

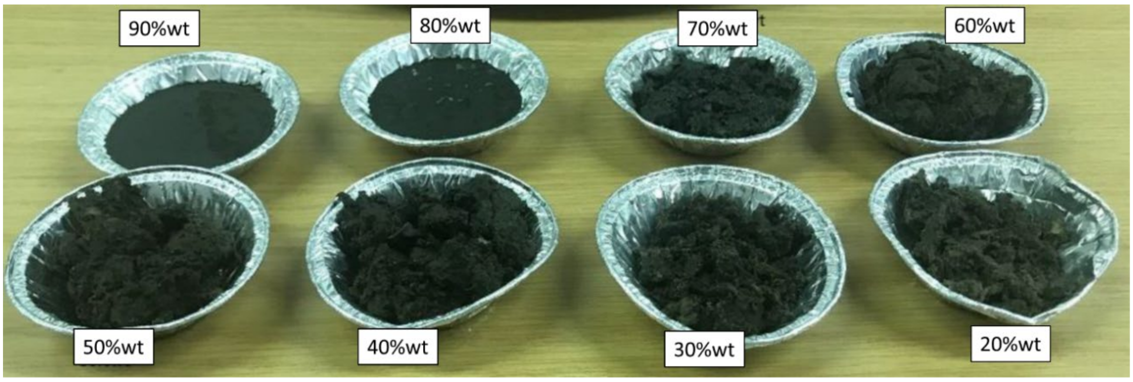


Figure 2.7. Aspects of VIP sludge at different MC (Mupinga, 2021)

2.5 RELATIONSHIP OF FAECAL MATERIAL PROPERTIES WITH MOISTURE BOUNDNESS

During the drying experiments, it was observed that some of the properties of the faecal samples varied along the process, among which: the thermal properties (thermal conductivity and heat capacity), chemical properties (concentration of ammonium, nitrates, nitrites and orthophosphates) and biological properties (Ascaris eggs viability). These changes could be related to the change of moisture boundness as drying progresses.

2.5.1 Thermal properties

The thermal conductivity refers to the ability of a substance or material to conduct heat, whereas the heat capacity gives the heat required for the material for the increase of its temperature. The value of both thermal properties is dependent on the amount of moisture in the material, as it was verified for VIP sludge in Figure 2.8 and Figure 2.9. At the initial MC (80%wt), their values are similar than those from water, namely 0.6 W/m/K for the thermal conductivity and 4 180 J/kg/K for the heat capacity. As moisture was removed, they dropped rapidly until achieving stabilization below 40%wt MC. According to the previous sections, this MC was achieved after removing the unbound and an important part of the capillary moisture. The remaining moisture (surface, chemically and biologically bound moisture) does not have therefore an influence on the thermal properties of the material, unlike the unbound and capillary moisture. The initial values of the thermal properties close to those of pure water was probably attributed to the presence of unbound moisture in the sludge.

The final thermal conductivity and heat capacity were 0.04 W/m/K and 400 J/kg/K that are close to the values from air. This could be probably due to an important volume of air trapped in the pores from the dried sludge, thereby controlling the value of the thermal properties.

A similar trend was observed for other types of sludge samples and fresh faeces (Septien, 2020).

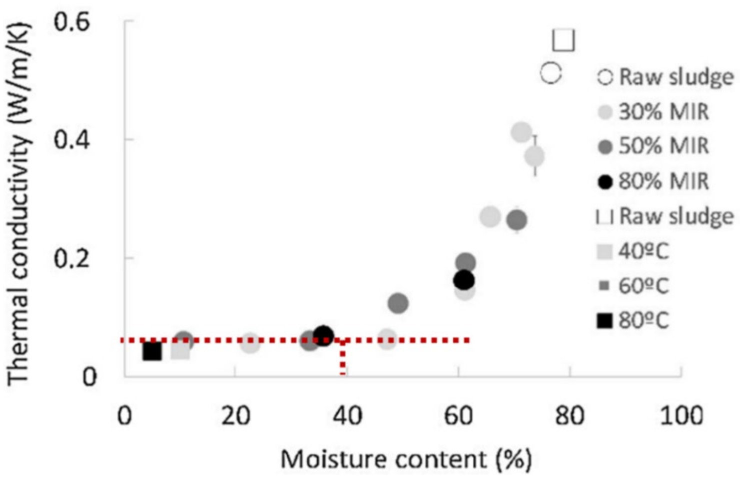


Figure 2.8. Thermal conductivity for VIP sludge at different MC (Septien *et al.*, 2020)

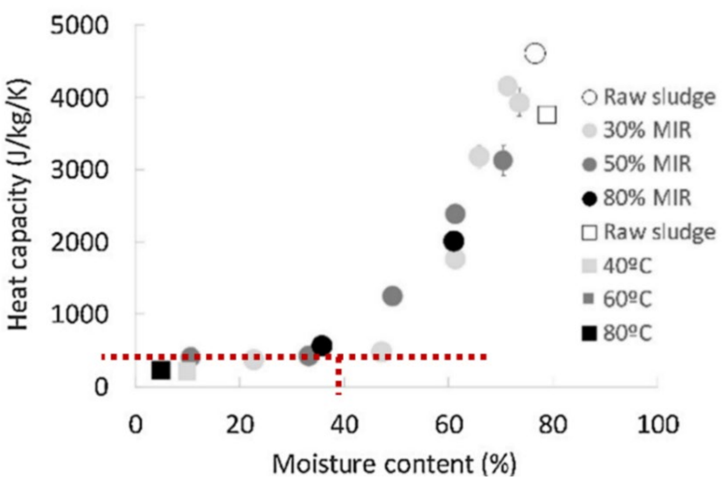


Figure 2.9. Heat capacity for VIP sludge at different MC (Septien *et al.*, 2020)

2.5.2 Chemical properties

Similarly to the thermal properties, a decrease of the ammonium and nitrites content was observed during drying of VIP faecal sludge until stabilisation at 40% MC (Figure 2.10). As the total nitrogen remained constant (implying that there were any losses of nitrogen through volatilization), it was hypothesized that the nitrogen undergone changes of its chemical form from free ammonium and nitrites to forms linked to the solid matrix that could not be detected by the chemical analysis. This modification of the nitrogen chemical form was likely linked to the moisture boundness changes. In particular, the removal of unbound and capillary moisture reduced the volume of water where the ammonium and nitrites ions were solubilized, possibly obliging to their precipitation and linkage to the solid structure of the sludge.

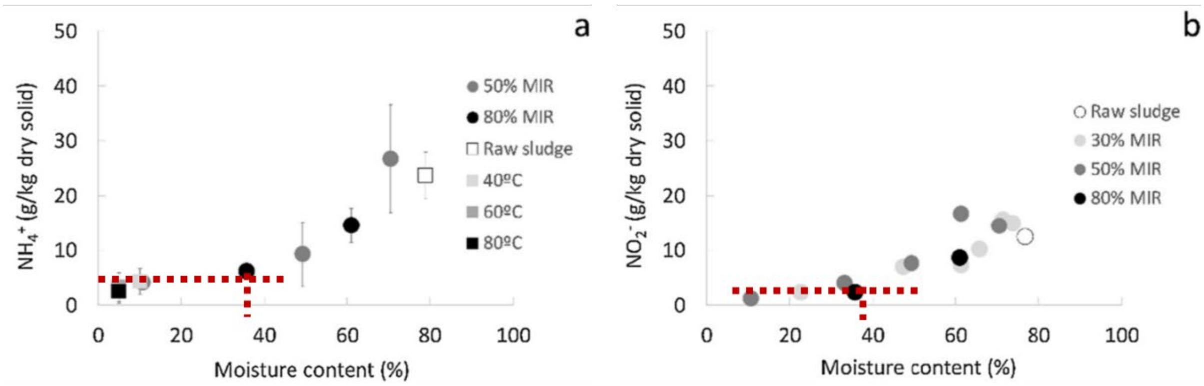


Figure 2.10. Ammonium (a) and nitrites (b) content measured for VIP sludge dried at different MC (Septien *et al.*, 2020)

In addition to this, the evolution of other compounds during drying also was studied. A similar trend than the ammonium and nitrites was remarked for the orthophosphate content that declined with the removal of MC during drying (Figure 2.11) while the total phosphorous remained constant. It could be then deduced that the changes of moisture boundness during drying also affected the phosphorous chemical form through the incorporation of the orthophosphates into the solid matrix of the sludge.

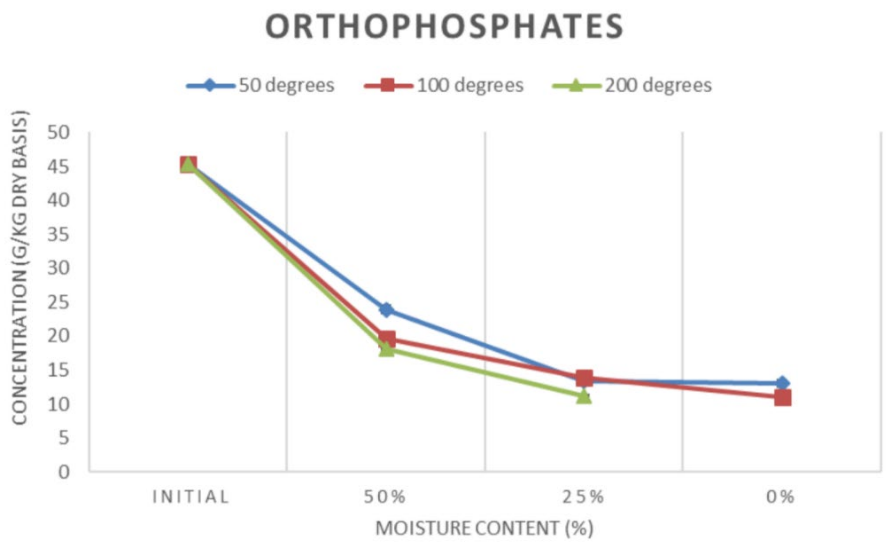


Figure 2.11. Orthophosphate content measured for VIP sludge dried at different MC and temperatures

2.5.3 Biological properties

In literature, there are well established tables of the minimum water activity for microbial development, as the one shown in Table 2.7. Indeed, below a given water activity, a specific microorganism cannot develop as it is unable to use the available bound moisture for its metabolism and development. This is one of the reasons that drying can disinfect materials such as faecal sludge, after reaching level of water activity where pathogens cannot survive. According to Table 2.7, most of bacteria, including the dangerous pathogen Salmonella, can be deactivated if the water activity of the sludge is reduced below 0.91. This implies that the bacteria survival is possible in capillary moisture at a certain extent after removing the unbound water, but it will be compromised by advanced removal levels of capillary moisture. The yeast and moulds show more resilience to lower levels of water activity compared to bacteria, and they might require the complete removal of the capillary moisture for their deactivation. A few microbes as the Xerophilic fungi and Osmophilic yeast exhibit remarkable tolerance to low water activities, as they can survive in medias with only strongly bounded available. In this case, the deactivation of these microbes will be only possible by removing all the moisture in the material or almost.

Table 2.7. Minimum water activity for microbial and spore development (Mujumdar and Devahastin, 2000)

Pathogen	Water activity
Pseudomonas, Bacillus cereus spores	0.97
B. subtilis, C. botulinum spores	0.95
C. botulinum, Salmonella	0.93
Most bacteria	0.91
Most yeast	0.88
Aspergillus niger	0.85
Most molds	0.80
Halophilic bacteria	0.75
Xerophilic fungi	0.65
Osmophilic yeast	0.62

The effect of sludge dryness was studied on the survival of Ascaris eggs, which has not been reported in literature in the best knowledge of the authors. This study was conducted by measuring the eggs viability in sludge dried at different MC after 6 weeks of storage. The results can be visualized in Figure 2.12.

It can be seen that the viability of the eggs was not affected for sludge with a MC above 50% during 6 weeks of storage. The viability of the eggs decreased for sludge dried at a MC of 40% or lower after a few weeks of storage. The decline of the eggs viability started sooner and led to a lower viability as the sludge was drier. Therefore, the Ascaris eggs viability is compromised if they are placed in a sludge with a MC below 40% (corresponding to a water activity lower than 0.95). This implies that the Ascaris eggs can be deactivated after removing the unbound moisture and part of the capillary moisture.

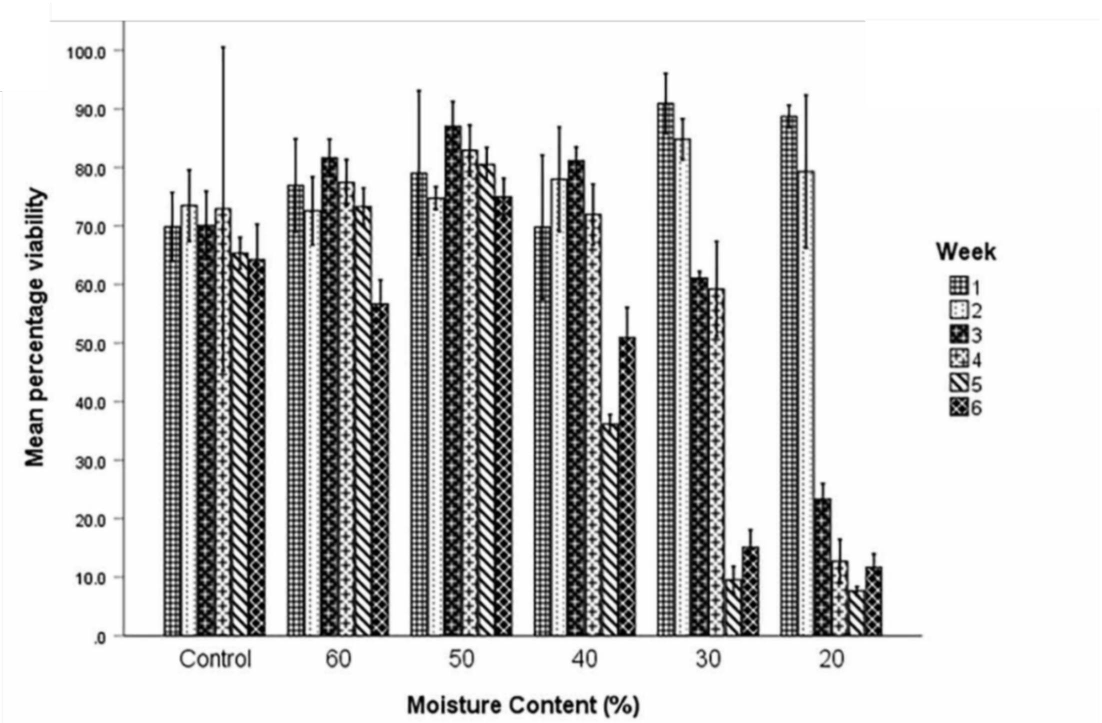


Figure 2.12. Ascaris eggs viability measured for VIP sludge dried at different MC during different storage time (Naidoo *et al.*, 2020)

2.6 CONCLUSION

In this chapter, the moisture boundness of faecal sludge and fresh faeces was studied through the results obtained in various past investigations that were not interlinked necessarily. After compiling and summarizing the results from this section, the distribution of the different types of moisture within the sludge was deduced, as follow:

- Unbound moisture found for MC above 60-75%wt;
- Capillary moisture found for MC between 60-75%wt and 10-30%wt;
- Physical, chemically and biologically bound moisture below a MC of 10-30%wt.

It can be observed that the limit between the different types of moisture varied in a range, as the moisture boundness information was obtained through different types of experiment and feedstock. Nonetheless, the results from the different investigation converged quite well in overall. The moisture boundness difference between the different types of faecal sludge was minimal, but a higher difference was observed between the faecal sludge and fresh faeces. The latter did not exhibit evidence of unbound moisture even at MC of 80%. This suggests that the moisture boundness of fresh faeces (type III and IV in the Bristol Stool Chart) was significantly higher than that of faecal sludge. Indeed, during storage, the faecal sludge could have received water from urine, flushing or infiltration, and could have undergone biochemical degradation, increasing its content in unbound moisture with respect to the initial faecal material.

The changes in moisture boundness during drying lead to modifications of the material. Typically, the raw faecal sludge is a viscoplastic fluid that can flow after applying a minimum shear stress due to the presence of unbound moisture. When the unbound moisture is removed, the sludge losses it ability to flow, leading to a sticky pasty-like material composed majorly of capillary moisture. The stickiness reaches a peak after removing part of the capillary moisture, causing the shrinkage of the capillaries and the formation of a dense and cohesive material as the flocs and particles get closer. After reaching this point, the removal of further capillary moisture creates voids in the material where the evaporated moisture is replaced by air, leading to the drop of stickiness. After removing the entire capillary moisture, the sludge becomes an hygroscopic solid, with moisture adsorbed at the surface and trapped in the structure of the material.

The removal of the unbound and capillary moisture has an effect on the properties of the sludge. It causes a tenfold decrease on the thermal properties of the material, namely thermal conductivity and heat capacity, from initial values close to that from pure water to values similar to air. Moreover, the chemical form of the nitrogen and phosphorous passes from that of soluble components (ammonium, nitrites and ortho-phosphate) to a more bound form with the solid matrix. Finally, the removal of the unbound and part of the capillary moisture leads to a desiccated environment where most of the pathogens, including bacteria and Ascaris eggs, cannot survive.

The outcomes from this Chapter are summarized in Figure 2.13.

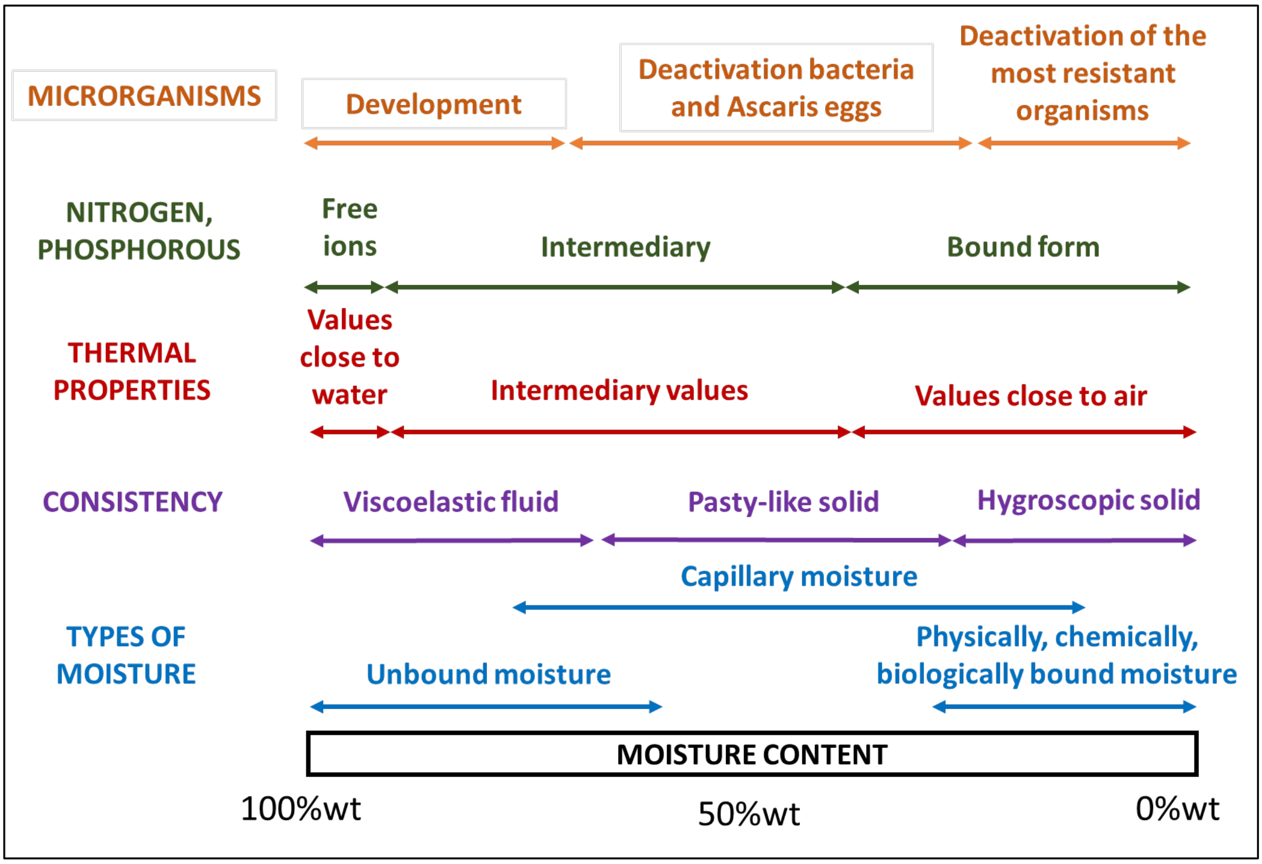


Figure 2.13. Scheme showing the types of moisture as a function of the MC and its effects on the properties of the faecal sludge

The present research project (C2019.2020-00262) will build on the knowledge and insight gained from the previous investigations about moisture boundness and compiled in this Chapter. In particular, this project is expected to bring an understanding of moisture boundness at a higher resolution, including the following aspects:

- Find a more precise limit between the different types of moisture;
- Characterize with finer detail the different types of unbound moisture (e.g. differentiation between the physically, chemically and biologically bound moisture);
- Correlation of the physiochemical and hydraulic properties of faecal sludge with moisture boundness (e.g. distribution of size of capillaries, content in extracellular polymeric substance, etc…), allowing to determine the type of involved interactions and their intensity.

2.7 REFERENCES

Chatema, T. M. (2021). EFFECT OF AGEING ON THE PHYSICOCHEMICAL PROPERTIES OF FRESH FAECES.

Getahun, S., Septien, S., Mata, J., Somorin, T., Mabbett, I., & Buckley, C. (2020). Drying characteristics of faecal sludge from different on-site sanitation facilities. Journal of Environmental Management, 261. https://doi.org/10.1016/j.jenvman.2020.110267

Makununika, B. (2017). Thermal Drying of Faecal Sludge from VIP latrines and Charcaterisation of Dried Faecal Material.

Mawejje, M. N. (2021). Solar Thermal Convective Drying Characteristics and Kinetics for Faecal Sludge Treatment.

Mujumdar, A. S., & Devahastin, S. (2000). Fundamental principles of drying. Exergex, Brossard, Canada.

Mupinga, R. T. (2021). Stickiness of faecal sludge for drying applications.

Naidoo, D., Archer, C. E., Septien, S., Appleton, C. C., & Buckley, C. A. (2020). Inactivation of Ascaris for thermal treatment and drying applications in faecal sludge. Journal of Water, Sanitation and Hygiene for Development, 10(2), 209-218.

Peeters, B., Dewil, R., Vernimmen, L., van den Bogaert, B., & Smets, I. Y. (2013). Addition of polyaluminiumchloride (PACl) to waste activated sludge to mitigate the negative effects of its sticky phase in dewatering-drying operations. Water Research, 47(11), 3600-3609.

Septien, S. (2020). Final technical report Project OPP1164143 – Characterization of faecal material behaviour during drying. Gates Open Res, 4(151), 151.

Septien, S., Mirara, S. W., Makununika, B. S. N., Singh, A., Pocock, J., Velkushanova, K., & Buckley, C. A. (2020). Effect of drying on the physical and chemical properties of faecal sludge for its reuse. Journal of Environmental Chemical Engineering, 8(1). https://doi.org/10.1016/j.jece.2019.103652

Septien, S., Pocock, J., Teba, L., Velkushanova, K., & Buckley, C. A. (2018). Rheological characteristics of faecal sludge from VIP latrines and implications on pit emptying. Journal of Environmental Management, 228, 149-157.

Vaxelaire, J. (2001). Moisture sorption characteristics of waste activated sludge. Journal of Chemical Technology & Biotechnology: International Research in Process, Environmental & Clean Technology, 76(4), 377-382.

Woolley, S. M., Cottingham, R. S., Pocock, J., & Buckley, C. A. (2014). Shear rheological properties of fresh human faeces with different moisture content. Water SA, 40, 273-276.

3 SORPTION ISOTHERMS

The sorption isotherms can be used as a valuable tool to characterize the drying process and the material to dry. It can provide valuable thermodynamic data, such as water activity, from which important information about moisture boundness can be obtained. This section presents the experimental work conducted in this work to determine the sorption isotherms of faecal sludge samples and model them.

3.1 INTRODUCTION

Sorption isotherms are a thermodynamic property that relates the relative humidity (RH) of the atmosphere surrounding the solid and its moisture content (MC) at a constant temperature in equilibrium. At equilibrium, the RH is equal to water activity, which can be used to estimate the nature of moisture boundness in solids. Furthermore, information from the sorption isotherms can be used to deduce other thermodynamic properties such as the heat of sorption which is used to calculate energy of drying, beneficial in developing and optimizing drying technologies.

This research evaluates two different experimental methods used to obtain the sorption isotherm data, from a range of sanitation waste matrices, at different temperatures, to determine the impact of onsite sanitation system and implications of using low grade heat to dry faecal sludge. The isotherm data will be used to construct sorption isotherms which will be used to determine the nature of moisture boundness in the samples at different moistures. The effect of the source of faecal sludge (FS) and temperature is investigated in this research. Furthermore, the sorption isotherm data is fitted to isotherm models previously developed for food products.

3.2 METHODOLOGY

3.2.1 Experimental results

3.2.1.1 Characterization of the samples

The samples investigated in this research were sourced from different onsite technologies, samples VIP1, VIP2, UD1 and UD2 were sourced different ventilated improved pit (VIP) latrines and urine dry diversion toilets (UDDTs) located on the outskirts of eThekwini Municipality, South Africa. Sample UD1 was collected from an active vault and UD2 was collected from a dehydrating or stand-alone vault. Septic tank (ST) septage ST1 with grey water (GW) and ST3 with black water (BW) were collected from different ST located in Pietermaritzburg, South Africa. A composite sample for ST1 and ST3 each was formed by mixing four different samples collected after increments of 25% discharge from the individual ST. All the samples were transported to the laboratory in the WASH R&D Centre, at the University of KwaZulu-Natal (Howard College campus) and stored in a cold room maintained at 4°C before use in experimentation.

Before sorption isotherm experimentation, an initial physicochemical characterization was conducted to differentiate the faecal sludge matrices. Total solids (TS), volatile solids (VS) and Chemical Oxidation Demand (COD), pH, and electrical conductivity (EC) were measured. These results are summarized detailed in Table 3.1.

Table 3.1. Results from faecal sludge (FS) characterization

Sample	TS	VS	VS/TS	pH	EC	Total COD
	g/g	g/g	-	-	mS/cm	g/g
VIP1	0.2081	0.0724	0.3478	3.88	0.5937	1119
VIP2	0.2677	0.1399	0.5227	3.64	0.4678	856
UD1	0.2131	0.0961	0.4509	3.62	0.5438	423
UD2	0.2862	0.1170	0.4089	4.73	0.5320	325

3.2.1.2 Determination of the sorption isotherms

The two methods used to obtain the sorption isotherm experimental data were the static gravimetric method using saturated salt solutions and a water activity meter (*AquaLab, TDL*).

Saturated salt solution

The saturated salt solution method has been widely reported for different type of agricultural products. Thus it has gained acceptance and has been recommended as the standard method of determining moisture sorption isotherms of food and agricultural products. Furthermore, Bourgalt et al (2019); Remington et al (2020); Vaxelaire (2001) have previously successfully used the saturated salt solution method for faecal sludge, fresh faeces and sewage sludge respectively.

Samples will be placed in a saturated salt solution containment in an atmosphere with specific humidity. Saturated salt solutions with relative humidities ranging from 0-98,6% will be prepared. They will reach equilibrium with the specific atmosphere without any mechanical agitation of the atmosphere surrounding the sample or the sample. A water bath/oven will produce different temperature environments. The proposed temperatures are 20℃,35℃,50 ℃ and 65 ℃. The experiment will be conducted in triplicates for each different type of salt solution at five different temperatures.

Aqualab TDL water activity meter

The AquaLab water activity meter is the fastest instrument to available that gives water activity readings, it retrieves reading in 5 minutes or less. It is also the most accurate measurement tool with a 0,0003 water activity (Meter Group Inc, 2018).

Samples will be placed in a water activity meter. The water activity meter measures the RH of the air in the sample chamber by emitting a finely tuned near-infrared laser beam across the headspace and retrieves the water activity at a specific equilibrium moisture content (EMC). The operating temperature for the water activity meter is limited to 50℃. Thus the water activity will be investigated at temperatures of 20℃, 35℃ and 50℃ and compared with results from the saturated salt solution experiments at the same temperatures. Note that only the static gravimetric method was used to generate data at 65°C due to the operational temperature limitations of 50°C by the water activity meter.

Initially, the water activity meter was used to investigate the difference between samples collected from the same type of onsite technologies UD1, UD2, and VIP1, VIP2 at room temperature. It was determined that FS sourced from different VIPs or UDDTs did not affect the sorption isotherms. Hence, the experiments that preceded were conducted using only VIP2, UD1, ST1, and ST3 samples which are referred to as VIP, UDDT, ST-wGW and ST-BW in this report.

3.2.2 Modelling

Nine models (listed in Table 3.2) identified in the initial stages of research were fit to experimental data for their applicable water activity ranges using non-linear regression in Excel. Various regression parameters were deduced through this method. Regression parameters and model equations were used to predict data which was compared to experimental data. Model validation criteria *RSE*, *RMSE*, *MAPE* and plot of residuals were used to ensure the validity of the models.

Table 3.2. Mathematical Isotherm models applied to experimental data

Model		Water activity range (a_w)	Equation
1.	Brunauer – Emmett – Teller (BET) Model	$a_w < 0,5$	$M_W = \frac{M_0\, C a_w}{(1 - a_w)(1 + (C - 1)a_w)}$
2.	Guggenheim -Anderson-de Boer Model	$0,03 < a_w < 0.95$	$M_W = \frac{M_0\, C\, K a_w}{(1 - K\, a_w)(1 - K\, a_w + C\, K\, a_w)}$
3.	Smith Model	$0,5 < a_w < 0.95$	$M_W = C_1 + C_2 \ln(1 - a_w)$
4.	Halsey	$0 < a_w < 1$	$M_W = M_0 \left(-\frac{A}{RT \ln a_w}\right)^{\frac{1}{n}}$
5.	Oswin	$0 < a_w < 1$	$M_W = C\left(\frac{a_w}{1 - a_w}\right)^n$
6.	Henderson	0< a_w <1	$M_W = \left(-\frac{\ln(1 - a_w)}{C}\right)^{\frac{1}{D}}$
7.	Peleg	$a_w < 0,95$	$M_W = A a_w{}^C + B a_w{}^D$
8.	Ferro fontan	$0,1 < a_w < 0.91$	$\ln \frac{A}{a_w} = B(M_W{}^{-})$
9.	Lewicki	0<aw<1	$M_W = A\left[\frac{1}{a_w} - 1\right]^{B-1}$

3.3 RESULTS

3.3.1 Method Comparison

The static gravimetric method generated points limited to a water activity between 0.1 and 0.9 in all the samples investigated. Thereby limiting the full extent of the free and unbound water region in the sorption isotherm. The water activity method provided the full extent of the sorption isotherms with points in the unbound, multilayer, and monolayer water region for samples with initial MC less than 80%(w.b) such as the UDDT and VIP sample (Figure 3.1 and Figure 3.2). Statistical analysis through multiple regression analysed the impact of the water activity and the static gravimetric method on the EMC (Table 3.2). Independently, the water activity meter and the static gravimetric method significantly impact the EMC ($p \leq 0.05$). Generally, the methods were not significantly different ($p \geq 0.05$) suggesting that the choice of method has no impact on the EMC. Only at 35°C the EMC measured using the water activity meter and the static gravimetric method on the UDDT sample were significantly different ($p \leq 0.05$) which is most likely due to inconsistency during the experimentation process (Table 3.2).

Furthermore, the moisture sorption isotherms of sample ST-wGW were investigated at temperatures of 35°C and 50°C and temperatures of 25°C, 35°C and 50°C for the ST-BW sample. A comparison of methods was made for these samples however, it was increasingly difficult to obtain sufficient data points from drying the samples in the oven to different MC consistent with the water activity method due to both samples having a low solids concentration. In turn, no data points for a water activity range of 0.2 to 0.9 were obtained using the water activity meter method. Thus, results from the comparison of the static gravimetric method and water activity method were not reliable. However, information about the boundness of moisture for water activity ranges of 0.1 to 0.9 using the static gravimetric method and water activity ranges less than 0.1 and greater than 0.9 using the water activity method (Figure 3.3 and Figure 3.4). It is therefore important to use a combination of the water activity method and the static gravimetric methods when analysing samples with a MC greater than 80%(w.b) such as ST-wGW and ST-BW due to the samples low solid concentration.

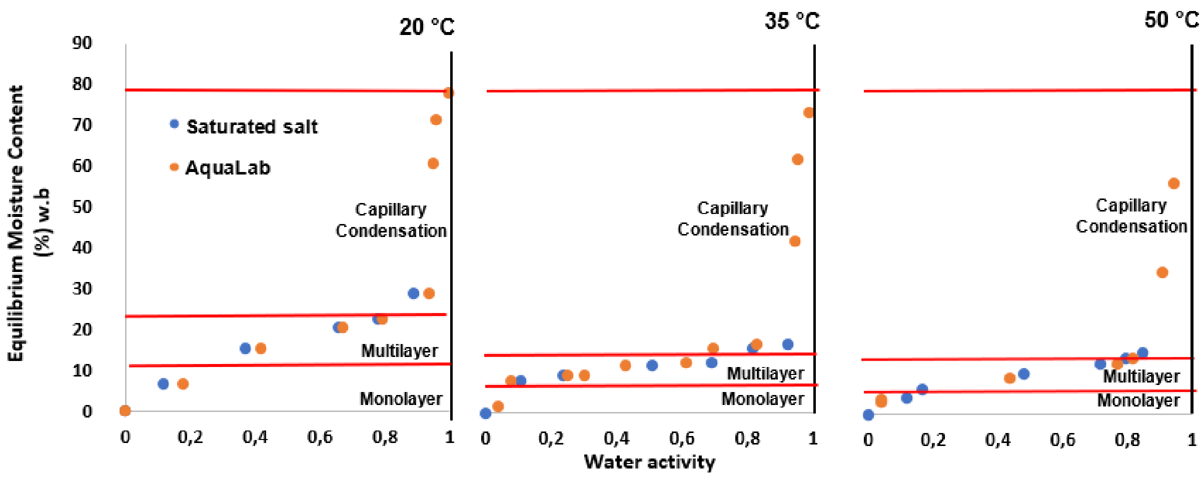


Figure 3.1 Comparison of methods in UDDT

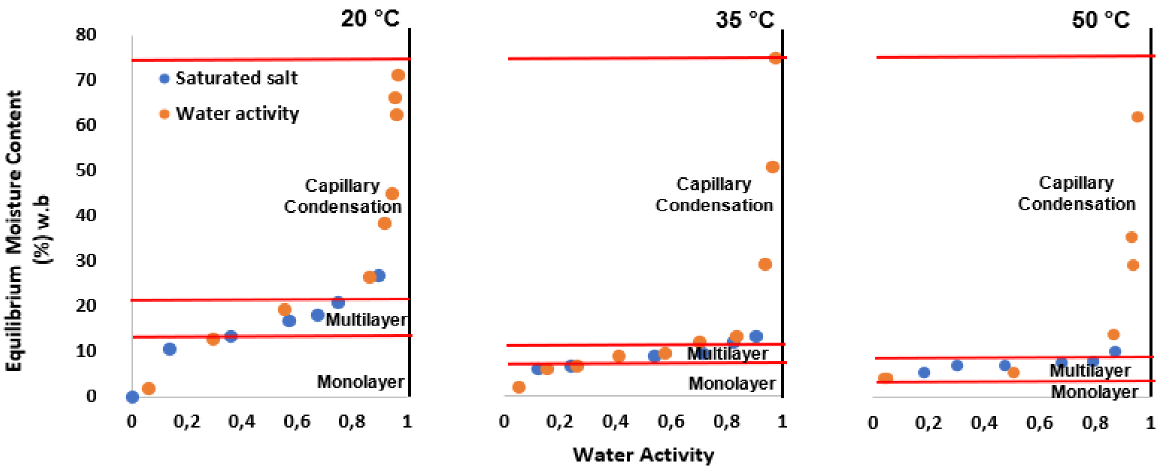


Figure 3.2. Comparison of methods in VIP

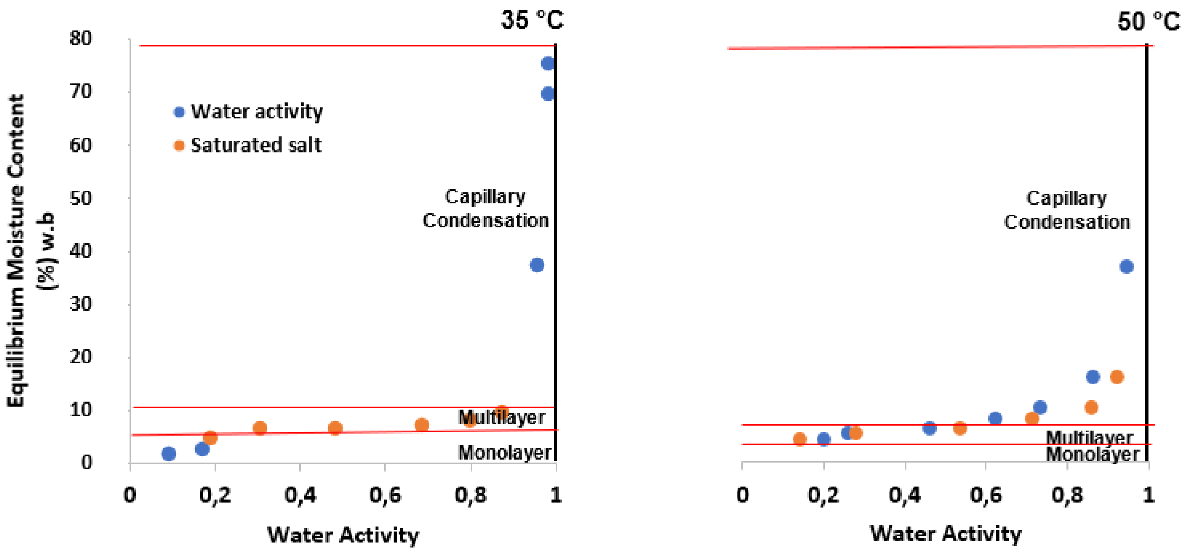


Figure 3.3. Comparison of methods in ST-wGW

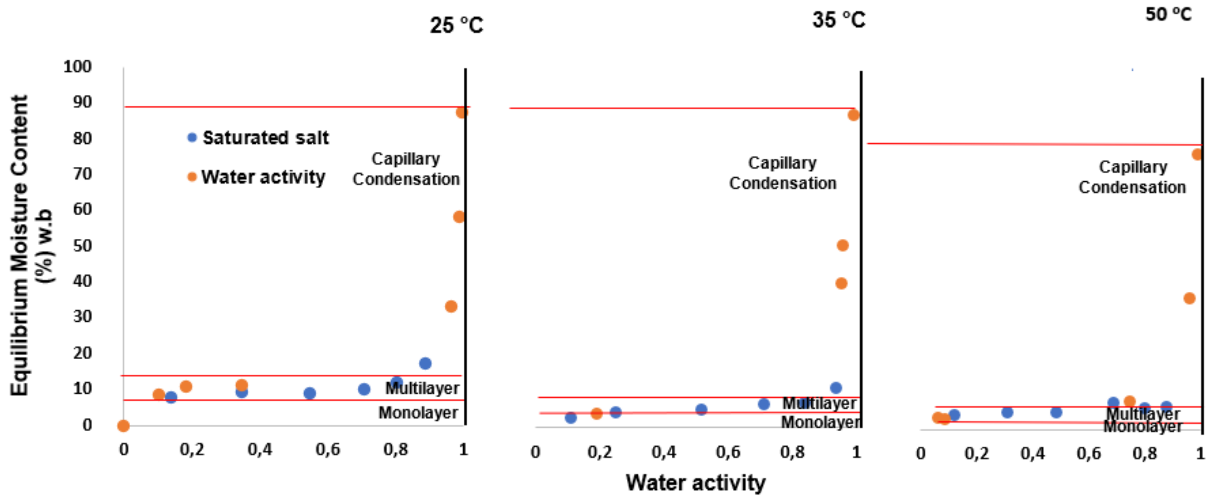


Figure 3.4. Comparison of methods in ST-BW

Table 3.3. Summary of regression coefficients from multiple regression with the equilibrium MC of the UDDT, VIP, ST-wGW and ST-BW samples as a dependent variable and the water activity and method of experimentation as predictor variables

			Coefficients	Standard Error	P-value
UDDT	50 °C	Intercept	-0.1136	0.1104	0.3306
		Method	0.0662	0.0626	0.3181
		Water Activity	0.3030	0.09395	0.0104
	35 °C	Intercept	-0.3976	0.1419	0.0207
		Method	0.429	0.0867	0.0207
		Water Activity	0.998	0.1231	0.00284
	20 °C	Intercept	-0.2001	0.1797	0.2916
		Method	0,.785	0.1035	0.4658
		Water Activity	0,.455	0.1719	0.0099
VIP	50 °C	Intercept	-0.1908	0.1236	0.1537
		Method	0.133	0.0699	0.1360
		Water Activity	0.2956	0.1047	0.01809
	35 °C	Intercept	-0.3115	0.1663	0,08564
		Method	0.1372	0.081499	0.1183
		Water Activity	0.4248	0.126474	0.005684
	20 °C	Intercept	-0.2459	0.120632	0.064213
		Method	0.1373	0.067845	0.065772
		Water Activity	0.4870	0.107139	0000672
ST-wGW	50 °C	Intercept	-0.4722	0.1570	0,0168
		Water Activity	0.5043	0.1381	0.0065
		Method	0.2635	0.0934	0.0225
	35°C	Intercept	-0.4623	0.2462	0.0801
		Water Activity	0.5638	0.2075	0.0159
		Method	0.2254	0.1301	0.1037
ST-BW	35°C	Intercept	-0.4605	0.1752	0.0340
		Water Activity	0.4010	0.1784	0.0593
		Method	0.3058	0.1189	0.0369
	25°C	Intercept	-0.3890	0.1660	0.0437
		Water Activity	0.4567	0.1375	0.0089
		Method	0.2328	0.0929	0.0336

3.3.2 Effect of temperature on sorption isotherms

Samples exhibited a type II sigmoid isotherm, a classic water sorption isotherm observed in multilayer sorption through physisorption (Shaw, 1992). Type II moisture isotherms are symbolized by a concave down before an inflection point and a concave up curve after the inflection point. Samples UDDT, VIP , ST-wGW and ST-BW showed a concave down shape at water activity values of 0 to 0.5, and a concave up shape was observed at water activity values of 0.5 to 1; whereby, an initial steep concave-down curve on the graph represents the strongly bound monolayer sorption of water molecules held to the surface of the solid by Van der Waals forces. This is followed by a less steep progression of the concave down curve and the start of the concave up curve, representing the less strongly bound multilayer sorption of water molecules on the surface of the solid. Capillary condensation of bound water molecules where the pores in the solid become filled with condensed liquid from the vapour phase below the saturation pressure of the pure fluid because of the increased Van der Waals interaction present inside the capillary is observed where there is a steep concave up curve that tends to an asymptote. Free water is observed at a water activity of 1 (Shaw, 1992). Table 3.3 shows the MC of different samples exposed to different temperatures.

Statistical analysis through multiple regression shows that temperature significantly impacts the EMC ($p \leq 0.05$) for UDDT, VIP and ST-BW samples (Table 3.4). A lower EMC is observed when samples are exposed to elevated temperatures (Figure 3.5 to Figure 3.8). This is more apparent in the monolayer and multilayer region of the isotherm. Samples have the similar EMC values in the unbound region at a water activity greater than 0.9 region where the isotherm tends to a vertical asymptote. The effect can be observed clearly when using the static gravimetric method which limits the sorption isotherms to a water activity less than 0.9.
However, the temperature did not have a significant impact on the EMC of the ST-wGW sample ($p \geq 0.05$) (Figure 3.4 and Figure 3.7).

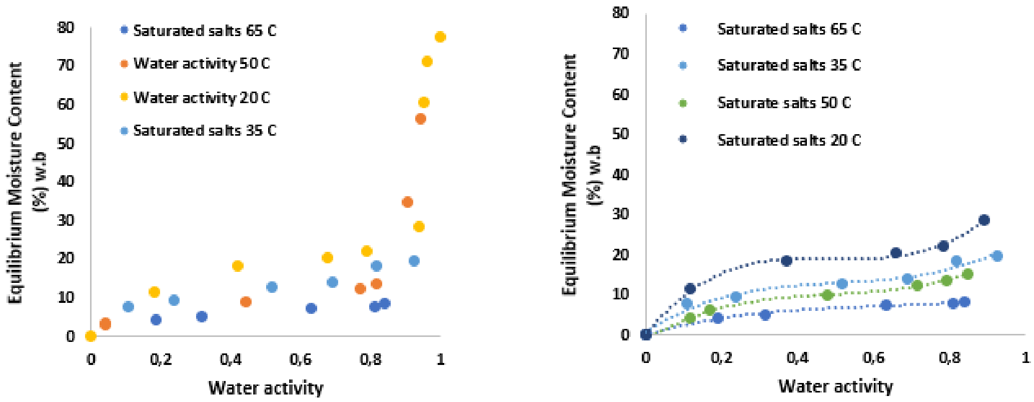


Figure 3.5. Effect of temperature on the UDDT sample

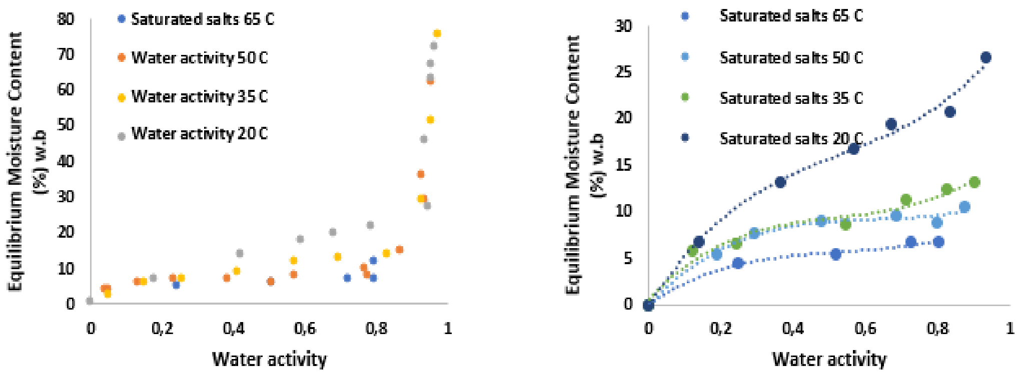


Figure 3.6. Effect of temperature on VIP

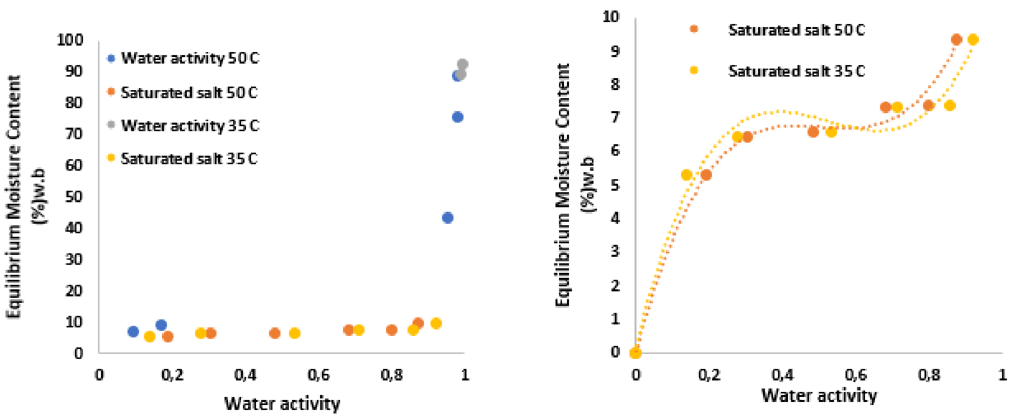
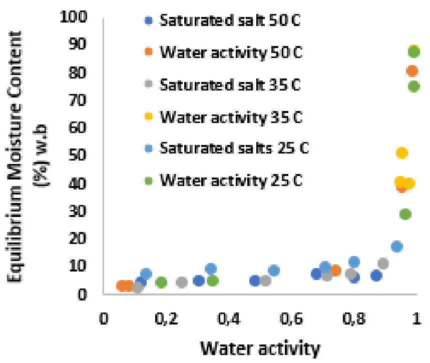


Figure 3.7. Effect of temperature on ST-Wgw



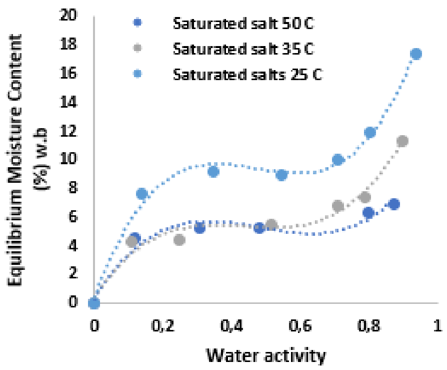


Figure 3.8. Effect of temperature on ST-BW

Table 3.4. MC for different samples at different temperatures

Sample name	Temperature	Nature of boundness	Water Activity (-)	MC (%) w.b
UD 1	20	Monolayer	0 to 0.2	0 to 12
	35			0 to 6
	50			0 to 4
	20	Multilayer	0.2 to 0.8	12 to 24
	35			6 to 14
	50			4 to 13
	20	Capillary condensation	0.8 to 1	24 to 78
	35			14 to 78
	50			13 to 78
VIP 2	20	Monolayer	0 to 0.1	0 to 15
	35			0 to 7.5
	50			0 to 3
	20	Multilayer	0.1 to 0.75	15 to 22
	35			7.5 to 10
	50			3 to 8
	20	Capillary condensation	0.75 to 1	22 to 75
	35			10 to 75
	50			8 to 75
ST 1	35	Monolayer	0 to 0.07	0 to 3
	50			0 to 2.5
	35	Multilayer	0.05 to 0.8	3 to 10
	50			2.5 to 7.5
	35	Capillary condensation	0.8 to 1	10 to 96
	50			7.5 to 96
ST 3	25	Monolayer	0 to 0.08	0 to 5
	35			0 to 2.5
	50			0 to 2.5
	25	Multilayer	0.08 to 0.75	5 to 10

	35			2.5 to 7.5
	50			2.5 to 5
	25	Capillary condensation	0.75 to 1	10 to 88
	35			7.5 to 88
	50			5 to 88

Table 3.5. Summary of regression coefficients from multiple regression with the equilibrium MC of the VIP 2 and UD 1 sample as a dependent variable and the water activity, method, and temperature as predictor variables

			Coefficients	Standard Error	P-value
VIP 2	Water activity	Intercept	-0.10002	0.096762	0.307503
		Water Activity	0.392661	0.062043	1.63E-07
		Temperature	-000352	0,001663	0.040674
UD 1	Saturated Salts aw ≤ 0.9	Intercept	0,12025	0.024066	0.000196
		RH	0.174567	0.022099	1.59E-06
		Temperature	-0.00221	0.000533	0.000993
	Water activity 0 ≤ aw ≤ 1	Intercept	0.082403	0.146385	0.581807
		Water Activity	0.493985	0.114587	0.000618
		Temperature	-0.00369	0.003073	0.248085
ST 1	Saturated Salts aw ≤ 0.9	Intercept	-0.50153	0.301662	0.108893
		RH	0.537583	0.130979	0.000379
		Temperature	0.000849	0.0057	0.882831
ST 3	Saturated Salts aw ≤ 0.9	Intercept	0.097799	0.020475	0.000245
		RH	0.074905	0.01724	0.000577
		Temperature	-0.00169	0.000464	0.002344

3.3.3 Modelling Sorption Isotherm Results

Of the nine models fit to the sorption isotherm experimental data, the Peleg, Ferro Fontan, and GAB models fit UDDT and VIP sorption isotherm experimental data the best for temperatures of 20°C, 35°C and 50°C (Figure 3.9 and Figure 3.10) These models had a Mean Percentage Error (MAPE) of less than 10% (Table 3.5) and their plot of residuals did not show a pattern thereby validating the models further. The Peleg and GAB models fit the ST-wGW experimental data at 35°C and 50°C (Figure 3.11) and ST-BW experimental data at 25°C and 35°C (Figure 3.12) the best with a MAPE less than 10% (Table 3.4).

Parameters deduced from the GAB model were used to estimate the monolayer capacity (Mo) and the heat of sorption from the energy constants C and K. Energy constant C is a partition function of the first molecule attached on a monolayer and the first molecule attached on a multilayer, and is a measure of strength of t water bound on the monolayer. The higher the value of C the stronger water is bound to the monolayer. K is the ratio partition function of molecules found in free water and molecules found in the multilayer. A value of K close to one indicates that there is no distinction between molecules of water existing in free water and molecules in the multilayer, the more sorbed molecules are structured in a multilayer, the lower value of K. Energy constants C and K were used to deduce the monolayer and multilayer enthalpy, the difference of the

two was estimated to be the net isosteric heat of sorption (Quirijns, et al., 2005). Results are summarized in Table 3.6. Furthermore, the impact of temperature on GAB parameters was investigated. The impact of temperature could not sufficiently be determined due to a lack of data points (Figure 3.13 to Figure 3.15). A high energy constant C and an energy constant K close to but less than 1 is observed for VIP, UDDT and ST-BW samples suggesting that the samples are characterized by strongly bound water and a slightly structured multilayer that has characteristics comparable to the molecules observed in free water.
The Ferro Fontan model was identified as the isotherm model that fits the UDDT and VIP data the best, while the Peleg model was identified the isotherm model that fits ST-wGW and ST-BW the best. Thus, these models were used along with the Clausius Clapeyron equation to estimate the isosteric heat of sorption as a function of MC (Figure 3.16) for the samples. The isosteric heat of sorption varies and inversely with the MC. Higher MC have isosteric heat of sorption nearing 0 suggesting that at these MC the isosteric heat is equal to the heat of vaporization of pure water. The heat of sorption as a function of MC was evaluated at the mean temperature 35°C (Figure 3.17). The results indicate that bound water is limited to MC of 60% in UDDT and VIP sample and monolayer 43% (w.b) in the ST-BW sample.

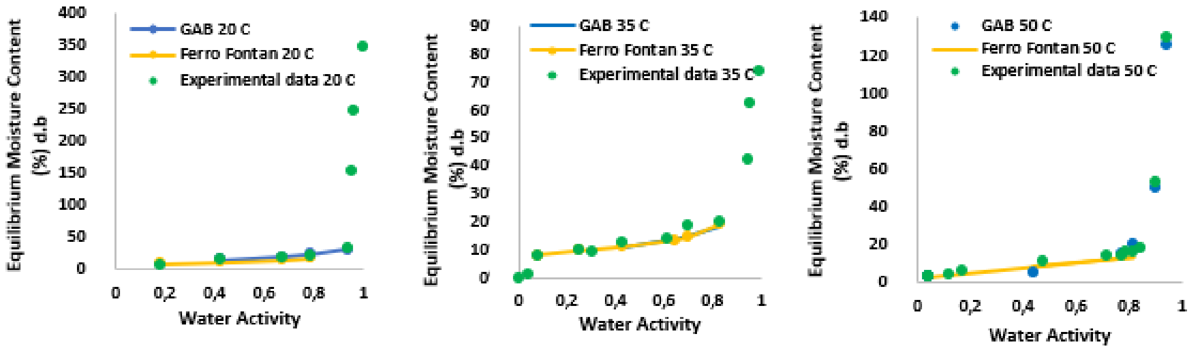


Figure 3.9. Model data fit UDDT experimental data at 20, 35 and 50°C

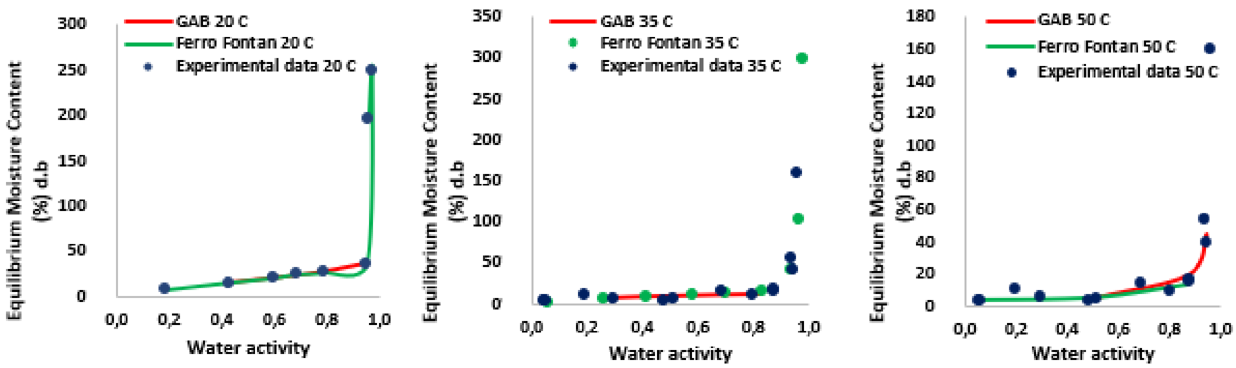


Figure 3.10. Model data fit VIP experimental data at 20, 35 and 50°C

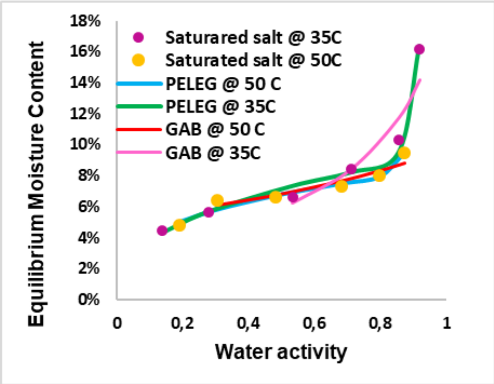


Figure 3.11. Model data fit ST1 experimental data at 35 and 50°C

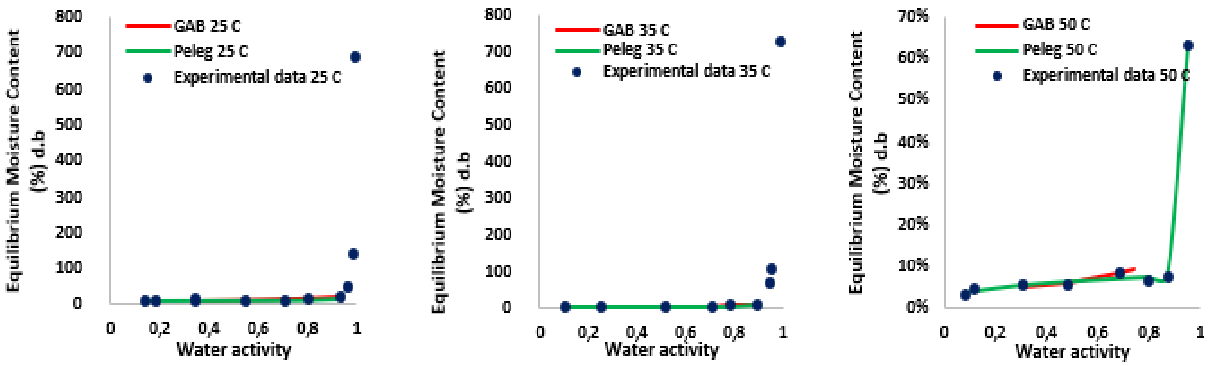


Figure 3.12. Model data fit ST-BW experimental data at 25, 35 and 50°C

Table 3.6. Error criterion used to select models that showed the best fit to sorption isotherm experimental data

Sample	T (°C)	Model		Model Validation Criteria	
			Water Activity Range	MAPE	RMSE
UD 1	20	PELEG	<0.95	5.5632	0.0715
	35	FERRO FONTAN	0.1-0.9	0.7238	0.0035
VIP 2	20	PELEG	<0,95	0.4730	0.0023
	35	FERRO FONTAN	0.1-0.9	9.9615	0.01957
	50	GAB	0.3-0.95	0.8348	0.0030
ST 1	35	PELEG	<0.95	2.8857	0.0474
	50	PELEG	<0.95	2.2204	0.0022
ST 3	25	PELEG	<0.95	3.0105	0.0053
	35	PELEG	<0.95	2.0456	0.0338

Table 3.7. GAB model parameters for UDDT, VIP and ST-BW at different temperatures.

Sample	UDDT			VIP			ST-BW		
	20°C	35°C	50°C	20°C	35°C	50°C	25°C	35°C	50°C

Monolayer capacity Mo (kg/kg) d.b	0.105	0.074	0.032	0.158	0.159	0.024	0.063	0.030	0,038
CX 10^6	1.017	0.89	0.89	6.39 X10^{-6}	24.5X 10^{-6}	9.04 X 10^{-3}	6.205	0.363	0.363
K	0.702	0.72	1.035	0.635	0.12634	1.01	0.703	0.834	0.792
Monolayer enthalpy H1 (kj/mol)	78.0	76.2	78.0	51.9	57.3	70.8	103	80.8	82.5
Multilayer enthalpy Hm (kj/mol)	46.9	44.6	44.9	47.2	48.9	44.9	46.8	46.0	46.0
Net isosteric heat(kj/mol)	33.8	32.6	35.1	77.5	14.1	27.9	58.8	37.3	39.6

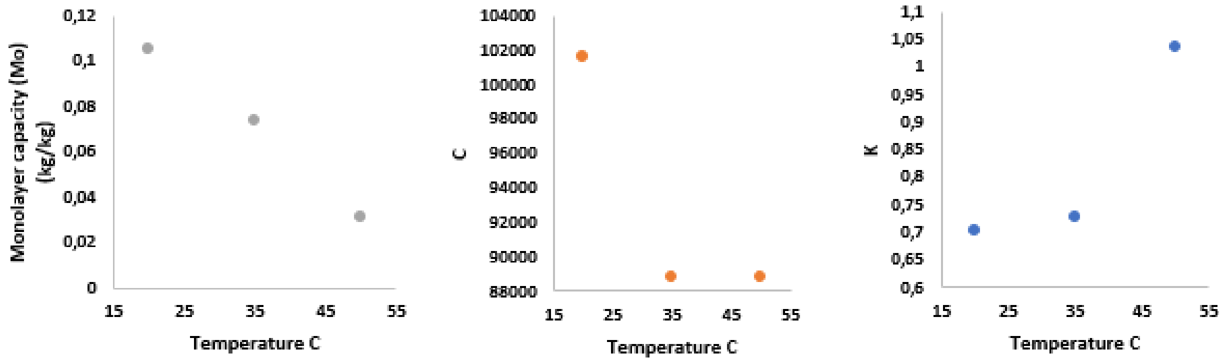


Figure 3.13. GAB parameters estimated through non-linear regression for UDDT sample at different temperatures

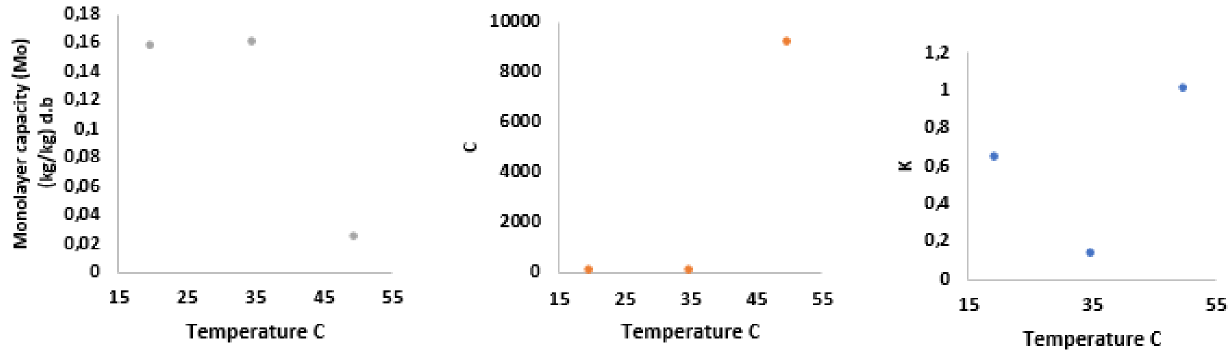


Figure 3.14. GAB parameters estimated through non-linear regression for VIP sample at different temperatures

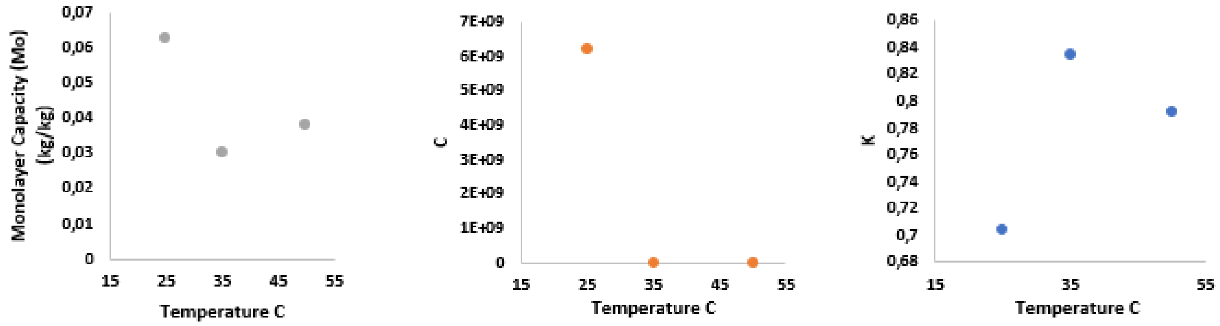


Figure 3.15. GAB parameters estimated through non-linear regression for ST-BW sample at different temperatures.

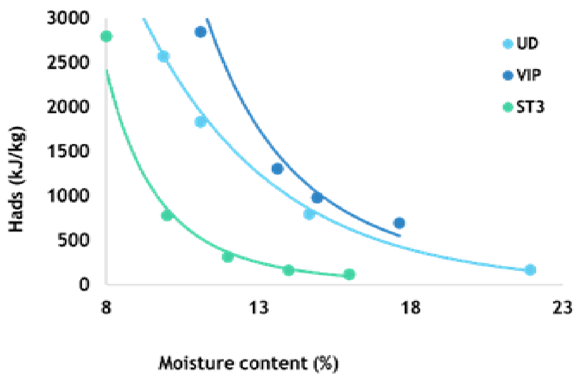


Figure 3.16. Net isosteric heat varied with MC (w.b) of UDDT, VIP and ST-BW samples

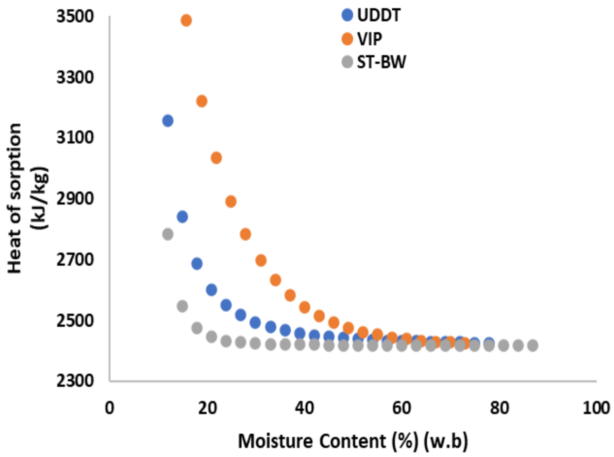


Figure 3.17. Heat of sorption of UDDT, VIP and ST-BW samples estimated at 35°C

Table 3.8. Energy requirements for the different FS samples.

Sample	Treatment goal	Water activity	MC (%) w.b	Energy kJ/kg
UDDT	Pathogen inactivation	<0.6	10 to 16	1640 t0 2379
VIP			10 to 16	7240 to 9092
ST-BW			7 to 9	620 to 2000

A general comparison of the samples investigated in this research shows that samples with a low solid concentration ST-BW require lower amounts energy as compared to UDDT and VIP samples for temperature of 20 to 50°C (Figure 3.16 and Figure 3.17). Furthermore, a lower solids concentration corresponds to a lower MC in terms of achieving the treatment goal of pathogen inactivation (Table 3.8).

3.4 CONCLUSION

In conclusion the static gravimetric and water activity methods employed in this research generate similar sorption isotherms. Samples with an initial MC less than 80% such as UDDT and VIP reach a MC of 24 and 22%(w.b) respectively at room temperature. Samples with an initial MC greater than 80% reach a MC ST-wGW and ST-BW of 10% and 7.5% (w.b) at room temperature. This information assists in predicting the final MC of samples dried at room temperature such as drying beds. At elevated temperatures, the final MC decreased, suggesting that at higher temperatures more moisture is lost to the atmosphere. The net isosteric heat deduced from the sorption suggests that more energy required to dry samples at lower MC associated with monolayer and multilayer sorption. At MC greater than 24., 22 and 10% (w.b) for the UDDT, VIP and ST-BW samples, the net isosteric heat of sorption starts approaching zero suggesting that moisture is less unbound these MC. Bound water is limited to MC of 60% and 43% (w.b) in UDDT an ST-BW samples respectively. While no free water was identified in the VIP sample. Pathogen activation is an end treatment goal in the drying process and it is achieved at a water activity values of 0.6.(insert Source) UDDT and VIP samples would have to be dried to MC ranging 10 to 16% (w.b) from their initial MC, this translates to an energy of 1640 to 2379 kj/kg for UDDT samples and 7240 to 9092 kj/kg in VIP samples excess of the heat of vaporization of pure water at the desired temperature. ST-BW requires an excess energy of 620 to 2000 kj/kg to dry the sample from 88% (w.b) to 7-9% for pathogen inactivation to take place.

3.5 REFERENCES

Andrade P, R. D., Lemus M, R., & Perez C, C. (2011). Models of sorption isotherms for food : Uses and limitations. *Universidad de Antioquia, Medelln, 18*(3), 325-354.

Brunauer, S., Deming, L. S., Deming, W. E., & Teller, E. (1940). On a theory of Van der Waals adsorption of gases. *American Chemical Society Journal*(62), 1723-1732.

John Worley, P. o. (2016). GREENHOUSES Heating, Cooling and Ventilation. *UGA Extension*, 12.

Makela, M., Edler, J., & Geladi, P. (2017). Low-temperature drying of industrial biosludge with simulated secondary heat. *Applied Thermal Engineering, 116*, 792-798.

Meter Group Inc. (2018). *Operator's Manual for Series 4 : AquaLab water activity.* Washington: Meter Group Inc.

Niwagaba, C. B., Mbeguere, M., & Strande, L. (2014). Faecal sludge quantification, characterization and treatment objectives. In *Faecal Sludge Mangement Systems: Approach and Implementation* (pp. 19-44). London: IWA Publishing.

Oswin, C. R. (1946). The kinetics of package life.III.Isotherm. *J Soc Chem Ind, 65*(12), 419-421.

Quirijns, E. J., van Boxtel, A. J., van Loon, W. K., & van Straten, G. (2005). Sorption isotherms, GAB parameters and isosteric heat of sorption. *Journal of the Science of Food and Agriculture, 85*, 1805-1814.

Shaw, D. J. (1992). *Introduction to Colloid and Surface Chemistry* (4th ed.). Oxford: Butterworth and Heinemann.

Vaxelaire, J. (2001). Moisture sorption characteristics of waste activated sludge. *Journal of Chemical Technology and Biotechnology, 76*(4), 377-382.

Vaxelaire, J., & Cezac, P. (2004). Moisture distribution in activated sludges. *Water Research, 38*(9), 2215-2230.

4 MOISTURE BOUNDNESS CHARACTERISATION

The previous sections (2 and 3) provided a general representation of moisture boundness in faecal sludge by identifying the different types of moisture, their distribution and their influence on the properties of the faecal material. This section allows to confirm the results already presented through a rigorous experimental work and complement it with information to understand moisture boundness with a deeper insight level.
It has to be mentioned that the experimental work and data analysis related to this section is still in course. Therefore, the results presented in this section are incomplete and the conclusions are partial. The complete set of results and analysis will be presented in the thesis of the PhD students, which is expected to be available by the end of 2023 or beginning of 2024. Once published, the dissertation will be available for consultation through the link displayed in the Supporting Document section. An updated version of this report with the missing results might also be uploaded in the library.

4.1 INTRODUCTION

The moisture content (MC) of FS varies from 99% to 70% depending upon various factors, mainly on the use of water for anal cleansing (Diener et al., 2014). For effective treatment of FS, the solid-liquid separation becomes an indispensable step as it decreases FS volume for cheaper transportation, reduced treatment footprint, and improved resource recovery potential of FS treatment products (Gold et al., 2016). However the removal of water from the solid phase is complicated by the interaction between the solid particles and water molecules, (termed as Moisture Boundness) whereby binding energies increase with increasing solids concentration. There is a need to understand bound water and the associated binding energies over a solids concentration profile and their influencing factors to effectively design water removal processes such as dewatering and drying technologies. This has been extensively investigated for sewered centralised sludges. However this knowledge is still limited for non-sewered decentralised sludge matrices.

There are apparent reasons for limited solid-liquid separation of sludge. Most of the free moisture and part of loosely bound moisture is removable, and the other factors which affect solid-liquid separation include the following (Mowla et al., 2013):

a. Settleability – Low settleability of solid particles, which are colloidal in nature
b. Compressibility – High compressibility of the solid particles
c. Permeability – Restricted water movement within the solids' matrix
d. Moisture boundness – High water-retention capacity of sludge

Various other properties affect the moisture boundness, amongst which are the solids particle size, particle size distribution, the zeta-potential and the chemical composition of the sludge, which vary according to the type of onsite sanitation (OSS).

FS contains moisture which can be categorically differentiated into two types (Schaum and Lux, no date). These categories are distinguished by the kind of bonds they form (Flaga, no date). These types are (Figure 4.1 and Figure 4.2):

1. Free moisture or unbound moisture – not bound to the particles. The free MC is not adsorbed by the sludge particles, and moves freely between the flocs. It can be separated by gravitational settling.
2. Bound moisture
    a. Interstitial moisture or capillary moisture – bound by capillary forces in-between the flocs
    b. Vicinal moisture or surface moisture – bound by adhesive and adsorption forces at the surface

c. Water of hydration or Intracellular moisture – chemically bound to the solid matrix, and bio-chemically bound within the cell body

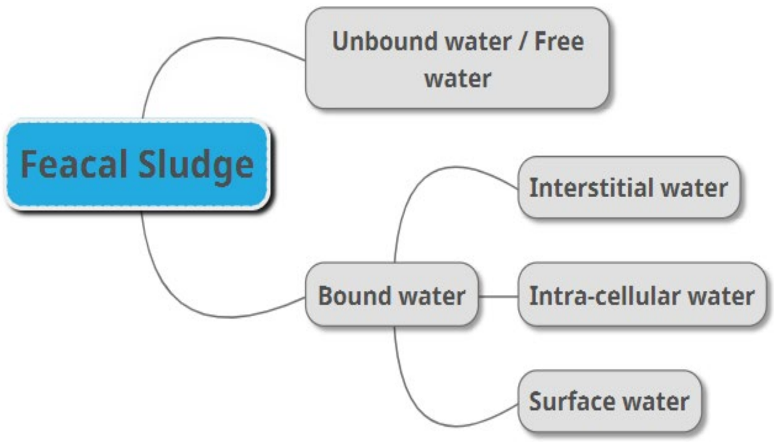


Figure 4.1. Different types of moisture in sludge

The bound moisture is the sum of interstitial moisture, vicinal moisture and water of hydration. The interstitial moisture is that moisture which is trapped within the sludge flocs. Experiments have shown that it is bound by active capillary forces which are weak forces. Also, osmotic pressure within the sludge flocs is another component of interstitial moisture (Mikkelsen and Keiding, 2002). The vicinal moisture is bound by adsorptive and adhesive forces, and can be separated only by disintegrating the floc, input of energy (thermal) or by disrupting the cell charge. The vicinal moisture is bound electro-statically to the solid particles, and cannot be removed by mechanical forces without pre-preparation. The water of hydration or intracellular moisture is contained within the cell bodies as water of hydration and is chemically bound to the solid matrix and bio-chemically bound to the solid particles (Kopp and Dichtl, 2001). There is little research on moisture in FS.

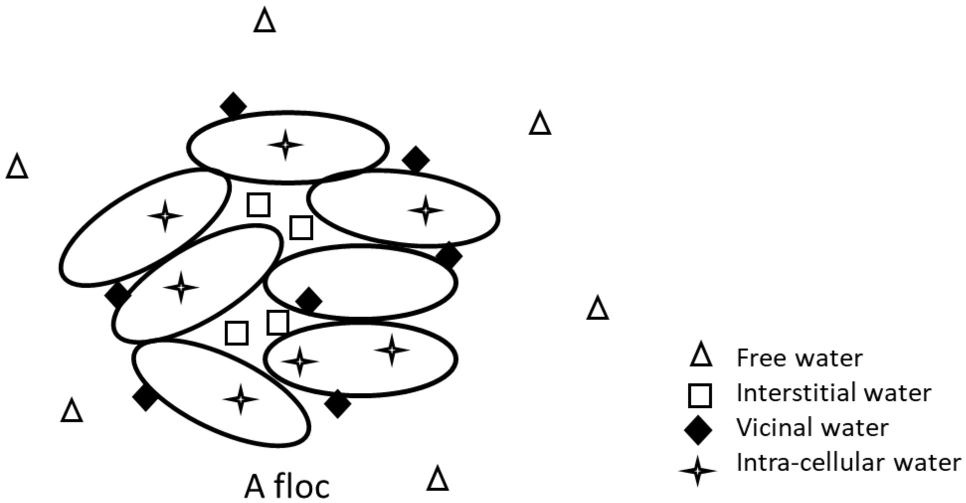


Figure 4.2. The moisture and its bonding in sludge (Chen, Yue and Mujumdar, 2002)

Further, the behaviour of the types of moisture in sludge is different. Unbound moisture vaporizes and freezes normally as water, at normal freezing temperatures. Thermodynamically, the bound moisture does not behave as pure water, as the chemical potential and binding strength are naturally different (Wu, 1998).The interstitial moisture because of the flocs and high dissolved solids concentration, freezes at temperature lower than the normal freezing temperature of water. The vicinal moisture freezes at very low temperatures, and the intracellular moisture do not enter the ice crystal lattice upon freezing. It can only be removed thermally above 105°C (Vesilind and Hsu, 1997).

4.2 METHODOLOGY

The research methodology for the investigation on moisture boundness was classified into 2 sections. This classification was primarily intended to structure the research and investigations.

4.2.1 Part 1: Investigate the type and proportions of moisture boundness of FS

Some of the mechanical dewatering technologies include belt press, screw press, filter press, centrifuge press, etc., and the standard non-mechanical method adopted to dewater is the use of drying beds (Alena et al., 2020; Schaum & Lux, n.d.). However, the conventional mechanical dewatering technologies reach not more than 15-25% of the dry solids content of sludge, mostly owing to the high bound MC and stabilized aggregation of microbes as colloids (Christine et al., 2019). In contrast to wastewater sludge, there is no comprehensive knowledge available on the dewatering performance of in relation to FS characteristics (Gold et al., 2017).

For all practical purposes, the limits of dewaterability are usually taken as a measure of the unbound moisture and most of the dewatering technologies focus on the removal of this unbound moisture though some loosely bound moisture is also removed depending upon the type and extent of mechanical force exerted. Hence, dewaterability has been defined as the Total Solids (TS) after the removal of free moisture (Gold et al., 2018a). The rate of dewatering is hence defined as the rate at which the free moisture is released from the sludge.

4.2.1.1 Part 1 A – Investigate the unbound-bound moisture interface

The investigations carried out to determine the boundary of unbound-bound moisture interface are mostly the experiments of dewaterability. Also, instrumentation-based investigations are also carried out, as listed in Table 4.1.

Table 4.1. Investigations for unbound-bound moisture interface

Experiments	Description
Instrumentation based	
Differential Scanning Calorimetry (DSC)	Based on the assumption that bound water would not freeze down to a threshold temperature, the heat absorbed in the heating stage is equal to the latent heat required for freezing
Thermogravimetric Analysis (TGA)	Based on the differences between the enthalpy required to evaporate unit mass water (having the different binding energies with the sludge) and the latent heat of pure water gasification
Sequencing Electron Microscopy	Structural imagery of FS samples at different MC

4.2.1.2 Part 1 B: Investigations the proportions of moisture in bound moisture fraction

Further, the proportions of bound moisture are investigated to get an overall understanding on the boundness of moisture. These investigations are classified into two sub-categories based on the approach taken for determining the moisture boundness characteristics as detailed in Table 4.2.

Table 4.2. Bound moisture characterization experiments

Experiments	Description
Temperature based	
Drying Test	Based on the rate of evaporation which depends on the type of bonds between the moisture and the solid particles
Instrumentation based	
Dynamic Sorption Isotherms	Based on the sorption process of water molecules into a specific material at a specific temperature illustrating where water molecules are progressively and reversibly released from hygroscopic forces in biological material as a result of mainly capillary effects and direct bonding
Water activity measurements	Based on the partial vapour pressure of moisture in a material to the standard state partial vapour pressure of pure water measured at the material's temperature. A good indicator of the amount of moisture that is bonded with the dry solid structure of material
Time Domain – Nuclear Magnetic Resonance	1H nuclei in a magnetic field absorb and re-emit electro-magnetic radiation at a specific resonance frequency

4.2.2 Part 2: The properties of FS and relating/influencing moisture boundness

The different parameters of FS have different effects on the moisture boundness properties, and further these properties also co-influence each other to enhance and / or reduce moisture boundness. This part of the research is to explore and analyse such correlations and co-influences. These identified influencing properties could be altered by different pre-treatment methods which would result in the conversion of bound moisture to unbound moisture and hence improve solid-liquid separation processes.

The moisture boundness characterization of FS involves analysis of parameters which influence and/or indicate moisture retention and its movement within the sludge. These parameters are categorised into the following 2 groups:

1. Structural properties
2. Physicochemical properties

The parameters investigated under each of the groups is provided in Table 4.3.

Table 4.3. Parameters to be investigated in moisture boundness properties

Structural	Physiochemical
Extracellular Polymeric Substances (EPS)	Total and soluble COD
Rheology	Total Organic Carbon
Particle Size Distribution (PSD)	Nutrients – N & P
Penetrometry	Sludge elemental composition – CNS
X-ray tomography	

4.2.2.1 Preliminary characterization of FS from different OSS

The preliminary characterization of FS from different OSS is necessary to establish the properties of FS, and assess the sample quality in line with the samples used in the previous studies. Further, to help develop better

insight into the sludge water boundness properties, preliminary characterization of the sludge samples is necessary.
The experiments which constitute the set of preliminary characterization include the following:

1. Total Solids (TS)
2. Volatile Solids (VS)
3. pH
4. Electrical Conductivity
5. Zeta potential

4.2.3 Samples

Four types of FS samples have been collected for the analysis. These four sample-types are originating from different types of OSS system, which are most prevalent in countries across Africa and Asia. They are:

1. Faecal Sludge from Ventilated Improved Pit Latrines (VIP)
2. Faecal Sludge from Urine Diverting Dehydrating Toilets (UDDT)
3. Faecal Sludge from septic tanks with greywater (ST-wGW)
4. Faecal Sludge from septic tanks with only blackwater (ST-BW)

The samples from VIP, UDDT and ST-wGW was collected from outskirts of Durban city, with help from Khanyisa Projects and Wastewater Treatment Works. The samples from ST-BW were collected from Pietermaritzburg, with assistance from Partners in Development, Pietermaritzburg.
The VIP latrines selected for the sampling are from the peri-urban areas, under the administrative jurisdiction of eThekwini Municipality. The VIP latrines are de-sludged once in 5 years, and hence the average age of the sludge from the VIP latrine would be considered as 2.5 years. The pits are emptied manually, using shovels. There is no considerable difference in the characteristics of the sludge across the front side and the backside, and with regard to the depth, except low MC with the sludge at the bottom most fraction (Septien et al., 2018). This representative sample is collected from each pit using a spatula from the container in which the sludge is emptied from the VIP latrine. Though the sludge does have large debris, care is exercised to avoid the debris in the samples.

For the samples from the UDD Toilets, the approach adopted for sampling is similar to that employed for sampling VIP latrine samples. The samples are selected from both the active vault as well as the stand-by vault or dehydrating vault.

For samples from ST, a composite sample is prepared by collecting the septage at four different levels of flow from the tanker. Once the valve of the tanker is opened, one sample is collected immediately. The second sample is collected after 25% of the discharge, third sample after 50% of the discharge and the last sample when the final 25% of the septage is being discharged. These 4 samples are mixed to make a composite sample.

All the samples were transported and stored in the cold storage room, maintained at 4°C in closed containers to ensure the properties are not altered.

4.3 OBJECTIVE 1 – UNBOUND-BOUND MOISTURE INTERFACE

4.3.1 Introduction

The moisture distribution in the sludge has always been and will be considered an important aspect for solving their dewatering and drying challenges. Further, the selection of an efficient dewatering and drying technique is crucial for effective costing. Solid-liquid separation is mainly performed so as to reduce the MC of the sludge and thereby:

- Reduce the transportation and treatment costs
- Reduce the post-treatment and disposal costs
- Enhance better sludge handling
- Reduce production of leachate
- Increase the calorific value of the sludge for energy derivation

The moisture distribution and the types of moisture in the sludge are classified depending on their physical, chemical or biochemical bonding to the solid particles, and a clear picture of the moisture distribution in the sludge can help improve the solid-liquid separation performance. The unbound bound moisture interface or the critical moisture content (CMC) is required to understand the extent of the removal of water by dewatering. Dewatering, defined as the extent of moisture removed by physical forces, is often termed as the limit of removal of unbound moisture.

Centrifugation, by the application of centrifugal force to increase the solid-liquid separation, is one of the mechanical methods taken to determine the CMC. Instrumentation approaches – Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) are used. The Scanning Electron Microscopy (SEM) of raw samples and dried samples are compared for structural and temporal changes in the sludge samples.

4.3.2 Experimental methodology

– The **centrifugation method** was used to measure the MC of the sludge after centrifugation for different rpms. The flocs settle quickly since the gravitational acceleration is much less than the centrifugation acceleration, and with the increasing rpm and thus increasing centrifugation acceleration, the sludge compressed till the unbound moisture and part of loosely bound moisture was removed.

An aliquot of 30 ml of the ST samples and 28 g of VIP and UD samples were centrifuged at 3000, 4000 and 5000 rpm for time intervals 20 mins to 100 min, in a table-top centrifuge (Hermle Labor Technik GmbH, Wehingen, Germany), and the supernatant was removed and MC was determined.

The MC of the sludge after centrifugation at different rates correspond to linear correlations, clearly inferring the dependence of moisture retention as a function of external force. Further, the moisture retention reaches an equilibrium at which point, further moisture removal by mechanical forces is not possible, or is hard. This state of equilibrium can be inferred as the interface of unbound-bound moisture, and a transition zone wherein some amount of loosely bound moisture, especially the interstitial moisture, is also removed. The results concur with the findings of the unbound-bound moisture interface carried out on sewage sludges (Jin et al., 2004).

- The **Differential Scanning Calorimeter (DSC)** records the calorimetric changes of the sample and is capable of measuring the changes exothermically when the sample freezes, and endothermically when the sample melts. This helps in quantification of the amount of unbound moisture with the underlying assumption that the unbound moisture behaves as pure moisture and freezes and melts at temperatures close to 0°C (Smidt & Tintner, 2007).

An aliquot of 8-10 mg of samples were put in the aluminium sample holder, having a central 0.7 mm pin hole. The thermograms were recorded between ambient temperature (25°C) to -60°C and -60°C to ambient at a heating/cooling rates of 10°C/min, in pure N_2 atmosphere (Lee & Lee, 1995; Zhang et al., 2019). The experiments were conducted in *DSC 600* (Perkin-Elmer, USA).

The DSC tests were conducted on the FS samples to observe the exothermic and endothermic peaks when the temperature of the sample was frozen up to -60°C. The peak was observed between -10°C to-20°C indicating the presence of bound moisture fraction. No additional peaks were observed after -20°C, and this could be due to the relatively low amount of bound moisture which goes below the minimum detection limits of the instrument. The endothermic and exothermic enthalpies of the sample is as given in Table 4.4. The enthalpy peak Delta H in the graph representing the need for more time and energy for evaporation.

The values of the peak integrals were consistent with slight changes in the endothermic peaks. The bound MC is calculated as a ratio of the enthalpy value of sample to the enthalpy of pure water (Wang et al., 2012) and is given by Equation 4.1.

$$\boldsymbol{W_b}(\%) = \mathbf{1} - \left(\boldsymbol{Q_{endo}}/\boldsymbol{Q_{pure}}\right) \mathbf{100} \qquad \textbf{Equation 4.1}$$

Where:
W_b - amount of bound water;
Q_{endo} - enthalpy value of samples, from the curves
Q_{pure} - enthalpy value of pure water, 334.6J/g
Since the amount of unbound moisture is directly proportional to the peak area in the enthalpy diagram, the proportionality constant of 0.0028 can be used from the calibration data, and the mass of the bound moisture is calculated as given below (Erdincler & Vesilind, 2003).

$$\boldsymbol{W_b}(\%) = \mathbf{1} - (\mathbf{0.0028} \text{ x } \boldsymbol{Q_{endo}}) \qquad \textbf{Equation 4.2}$$

Where:
W_b - amount of bound water;
Q_{endo} - enthalpy value of samples, from the curves

– The **Thermo-Gravimetric Analyzer (TGA)** is capable of controlled and ramping temperatures, and hold isothermally for fixed periods of time. The TGA instrument measures the weight of the sample as a function of time and temperature (Lin et al., 2020).

The TGA analysis was carried out using pure N_2 as the carrier gas at a flow rate of 10mL/min, with 35-40 mg of samples and cell temperature from ambient (25°C) to 90°C at the rate of 10°C/min, and maintained in isothermal condition for 45 mins using *Universal Analysis 2000* (TA Instruments, USA) (Deng et al., 2011; Zhang et al., 2019).

Imagery of sludge samples at different MC is taken using **Scanning Electron Microscopy (SEM)** to study the microstructural properties of the sludge and speculate the moisture boundness accordingly.

The SEM images were obtained on a scanning electron microscope *FEGSEM* (Carl Zeiss, Germany) operated at 5 kV accelerating voltage. As the raw samples were with moisture, they had to be pre-treated in 2% glutaraldehyde at 4°C for 6 hours, and washed with 0.1 mol/L phosphate-buffered saline at the pH 7.2. The samples were then dehydrated with ethanol and dewatered with 100% acetone and isoamyl acetate (Aksoy et al., 2019; Wang et al., 2012).

4.3.3 Results and Discussion

DSC is a simple thermodynamic interpretation to show the liquid-solid binding strength and its role in depression of the freezing point of bound moisture. Unbound moisture was easily differentiated from the un-freezable bound moisture that did not freeze at normal freezing temperature of pure moisture by DSC.

Table 4.4. Bound moisture calculations by the two methods

	VIP	UDDT	ST-BW	ST-wGW
Enthalpy J/g	158.05	162.34	123.55	132.6
Unbound-Bound MC interface % – Equation 4.2	52.77	51.48	63.08	60.37
Unbound-Bound MC interface % – Equation 4.3	55.75	54.55	65.41	62.87

The correlation between the two approaches is almost same with r=1 (Figure 4.3).

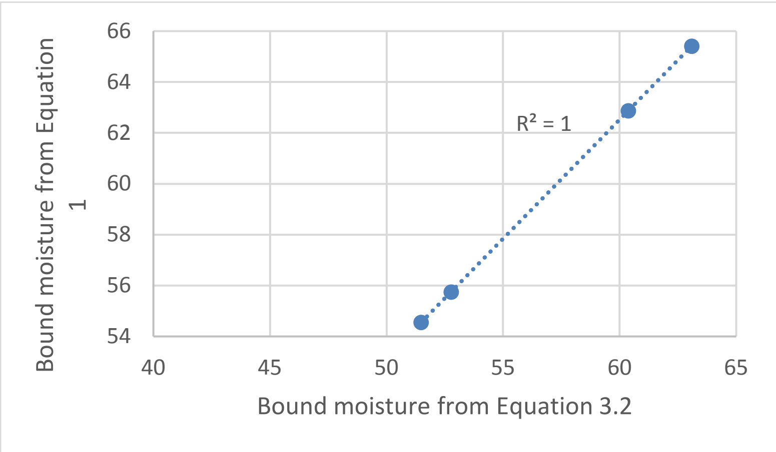


Figure 4.3. Correlation of results of bound moisture from the two approaches

TGA is used to characterize the variation in the same mass with temperature (Figure 4.4) and DTG curve is the change in mass with time (Figure 4.5). Comparing these curves, the dip in the temperature of TGA with the fall in the mass rate, it can be deduced that point of moisture could possibly be when all the unbound moisture is removed, and the falling mass rate (also the drying rate) denotes that the remaining moisture in the samples is bound.

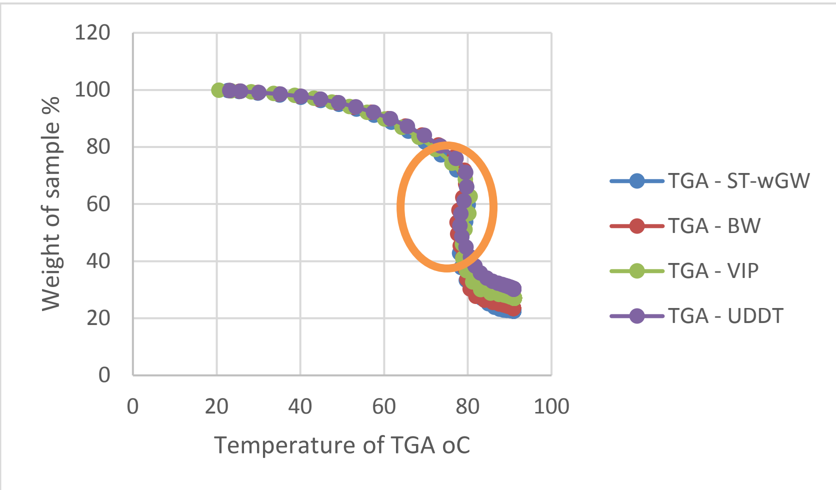


Figure 4.4. TGA curve of the samples

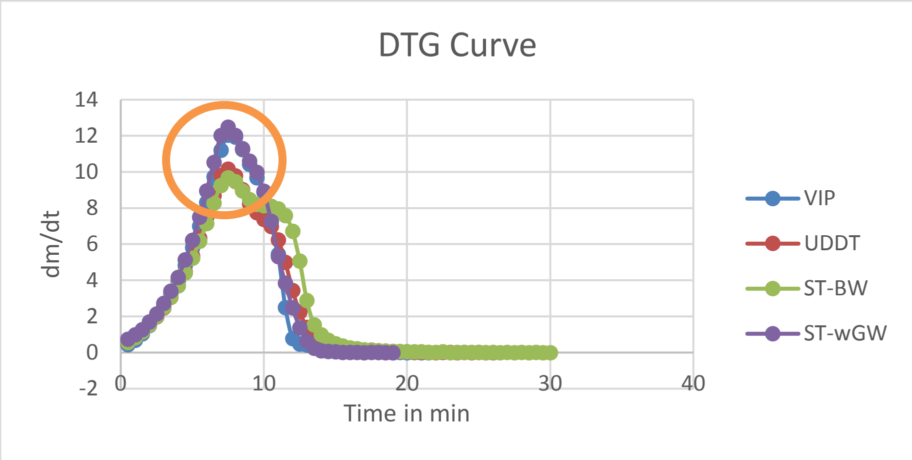


Figure 4.5. DTG curve of the samples

Accordingly, the bound moisture fraction from the TGA results is as given in the Table 4.5.

Table 4.5. Bound moisture fraction from TGA and DTG curves

	VIP	UDDT	ST-BW	ST-wGW
Unbound-Bound MC interface %	51.07	56.57	62.29	59.72

The moisture removal by drying does result in shrinkage but also creates pores. Comparing the imageries of the raw and dried samples from the scanning electron microscopy, the glass transition theory seems applicable, which implies that more the number of pores, or less collapse, more stable is the micro-structural properties of the sludge. We also have noticed that the dried samples are highly porous in nature. Comparative imagery must be carried out between low and high temperature drying to assess the open structure and stability of the sludge. This further has an influence of the possible applications of the sludge.

The images of the pre-treated sludge samples (Figure 4.6) – raw and completely dried – showed greater porosity and open structure of the sludge. The raw samples show an irregular and loose structure, with a lot of floc, and the dried samples showing more pores. Shrinkage was also observed in the fully dried sample. The imagery of the samples with intermediate MC is in progress.

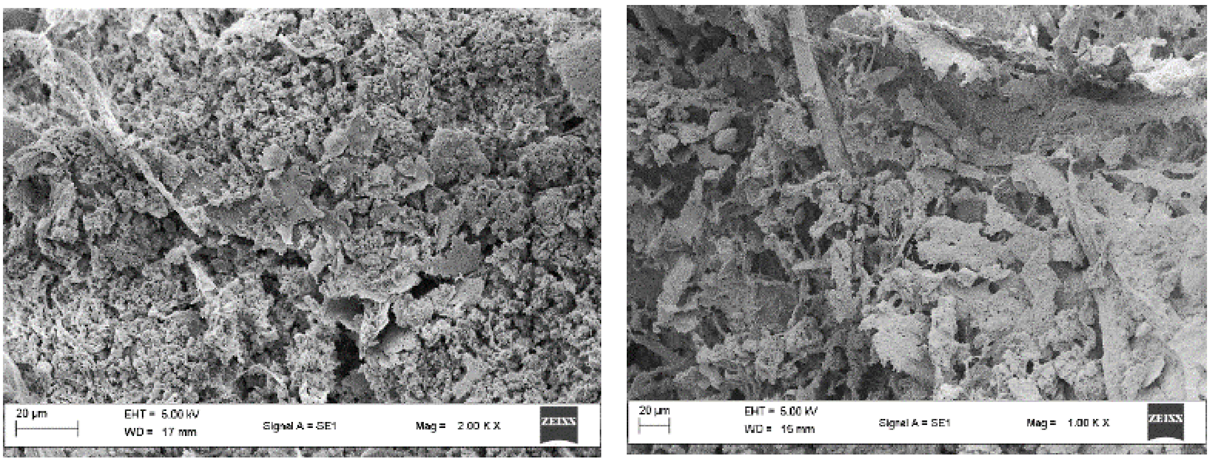


Figure 4.6. The raw and dried VIP sample at 20 µm

4.3.4 Conclusions

The CMC, or the end of constant drying rate period, denotes the removal of unbound moisture, and the remaining moisture in the sludge is mostly only bound moisture. This unbound-bound moisture interface is not one single point, but a range. This is because of the presence of loosely bound interstitial moisture which acts as a transition zone.

The bound moisture fraction from the DSC measures the enthalpy of the unbound moisture which readily freezes at the normal freezing point like that of pure water. The thermo-dynamic interpretation of the solid-liquid binding strength plays an important role in the depression of the freezing point of bound moisture. Hence, this is a more reliable measurement of the bound moisture fraction. However, since the quantity of sample used for the experiment is small, the presence of any impurities since as small stones, sand, etc. can influence the outcome, and thereby, the replicability of the results is not always accurate. Further, DSC relies on the presence and property of unbound moisture to freeze before that of the bound moisture.

Similar to the DSC, the TGA measure the samples weight (TGA curve), the weight loss rate (DTG curve) and the cell temperature. The weight loss rate starts to reduce after the unbound moisture is removed, and the moisture remaining after the fall is bound MC. This corresponds to the loss in cell temperature, indicating the need of additional energy to remove the bound moisture. This further confirms the interactions between the water molecules and the solid particles. The DTG profiles for the different samples were notably different which also denotes the change in the moisture distribution in different samples. Further, both the DSC and the TGA results are close to each other, and compares well with the water activity measurements.

The SEM imagery provides insights on the microstructure of the sludges, and evidently, the presence of pores denotes stable microstructure and the shrinkage of the samples during drying is not high to destabilize the pores and thus, effect the sludge stability.

Figure 4.7 and Table 4.6 show the unbound/bound moisture limit from different types of experiment. It can be seen that the results from different analysers and faecal sludge are in the same range.

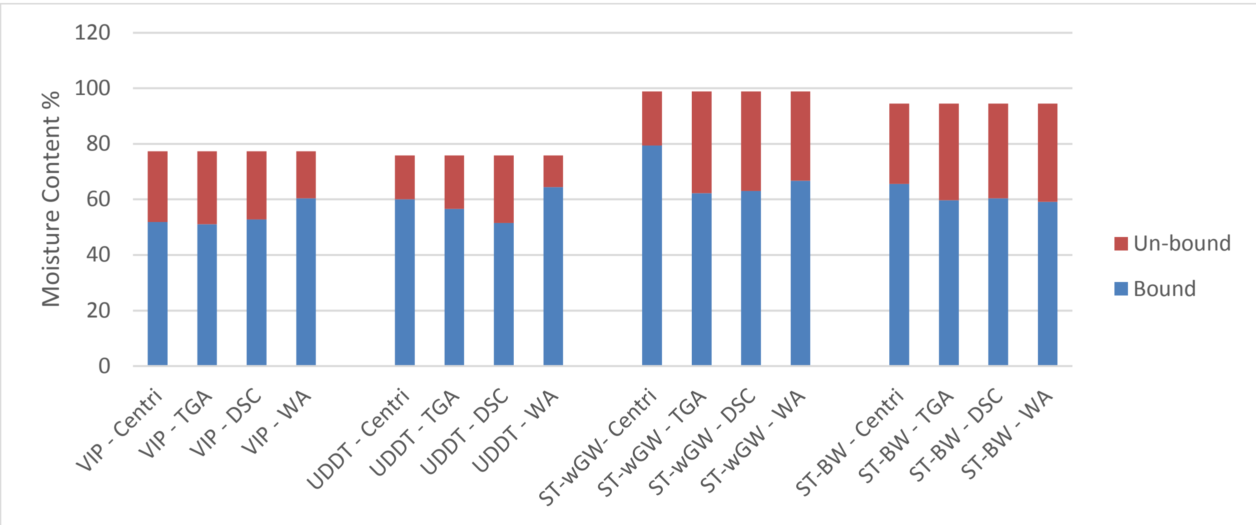


Figure 4.7. Unbound-bound moisture interface of FS samples from different experiments

Table 4.6. Range of unbound-bound moisture interface for FS sludges

	VIP	UDDT	ST-wGW	ST-BW
Unbound-bound moisture interface range %	51.07-60.35	51.48-64.38	62.29-66.71	59.11-60.37

4.3.5 Objective 2 – Determination of bound moisture fractions

4.3.5.1 Introduction

The bound moisture or immobilised moisture is physically or chemically (or both) bound to the solid particles in the sludge. Based on the type of interaction of the moisture molecules with the solid particles, one of the approaches of classification is adopted in this research and is given below (Erdincler & Vesilind, 2003; Vaxelaire et al., 2000).

a. Interstitial moisture or capillary moisture
b. Vicinal moisture or surface moisture
c. Intra-cellular moisture or water of hydration

The removal of bound moisture involves overcoming the binding strength of the moisture with the solid particles, and different types of bound moisture will require different approaches. Thus, knowing the proportions of the types of bound moisture will help in their removal with appropriate technologies, as relevant to that fraction.

The bound moisture fractions are experimentally determined by different methods and the results are compared for consistency. **Drying test** is one of the classical methods adopted to determine each fraction of the moisture. The different drying rates correspond to different types of moisture –

- Constant drying rate period – Unbound moisture
- First falling rate period – Interstitial moisture
- Second falling rate period – Vicinal moisture
- Residual moisture – Water of hydration
-

The **water activity**, defined as the ratio between the partial vapour pressure of moisture in a substance and the standard state partial vapour pressure of moisture, is measured at different MC to get the moisture vapour sorption curves for the samples. Similarly, the dynamic vapour sorption provides both adsorption and desorption isotherms at different humidities.

4.3.5.2 Experimental methodology

– The process of drying involves heat and mass transfer between a hygroscopic material and the drying air. The drying test is a quantitative measurement method to determine the moisture distribution in the sludge and one of the most common experiments carried out. The transport of moisture within the sludge occurs due to various mechanisms, surface diffusion and hydrostatic pressure differences being of interest in the convective drying test (Barbosa de Lima et al., 2015). The objective of this experiment is to determine the moisture evolution during convective drying of the FS samples.

The MC of the samples is calculated according to the equation:

$$\boldsymbol{MC}\% = \mathbf{100}\% \times (\boldsymbol{W_1} - \boldsymbol{W_2})/\boldsymbol{W_1}$$ **Equation 4.3**

Where:
WC is the water content of sludge, W_1 is the mass of wet sludge, and W_2 is the mass of sludge when it is dried to constant weight at 105 ◦C (Vaxelaire & Cezac, 2004).

The plot of drying rate versus moisture is the drying curve, also known as Krischer's curve. A typical drying curve is divided into three sections. The first phase after the initial warm up phase of the sample is the constant drying rate period, where the equivalence between heat and mass transfer occurs. This occurs since there is always a film of unbound moisture available at the evaporating surface. The constant drying rate period reflects the extent of unbound moisture in the sample. The end point of the constant drying rate period is the point of critical moisture and this corresponds to the limit of unbound moisture, or the unbound-bound moisture interface.

Post the critical MC, the drying surface gets unsaturated since the migration of moisture to the surface is affected due to internal transport limitations. This is the falling drying rate period, further classified into first falling rate and second falling rate periods, corresponding to interstitial moisture and vicinal moisture respectively (Mujumdar & Devahastin, 2011).

The test rig for convective micro-drying (Figure 4.8), at the Department of Chemical Engineering, University of Liege, is designed to handle small samples between 0.5 g to 5 g. The samples are extruded in a circular die of 10 mm diameter and cut at a height of 10-12 mm. The sample is placed in the drying chamber, suspended by a supporting grid, connecting to a precision weighing scale (Sartorius, Germany). The weighing scale is connected to the computer and the weight is recorded every 5 secs.

Compressed air is fed into the system, which is heated to the pre-set temperature before entering the drying chamber. Low temperature drying at 40°C was carried out for all the samples. The drying air velocity is controlled with a pneumatic valve.

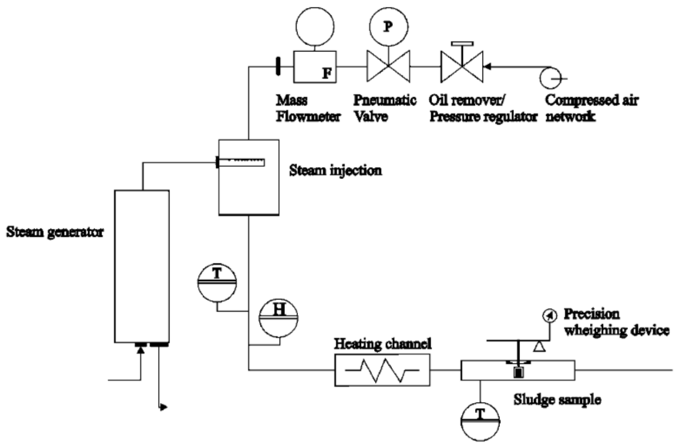


Figure 4.8. Micro-dryer set-up (Leonard et al., 2002)

Upon plotting the Krischer's curve for the samples, the constant drying rate period is not quite visible in the drying test for all the samples, as experienced by other researchers (Vaxelaire & Cezac, 2004). However, by plotting the inverse of drying rate on a logarithmic scale, and the corresponding MC in kg/kgTS, the different slopes of the curve can be corresponded to the different types of moisture.

–The **moisture sorption isotherms (MSI)** are used to describe the sorption characteristics and illustrate the release of water molecules in biological materials. At a particular atmospheric condition, the MC of the biological material changes and stabilizes at the equilibrium moisture content (EMC) and this is denoted by MSI. The movement of the MC from wet material to dry is given by desorption isotherm, and from dry material to wet is given by adsorption isotherm (Simo-Tagne et al., 2019). This method is successfully used in the food industry (Bourgault et al., 2019). Its application in understanding the hygroscopic behaviour and moisture distribution characteristics of FS from different OSS is investigated in this study.

The sorption isotherms provide the behaviour of moisture held in the wet material (Figure 4.9). The three zones which gets formed when both adsorption and desorption isotherms are plotted for a material (Freire et al., 2007).

- Region A – Water is tightly bound to the wet material
- Region B – Water is loosely bound, and confined to smaller capillaries
- Region C – Water is very loosely bound, and held in larger capillaries

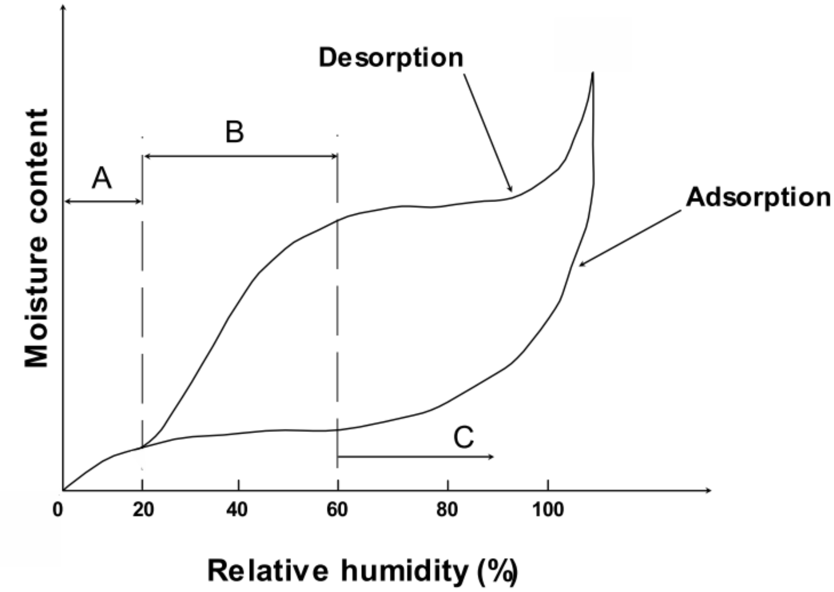


Figure 4.9. Sorption isotherms and regions

Furthermore, for the dynamic sorption method, the equilibrium criteria is not as severe as for the saturated salt solution method, and the sorption isotherm can be obtained relatively quickly, in a few days, compared to a few weeks for the static method (Arlabosse et al., 2003).

The sorption isotherms were determined using a dynamic vapour sorption instrument (*DVS Intrinsic*, Surface Measurement Systems, United Kingdom) varying the relative humidity (RH). The FS samples from different OSS were weighed to about 4 mg, and two sorption cycles was applied in this work – From 0% RH to 90% RH and back to 0% RH, and from 90% RH to 0% RH and back to 90% RH. The incremental step was 10% RH, and the isotherms were conducted at 40°C. The samples were maintained at a constant RH until the mass variation per minute (dm/dt) was < 0.0015% for 10 minutes. The raw DVS was analysed initially using the *DVS Standard Analysis System*, which allowed the construction of hysteresis curves (Simo-Tagne et al., 2019).

The dynamic sorption method was employed to produce the sorption isotherms and to investigate the critical moisture equilibrium of the FS (Freire et al., 2007). The results from the DVS will be compared with the results from the static sorption experiments using saturated salt solutions by Ms. Larona Malope, MSc candidate in the project.

Water activity of samples with different MC was measured with a AquaLab Tuneable Diode Laser (TDL) (Meter Group, Pullman, WA, USA) water activity meter at 22°C. Water activity is a function of MC, and is a good indicator on moisture boundness in the sludge, with high water activity indicating unbound moisture, and lower measurements corresponding to different proportions of bound moisture.

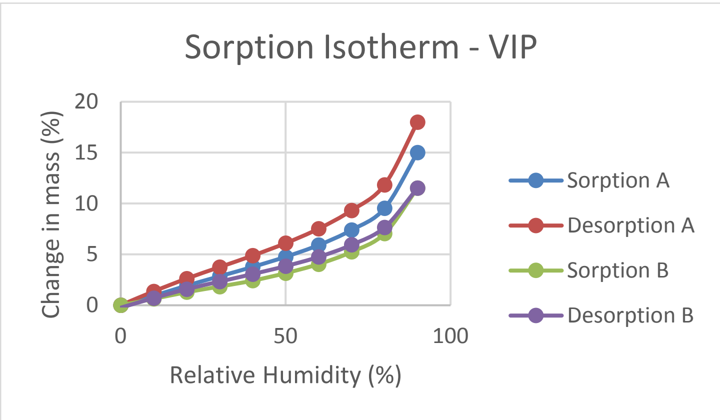
– Time-domain NMR (nuclear magnetic resonance) and low frequency NMR are reliable methods recognised for the measurement of MC. NMR can accurately and efficiently measure moisture distribution, but it still is fairly underutilized method in this field. Parameters such as number of scanning times, sample weight influence the accuracy of the results (Liukkonen & Selin, 2016; H. Mao et al., 2016).

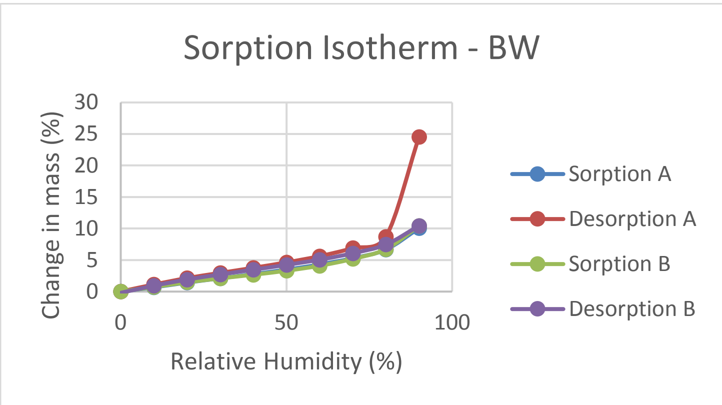
A Bruker Avance III 500 MHz NMR spectrometer (Bruker Biospin, Germany) operating at a Larmor frequency of 500.13 MHz (11.75 T) was used in conjunction with a Bruker broadband observe (BBO) high resolution solution probe. Standard 5mm diameter NMR tubes (Norell S 5-400-7) were used. A sample volume of 200 μl guaranteed that the entire sample was inside the active space of the radiofrequency coils from the BBO probe.

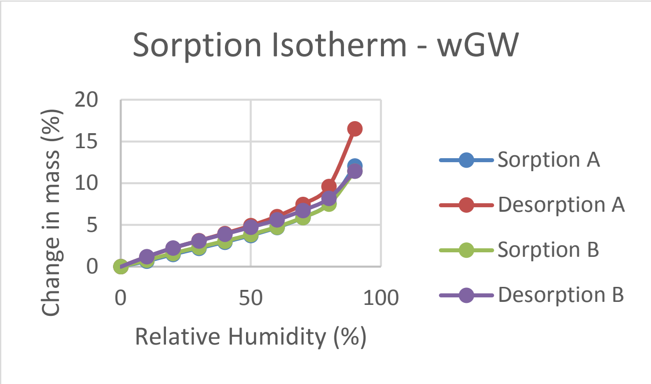
The parameter settings used is as follows:
The 1H T2 relaxation curve of the samples was measured with TD-NMR using a Bruker minispec mq20 instrument (Bruker BioSpin, Billerica, MA, USA) at a 1H frequency of 20 MHz at 25 ◦C without dilution. One-shot T2 data were acquired using a standard Carr-Purcell-Meiboom-Gill sequence developed in the 1950s. The acquisition parameters were as follows: number of scans = 8, time between each pulse (τ spacing) = 3.5 ms, total echoes = 3000, recycle delay = 11 s, and dummy shot = 1 (F. Mao et al., 2020; Okada et al., 2021).

4.3.5.3 Results and Discussion

The EMC for each sample was determined from the adsorption-desorption isotherms measured in the DVS from the last fifteen data at each plateau, corresponding to the respective RH (Figure 4.10). The desorption and adsorption isotherms for the two sorption cycles – 90%-0%-90% and 0%-90%-0% – is analysed for the EMC. Results A are for the cycle 90%-0%-90% and results B are for 0%-90%-0%.







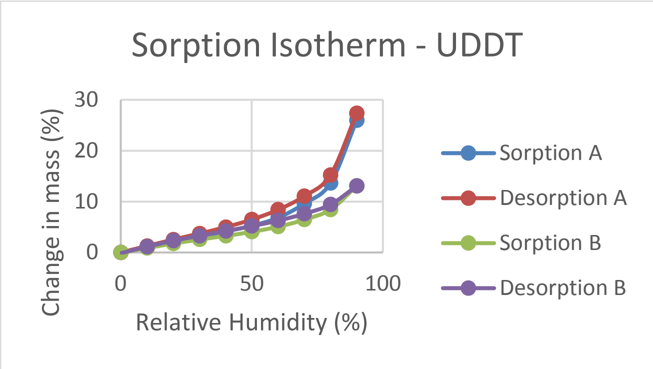


Figure 4.10. Adsorption and desorption isotherms for different samples

The water activity measurements were close to 1 up to almost 55-60% of MC of the samples, corresponding to the presence of unbound moisture. A gradual fall in the water activity was observed, corresponding to the first falling rate period, and then a significant drop in its values below 30% MC, corresponding to the second falling rate period. There is no difference in the water activity measurements between samples from different OSS.

At a given RH, it is possible to measure the water activity of the sample in its thermodynamic equilibrium with its atmosphere. Thus, the different types of bound moisture can be distinguished accordingly to the water activity measurements (Figure 4.11). The DVS and water activity meter provide a simple method to estimate to decent accuracy the sorption isotherms for FS samples.

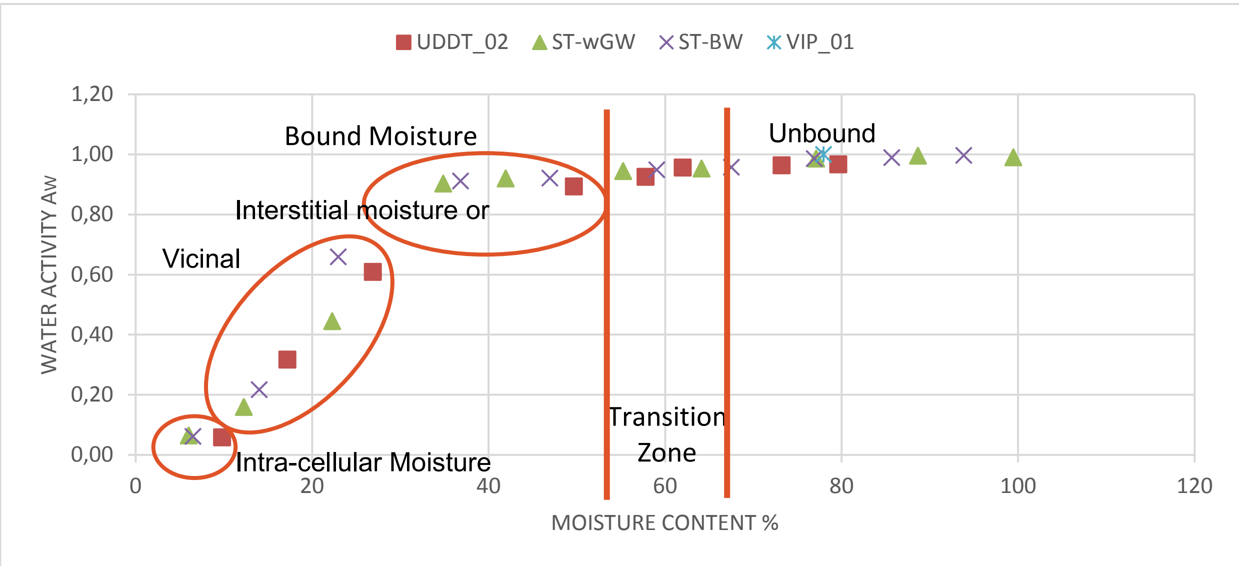


Figure 4.11. Water activity measurement at different MC of FS samples

4.3.5.4 Conclusions

Continuous classification of the bound moisture fractions in the different faecal sludges – VIP, UDDT, ST-wGW and ST-BW – were conducted based on the low T convective drying test, dynamic sorption isotherms and water activity measurements (Figure 4.12). The experiments with NMR are on-going. The contents of interstitial, vicinal and intra-cellular moisture can be calculated and quantified by these tests. The quantity of unbound moisture is different for all samples, since the initial MC varied with ST-wGW at 98.9% MC and UDDT at 61.67%. Lower unbound moisture, as in VIP and UDDT samples tend to provide better bound moisture values that get closer together.
More than 35% of the bound moisture is interstitial moisture, with ST samples having greater interstitial moisture. The interference of other materials like soaps, oil, etc., especially in the case of ST-wGW results in larger fraction of interstitial moisture.

This representation of moisture distribution within the different FS is an interesting approach of characterizing the ability of these sludges to dewater and dry. There are various pre-treatment and treatment methods to effectively achieve solid-liquid separation, with implicit hypotheses assumed with regard to the moisture boundness and their different binding strengths. However, further research with more samples is necessary to accurately and consistently evaluate the distribution of moisture, and this becomes more pertinent for FS due to its high variability.

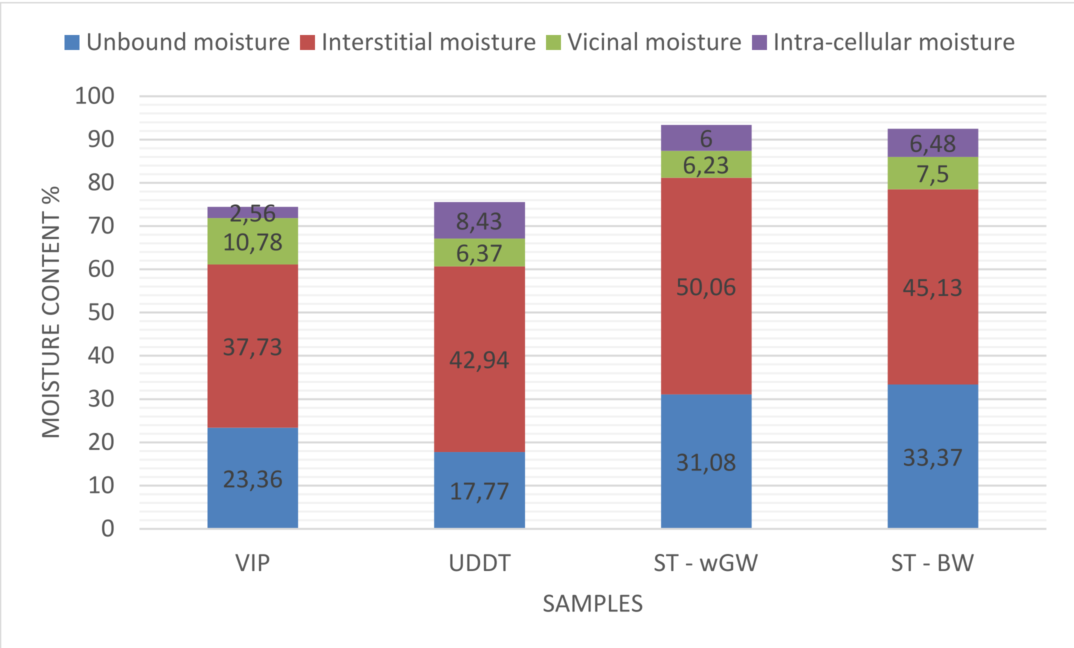


Figure 4.12. Moisture distribution in FS sludges

4.3.6 Objective 3 – Moisture boundness INFLUENCING Properties of Faecal Sludge

4.3.6.1 Introduction

The solid-liquid separability of FS is impacted by sludge characteristics which influence bound water (Gold et al., 2018b). The presence of nutrients as well as high calorific value of these sludges add the next layer of circularity, and the need to investigate the solid-liquid separation abilities and the properties that influence or/and are influenced by solid-liquid separability of FS (Gold et al., 2016).

The cohesive-adhesive properties of sludge define its stickiness which is influential in choosing some of the drying technologies for solid-liquid separation. The application of ball penetrometer is carried out to investigate the cohesion and adhesiveness of the FS samples. Dynamic rheological studies on high solids FS samples have not been carried out, taking into consideration the edge effects – fractures and cracks. These experiments lead to determination of dilatancy, which is an instrumental property of sludge influenced by its moisture boundness. Extracellular polymeric substances (EPS) plan an important role in the solid-liquid separability, with EPS and bio-flocculation correlated, but its exact effect is still unknown (Yang & Li, 2009). Particle size distribution (PSD) is another important structural property of sludges which influences its flocculation, settling as well as compactibility.

4.3.6.2 Experimental methodology

- In soil mechanics, **full-flow ball penetrometers** (Figure 4.13) are used for measurements of shear strength of clay soils in both offshore field investigations as well as laboratory tests, with the shear strength of the sample derived from the resistance to penetration. The stages of penetration are defined by three stages – shallow failure mechanism, transitional failure mechanism and full-flow failure mechanism. During the penetration of the ball in the sample, the surrounding sample undergoes shear strains and softens, and a certain extent of the softening is measured in the resistance (Léonard et al., 2014).

The Herschel-Bulkley fluid model is applied, such that shear strength (τ) is a function of yield stress (τ_o) and shear strain rate (γ) according to the power law:

$$\tau = \tau_o + K.\gamma^n$$ **Equation 4.4**

The specific resistance to penetration (q) is calculated by dividing the shaft force during penetration (F) by the projected area of the ball (A). The shear strength (τ) of the sludge is then calculated by dividing the specific resistance to penetration by a correction factor N_b.

$$\tau = \frac{q}{N_b} = \frac{F}{A.N_b}$$ **Equation 4.5**

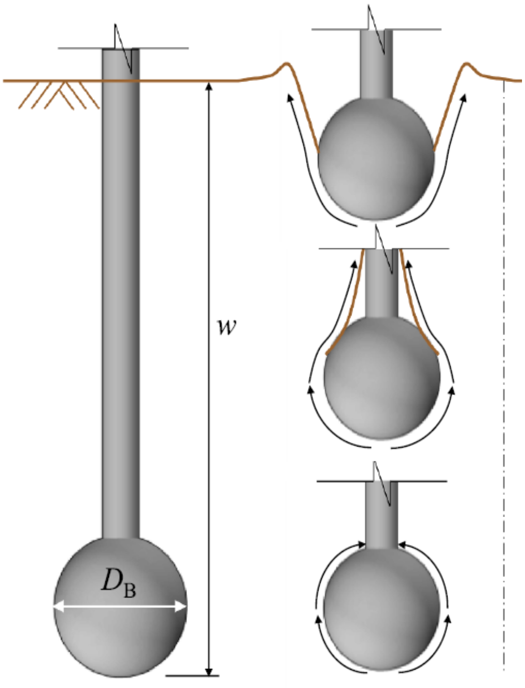


Figure 4.13. Mechanism of ball penetration (Chen et al., 2021)

LS1-302 (Ametek Lloyd Instruments, England) with a 30 mm diameter spherical geometry probe (ball penetrometer) was used for the analysis of the VIP and UDDT samples. The penetration depth of the probe was 15 mm, at a constant speed of 1 mm/s. The experiment included the load phase (compression) and a discharge phase (relaxation) as shown in Figure 4.14.

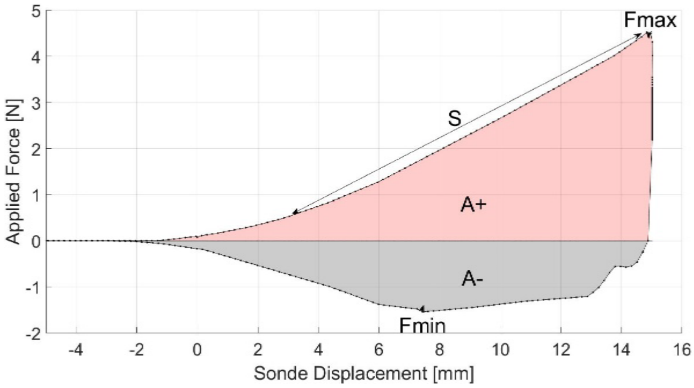


Figure 4.14. Penetration curve (schematic)

– **Dynamic rheological measurements** including identification of viscoelastic properties (elastic and viscous behaviour) was done in oscillatory mode. This involves application of a sinusoidal stress to the sample and measuring the sinusoidal strain (and vis-a-versa). The stress and strain sine waves are given by:

$\boldsymbol{\tau(t) = \tau_0 \sin \omega t)}$ **Equation 4.6**

$\boldsymbol{\gamma(t) = \gamma_0 \sin(\omega t + \delta)}$ **Equation 4.7**

Where

τ_0 and γ_0 are the maximum amplitudes of stress and strain respectively, ω is the pulsation defined by 2πf, f is the frequency and δ is the phase difference between the deformation and the constraint.

Two fundamental quantities of dynamic rheology are used to characterize the viscoelasticity of materials:

- The elastic modulus or storage modulus, G', which characterizes the energy stored in elastic form and retrievable by the sample (elastic component);
- The viscous modulus or loss modulus, G'', which characterizes the energy lost by friction (viscous component)

$\boldsymbol{G' = G^* \cos \delta}$ **Equation 4.8**

$\boldsymbol{G'' = G^* \sin \delta}$ **Equation 4.9**

Where:

The complex viscoelastic modulus G∗ is defined as the ratio of the amplitudes stress and strain.

Dynamic rheological measurements were carried out using *Anton Paar Physica MCR 302* (Anton Paar Benelux BV, Belgium). The instrument is a controlled stress rheometer equipment with a 50 mm parallel plate geometry. The gap between the fixed plate and the oscillating plate was fixed at 2 mm. The samples were homogenized before adding onto the plate on the rheometer, at a temperature of 20°C.

The samples were submitted to a frequency sweep, from 0.1 to 100 rad.s^{-1}, at a constant shear strain in the linear viscoelastic region, and the strain sweep experiments were conducted from 0.01% to 10%. Each measurement was performed in triplicate. The G’ which is the storage modulus and G’’, the loss modulus, are presented in the results, which are the function of the frequency (Agoda-Tandjawa et al., 2013).

Further, the properties of **dilatancy** of the sludges was measured. The characterization of the rheological properties of sludges with high TS has not been carried out as extensively as that of low TS sludges because of edge effects in the form of cracks and fractures and dilatancy, described as "*disordered dense packing of hard grains cannot undergo a shear without simultaneously expanding in the direction perpendicular to the shear plane*". Hence, no reliable steady-state data is possible for sludges with high TS (Mouzaoui et al., 2018). To avoid this, Mouzaoui et al. (2018) developed a specific procedure to take into account the edge effects and also measure the normal force simultaneously with the tangential shear stress using a rotational rheometer.
Samples with different TS of VIP and UDDT sludge ranging from 10% to 43% were prepared with dilution (low TS) and centrifugation (high TS). The rheological investigations were carried out with a stress-controlled *RS600 instrument HAAKE Rheostress 600* (Thermo Scientific, Germany), and the data analysis was assisted by the RheoWin software. Serrated parallel plates of 35 mm diameter was used with a gap of 2 mm.
For different TS concentrations, a stress sweep was applied to the sample which consisted of successive steps of constant dynamic stress of increasing intensity (Figure 4.15). A pause was marked between each step as a reference to allow for the correction of edge effects. Tests were conducted in triplicates to evaluate reproducibility, and normal force was recorded along with the tangential shear stress (Mouzaoui et al., 2018).

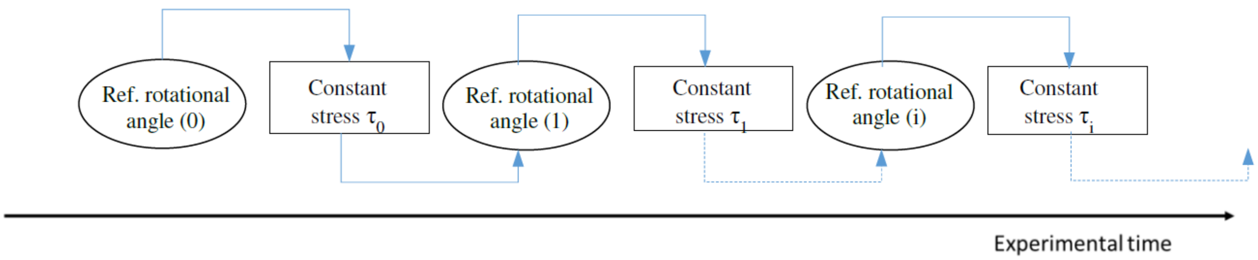


Figure 4.15. Experimental procedure to measure the real sheared surface

– **Extracellular polymeric substances (EPS) i**s one of the most influential substance in terms of the moisture retention characteristics of the sludge because of its cross-linking polymeric network like structure (Comte et al., 2007). It is composed of many organic substances mainly proteins, polysaccharides, humic acids, and little fractions of lipid, nucleic acid, and amino acid, etc. (Xie et al., 2014). EPS which exists outside the cells is further divided into Loosely Bound EPS (LB-EPS) and tightly bound EPS(TB-EPS) and can be generally separated by centrifugation.

There is no standard method for the extraction of EPS and its fractions – LB-EPS and TB-EPS, and can be extracted by different physical and chemical methods. In this research, the EPS and its fractions are extracted from the FS samples by adopting a two-step heat extraction method (Christine et al., 2019; Li & Yang, 2007; Yang & Li, 2009). The method involves centrifuging the sample in a 50 ml centrifugal tube at 4000 rpm for 5 min, discarding the supernatant and resuspending the sludge pellet in 0.05% NaCl (w/v) warm solution at 50°C and sheared using a vortex mixer for 1 min. The suspension is centrifuged at 4000 rpm for 10 min, and the organic matter in the supernatant was regarded as LB-EPS. For TB-EPS, the sludge pellet was resuspended again in 0.05% NaCl solution and this suspension is heated in a water bath for 30 min at 60°C. Post heating, the suspension was centrifuged at 4000 rpm for 15 min, and the supernatant liquid was regarded as TB-EPS. The cell lysis is said to be of no significance after such extraction process.

Since proteins and carbohydrates form the major components of EPS, measurement of proteins and carbohydrates of the extracted fractions is carried out. For the measurement of proteins, measuring amino acids gives accurate approximation of the proteins present. However, due to cost and time considerations, the use of total nitrogen for determining crude protein is widely used (Suzanne N.S., 2010). In this research, the nitrogen is determined by the nitrogen combustion method, and the protein content is calculated as a ratio of protein to nitrogen. Nitrogen is quantified in the CNS analyzer.
Crude Protein Percent (CP%) (FAO, 2011) is given by:

$\%CP = \%N \times F$ **Equation 4.10**

Where:
F is 6.25 (from 6.25 g proteins per g·N)
The total carbohydrates, including mono-, di-, oligo-, and polysaccharides, was analyzed by the phenol-sulphuric acid method (Suzanne N.S., 2010).

– The **PSD** of any sludge is one of the essential characteristics. Since many factors determine the composition of faecal sludge in a containment system, many small particles and fibrous substances are present. PSD of the samples was measured by the Malvern Mastersizer 3000 (Malvern Instruments Ltd, Worcestershire, United Kingdom) with Hydro EV Flexible volume wet dispersion. The particle size distributions (PSDs), i.e. particle size at 10% (Dv10), 50% ((Dv50), median diameter), and 90% (Dv90) of the volume distribution were calculated using the Mastersizer 3000 software (version 5.54). The measurement was carried out in triplicates.

4.3.6.3 Results and Discussion

The penetration curve (Figure 4.16) demonstrates the maximum positive peak (Fmax in N) which corresponds to the force necessary for the probe to penetrate the sample to the maximum depth, Fmax is generally assimilated to the sample firmness or hardness parameter. The positive area (A+ in mJ/Nmm) under the curve up to Fmax corresponds to the work carried by the probe during the compression test on the product. The minimum negative peak (Fmin in N) is the force required to separate the sample from the probe, Fmin is defined as the adhesive force of the sample, the more this force is negative, the more the product is adhesive. The negative area (A-, mJ/Nmm) under the curve is the work needed to detach the probe from the sample, A- is likened to an adhesiveness, stickiness or tackiness index (energy required to remove the sample from surfaces)(Hil et al., 2005; Mezger, 2020).

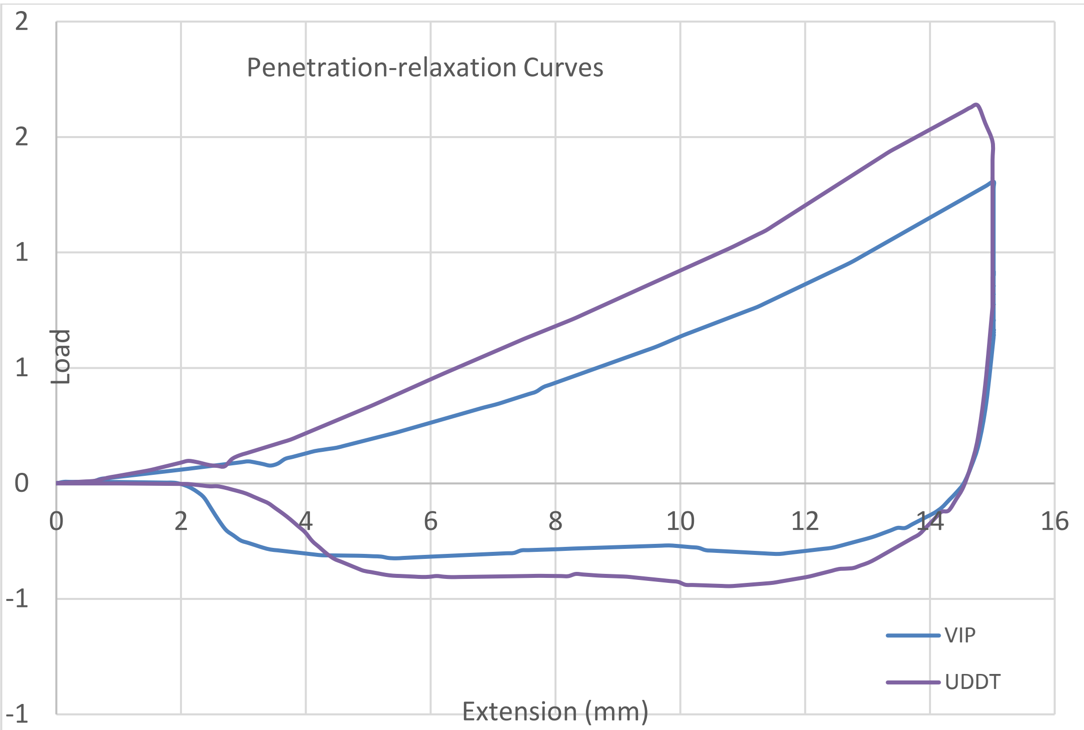


Figure 4.16. Penetration-relaxation curves of FS (mean curve for three replicates)

Sludge, at lower TS, can be described as a diluted suspension and classical rotational rheometer can be adapted. As the TS exceeds the plastic limit, the sludge can no longer be considered continuous and is more granular in nature. Thus, at higher TS, cracks and fractures are highlighted, and this is captured during the shearing tests. This indicates the frictional interactions of solid-solid particles, influenced by solid-liquid interactions. This is observed in pasty materials such as concrete, fresh cement, clay, etc. These frictional interactions cause dilatancy, wherein the yield stress is no longer associated with the viscous forces but with the frictional contacts of solid-solid particle interactions (Figure 4.17).

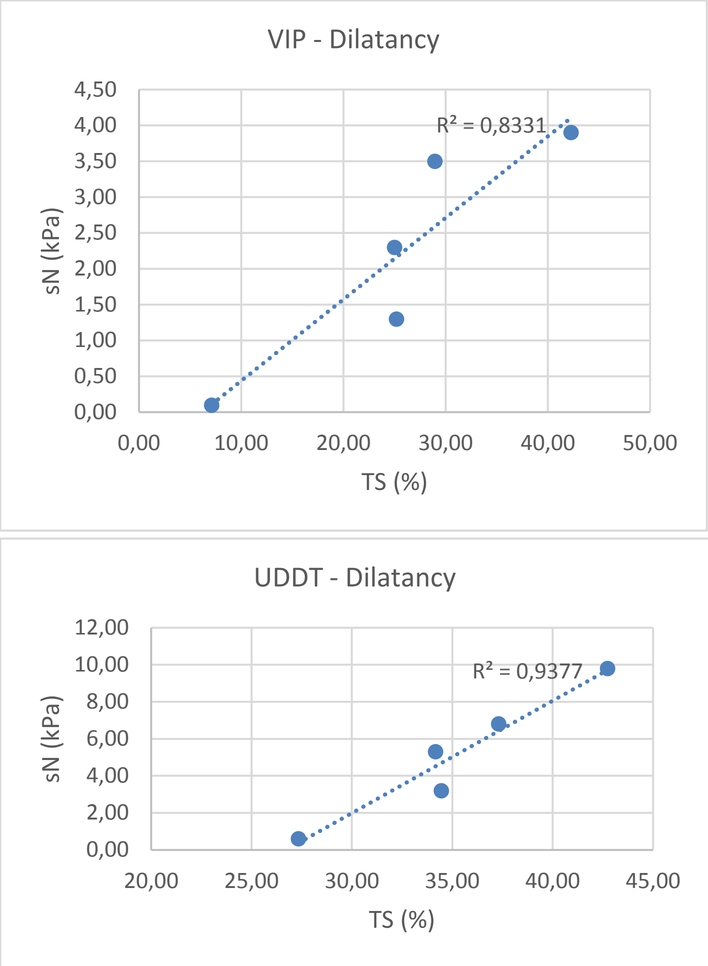


Figure 4.17. Dilatancy of VIP and UDDT sludges

TB-EPS has a significant impact on the stability of the sludge, and UDDT and ST-wGW sludges have the highest stability (Table 4.7), also indicated by their VS/TS value. The high TB-EPS in ST-wGW can also be attributed to the other substances such as soaps and oils present. Further, the Carbohydrate to Protein ratio is high in the ST samples, and lowest with the UDDT sludge. Further, the carbohydrate content is significantly higher than that of the proteins, whereas in sewage sludges, the proteins are higher than the carbohydrates. This can be attributed to the anaerobicity of the sludges (Peng et al., 2012).

Table 4.7. EPS – Crude protein and carbohydrate measurements of FS

Parameter	VIP	UDDT	ST – wGW	ST – BW
LB-EPS - Crude Protein g/g DM	0.995	0.259	6.803	3.904
TB-EPS - Crude Protein g/g DM	0.536	0.737	14.466	2.781
Total EPS Crude Protein g/g_DM	1.531	0.996	21.269	6.686
LB-EPS - Carbohydrates - g/g DM	84.029	93.651	326.679	305.884
TB-EPS - Carbohydrates - g/g DM	136.696	177.268	360.456	328.728
Total-EPS - Carbohydrates - g/g DM	220.725	347.428	687.135	634.612
C:P ratio	0.00694	0.00287	0.03095	0.01053

Further, literature says that the flocs are fragmented under anaerobic conditions and hence, anaerobic sludges have reduced particle size. Nearly 90% of the particle are less than 1100 µm for UDDT and VIP sludges (Figure 4.18), and for ST sludges, the is still lower (90% of the particles below 350 µm). This reduced particle size increases the specific surface area, and aid in solid-liquid separability (Cetin & Erdincler, 2004). It is also argued that anaerobically digested sludges have increased EPS, and hence have higher moisture retention (Houghton et al., 2001). More research is necessary, specifically with anaerobic sludges from OSS to correlate the anaerobic transformations with the binding strength and the roles of particle size and EPS.

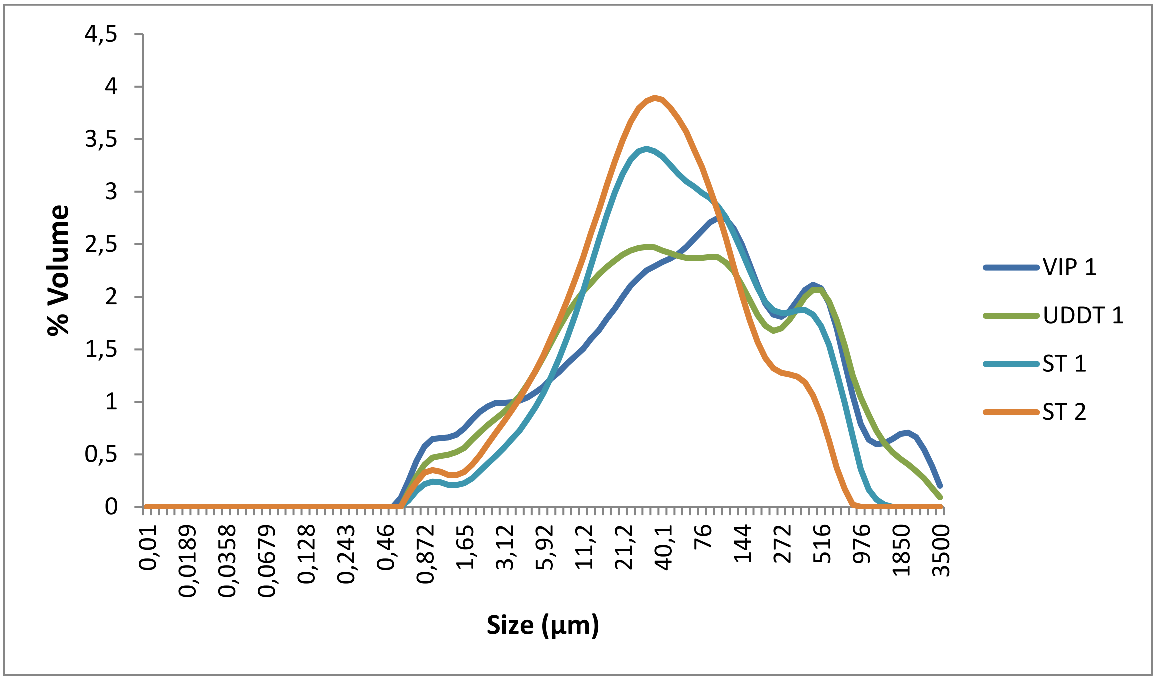


Figure 4.18. Particle size distribution of FS

4.3.6.4 Conclusions

The penetrometer penetration tests, carried out on VIP and UD samples, reflected the cohesive forces between the individual solid particles, with the solid particle-particle interactions and the adhesive forces between the solid particle-moisture interactions.

Rheological behaviour of FS remains similar and consistent with regard to the visco-elastic region across all types with shift of elastic modulus (G’) and viscous modulus (G’’) with increasing solids’ content. Recoverable deformation observed in the visco-elastic region, corresponding to unbound moisture; followed by permanent deformation caused by the plastic property.

Decreasing G’ and G” with increasing water activity observed, which indicates that besides the solid particle-particle interactions, the solid-moisture interactions contribute strongly in the rheology of FS. EPS holds moisture, and the high protein fraction was observed in ST samples, and particularly in ST-wGW, thereby correlating to high bound moisture from compactibility (centrifugation) tests. Inverse relationship between high TS% and total protein, attributable to the moisture holding capacity of proteinaceous part of EPS.

EPS and particle size influence moisture retention characteristics, and low particle size infers fragmented flocs, evidently due to the anaerobicity of all the sludges. This also increases EPS, which holds moisture. More research on the anaerobic sludges from OSS is necessary to understand the roles of particle size and EPS, specifically due to anaerobic transformations of the organic matter over time.

The next step will be to correlate these properties of faecal sludge to its moisture boundness distribution.

4.4 REFERENCES

Agoda-Tandjawa, G., Dieudé-Fauvel, E., Girault, R., & Baudez, J. C. (2013). Using water activity measurements to evaluate rheological consistency and structure strength of sludge. *Chemical Engineering Journal*, *228*, 799-805. https://doi.org/10.1016/j.cej.2013.05.012

Aksoy, A., Karasu, S., Akcicek, A., & Kayacan, S. (2019). Effects of different drying methods on drying kinetics, microstructure, color, and the rehydration ratio of minced meat. *Foods*, *8*(6). https://doi.org/10.3390/foods8060216

Alena, B., Marianna, K., & Yeukai, M. S. (2020). *Dewatering as a primary treatment of faecal sludge in individual residential sector (a technologies review)*. *January*. https://doi.org/10.1051/e3sconf/202016902008

Arlabosse, P., Rodier, E., Ferrasse, J. H., Chavez, S., & Lecomte, D. (2003). Comparison between static and dynamic methods for sorption isotherm measurements. *Drying Technology*, *21*(3), 479-497. https://doi.org/10.1081/DRT-120018458

Barbosa de Lima, A. G., Delgado, J. M. P. Q., Neto, S. R. F., & Franco, C. M. R. (2015). Intermittent drying: Fundamentals, modeling and applications. In *Drying and Energy Technologies* (pp. 19-41). Springer International Publishing. https://doi.org/10.1007/978-3-319-19767-8_2

Bourgault, C., Lessard, P., Remington, C., & Dorea, C. C. (2019). Experimental determination of moisture sorption isotherm of fecal sludge. *Water (Switzerland)*, *11*(2), 1-7. https://doi.org/10.3390/w11020303

Cetin, S., & Erdincler, A. (2004). The role of carbohydrate and protein parts of extracellular polymeric substances on the dewaterability of biological sludges. *Water Science and Technology*, *50*(9), 49-56. https://doi.org/10.2166/wst.2004.0532

Chen, X., Han, C., Liu, J., & Hu, Y. (2021). Interpreting strength parameters of strain-softening clay from shallow to deep embedment using ball and T-bar penetrometers. *Computers and Geotechnics*, *138*(April), 104331. https://doi.org/10.1016/j.compgeo.2021.104331

Christine, M., Sene, A., Zhanga, K. K., Penga, S., & Zhanga, Y. (2019). Sludge dewaterability : The variation of extracellular polymeric substances during sludge conditioning with two natural organic conditioners. *Journal of Environmental Management*, *251*(April), 109559. https://doi.org/10.1016/j.jenvman.2019.109559

Comte, S., Guibaud, G., & Baudu, M. (2007). Effect of extraction method on EPS from activated sludge: An HPSEC investigation. *Journal of Hazardous Materials*, *140*(1-2), 129-137. https://doi.org/10.1016/j.jhazmat.2006.06.058

Deng, W., Li, X., Yan, J., Wang, F., Chi, Y., & Cen, K. (2011). Moisture distribution in sludges based on di ff erent testing methods. *Journal of Environmental Sciences*, *23*(5), 875-880. https://doi.org/10.1016/S1001-0742(10)60518-9

Diener, S., Semiyaga, S., Niwagaba, C. B., Muspratt, A. M., Gning, J. B., Mbéguéré, M., Ennin, J. E., Zurbrugg, C., & Strande, L. (2014). A value proposition: Resource recovery from faecal sludge – Can it be the driver for improved sanitation? *Resources, Conservation and Recycling*, *88*, 32-38. https://doi.org/10.1016/j.resconrec.2014.04.005

Erdincler, A., & Vesilind, P. A. (2003). Effect of sludge water distribution on the liquid-solid separation of a biological sludge. *Journal of Environmental Science and Health – Part A Toxic/Hazardous Substances and Environmental Engineering*, *38*(10), 2391-2400. https://doi.org/10.1081/ESE-120023439

FAO. (2011). Quality assurance for animal feed analysis laboratories. FAO Animal Production and Health Manual No. 14. In *Food and Agriculture Organization of the United Nations*.

Flaga, A. (n.d.). *SLUDGE DRYING*.

Freire, F. B., Freire, F. B., Pires, E. C., & Freire, J. T. (2007). Moisture adsorption and desorption behavior of sludge powder. *Environmental Technology*, *28*(11), 1195-1203. https://doi.org/10.1080/09593332808618880

Gold, M., Dayer, P., Christine, M., Sene, A., Seck, A., Niang, S., Morgenroth, E., Strande, L., Gold, M., Dayer, P., Christine, M., Sene, A., Seck, A., Niang, S., Morgenroth, E., & Strande, L. (2016). *Locally produced natural conditioners for dewatering of faecal sludge*. *3330*. https://doi.org/10.1080/09593330.2016.1165293

Gold, M., Ddiba, D. I. W., Seck, A., Sekigongo, P., Diene, A., Diaw, S., Niang, S., Niwagaba, C., & Strande, L. (2017). Faecal sludge as a solid industrial fuel: A pilot-scale study. *Journal of Water Sanitation and Hygiene for Development*, *7*(2), 243-251. https://doi.org/10.2166/washdev.2017.089

Gold, M., Harada, H., Therrien, J. D., Nishida, T., Cunningham, M., Semiyaga, S., Fujii, S., Dorea, C., Nguyen, V. A., & Strande, L. (2018a). Cross-country analysis of faecal sludge dewatering. *Environmental Technology (United Kingdom)*, *39*(23), 3077-3087. https://doi.org/10.1080/09593330.2017.1374472

Gold, M., Harada, H., Therrien, J., Nishida, T., Cunningham, M., Semiyaga, S., Fujii, S., Dorea, C., Nguyen, A., & Strande, L. (2018b). *Cross-country analysis of faecal sludge dewatering*. *3330*. https://doi.org/10.1080/09593330.2017.1374472

Hil, A., Judenne, E., & Remy, M. (2005). The effect of lime treatment on sludge rheological properties. *Technology*, *November*, 1-6.

Houghton, J. I., Quarmby, J., & Stephenson, T. (2001). Municipal wastewater sludge dewaterability and the presence of microbial extracellular polymer. *Water Science and Technology*, *44*(2-3), 373-379. https://doi.org/10.2166/wst.2001.0792

Jin, B., Wilén, B. M., & Lant, P. (2004). Impacts of morphological, physical and chemical properties of sludge flocs on dewaterability of activated sludge. *Chemical Engineering Journal*, *98*(1-2), 115-126. https://doi.org/10.1016/j.cej.2003.05.002

Kopp, J., & Dichtl, N. (2001). *Influence of the free water content on the dewaterability of sewage sludges*. 177-184.

Lee, D., & Lee, S. F. (1995). *Measurement of Bound Water Content in Sludge : The Use of Differential Scanning Calorimetry (DSC)*. 359-365.

Leonard, A., Blacher, S., Marchot, P., & Crine, M. (2002). Use of X-ray microtomography to follow the convective heat drying of wastewater sludges. *Drying Technology*, *20*(4-5), 1053-1069. https://doi.org/10.1081/DRT-120004013

Léonard, A., Blandin, G., & Crine, M. (2014). Importance of Rheological Properties When Drying Sludge in a Fixed Bed. *Laboratory of Chemical Engineering, University*, *III*(Kudra), 102-150.

Li, X. Y., & Yang, S. F. (2007). Influence of loosely bound extracellular polymeric substances (EPS) on the flocculation, sedimentation and dewaterability of activated sludge. *Water Research*, *41*(5), 1022-1030. https://doi.org/10.1016/j.watres.2006.06.037

Lin, F., Li, J., Chai, X., Zhang, Z., & Liu, M. (2020). Determination of water distribution in sludge by a multiple headspace extraction analytical technique. *Journal of Chromatography A*, *1628*, 461449. https://doi.org/10.1016/j.chroma.2020.461449

Liukkonen, M., & Selin, J. (2016). *Water Content Analysis of Sludge using NMR Relaxation Data and Independent Component Analysis*. 317-320. https://doi.org/10.3384/ecp17142317

Mao, F., Zhao, Y., Zhang, Y., Chen, Z., & Yin, L. (2020). Measurement and modelling of moisture distribution and water binding energy of dredged sludge. *Water (Switzerland)*, *12*(12). https://doi.org/10.3390/w12123395

Mao, H., Wang, F., Mao, F., Chi, Y., Lu, S., & Cen, K. (2016). Measurement of water content and moisture distribution in sludge by 1H nuclear magnetic resonance spectroscopy. *Drying Technology*, *34*(3), 267-274. https://doi.org/10.1080/07373937.2015.1047952

Mezger, T. (2020). The Rheology Handbook. In *The Rheology Handbook*. https://doi.org/10.1515/9783748603702

Mikkelsen, L. H., & Keiding, K. (2002). Physico-chemical characteristics of full scale sewage sludges with implications to dewatering. *Water Research*, *36*(10), 2451-2462. https://doi.org/10.1016/S0043-1354(01)00477-8

Mouzaoui, M., Baudez, J. C., Sauceau, M., & Arlabosse, P. (2018). Experimental rheological procedure adapted to pasty dewatered sludge up to 45% dry matter. *Water Research*, *133*, 1-7. https://doi.org/10.1016/j.watres.2018.01.006

Mowla, D., Tran, H. N., & Allen, D. G. (2013). A review of the properties of biosludge and its relevance to enhanced dewatering processes. *Biomass and Bioenergy*, *58*, 365-378. https://doi.org/10.1016/j.biombioe.2013.09.002

Mujumdar, A. S., & Devahastin, S. (2011). *FUNDAMENTAL PRINCIPLES OF DRYING*.

Okada, K., Hayashi, Y., Kumada, S., & Onuki, Y. (2021). Nondestructive Investigation of the Agglomeration Process for Nanosuspensions via NMR Relaxation of Water Molecules. *European Journal of Pharmaceutical Sciences*, *164*(June). https://doi.org/10.1016/j.ejps.2021.105908

Peng, G., Ye, F., & Li, Y. (2012). Investigation of extracellular polymer substances (EPS) and physicochemical properties of activated sludge from different municipal and industrial wastewater treatment plants. *Environmental Technology*, *33*(8), 857-863. https://doi.org/10.1080/09593330.2011.601763

Schaum, C., & Lux, J. (n.d.). *Sewage Sludge Dewatering and Drying*.

Septien, S., Pocock, J., Teba, L., Velkushanova, K., & Buckley, C. A. (2018). Rheological characteristics of faecal sludge from VIP latrines and implications on pit emptying. *Journal of Environmental Management*, *228*, 149-157. https://doi.org/10.1016/j.jenvman.2018.08.098

Simo-Tagne, M., Bennamoun, L., Léonard, A., & Rogaume, Y. (2019). Determination and modeling of the isotherms of adsorption/desorption and thermodynamic properties of obeche and lotofa using nelson's sorption model. *Heat and Mass Transfer/Waerme- Und Stoffuebertragung*, *55*(8), 2185-2197. https://doi.org/10.1007/s00231-019-02577-2

Smidt, E., & Tintner, J. (2007). Application of differential scanning calorimetry (DSC) to evaluate the quality of compost organic matter. *Thermochimica Acta*, *459*(1-2), 87-93. https://doi.org/10.1016/j.tca.2007.04.011

Suzanne N.S. (2010). *Food Analysis Laboratory Manual*. http://cst.ur.ac.rw/library/Food Science books/batch1/Food Analysis Laboratory Manual Second Edition.pdf

Vaxelaire, J., & Cezac, P. (2004). *Moisture distribution in activated sludges : a review*. *38*, 2215-2230. https://doi.org/10.1016/j.watres.2004.02.021

Vaxelaire, J., Mousques, P., Bongiovanni, J. M., & Puiggali, J. R. (2000). Desorption isotherms of domestic activated sludge. *Environmental Technology (United Kingdom)*, *21*(3), 327-335. https://doi.org/10.1080/09593332108618114

Vesilind, P. A., & Hsu, C. C. (1997). Limits of sludge dewaterability. *Water Science and Technology*, *36*(11), 87-91. https://doi.org/10.1016/S0273-1223(97)00673-2

Wang, W., Li, A., & Zhang, X. (2012). DSC and SEM analysis on bound water characteristics in sewage sludge. *Advanced Materials Research*, *347-353*, 2085-2089. https://doi.org/10.4028/www.scientific.net/AMR.347-353.2085

Wu, C. C. (1998). *BOUND WATER CONTENT AND WATER BINDING STRENGTH ON SLUDGE FLOCS*. *32*(3), 900-904.

Xie, S., Wu, Y., Wang, W., Wang, J., Luo, Z., & Li, S. (2014). Effects of acid/alkaline pretreatment and gamma-ray irradiation on extracellular polymeric substances from sewage sludge. *Radiation Physics and Chemistry*, *97*, 349-353. https://doi.org/10.1016/j.radphyschem.2013.07.026

Yang, S. fang, & Li, X. yan. (2009). Influences of extracellular polymeric substances (EPS) on the characteristics of activated sludge under non-steady-state conditions. *Process Biochemistry*, *44*(1), 91-96. https://doi.org/10.1016/j.procbio.2008.09.010

Zhang, W., Xu, Y., Dong, B., & Dai, X. (2019). Characterizing the sludge moisture distribution during anaerobic digestion process through various approaches. *Science of the Total Environment*, *675*, 184-191. https://doi.org/10.1016/j.scitotenv.2019.04.095

5 OUTCOMES OF THE PROJECT AND WAY FORWARD

This section presents the outcomes of the project in terms of the created products, capacity building and knowledge dissemination.

5.1 CREATED PRODUCTS

This project created the following products: (i) dataset and knowledge; (ii) model.

5.1.1 Data generation and knowledge creation

The main outcomes from this project are the generated data and the created new knowledge to better understand moisture boundness in faecal sludge, which is expected to improve the faecal sludge and faeces handling and treatment processes in sanitation systems.

The major take-away message are as follow:

- The limit of bound-unbound moisture varies between 50 to 70% MC, depending on the analytical technique employed and type of faecal sludge. This limit could indicate the extent to which the sludge could be dewatered by a passive or moderate intensity method leading to the removal of unbound moisture. The faecal sludge from VIP latrines and UDDT seems to have unbound/bound moisture limit occurring at lower MC than for ST sludge, signifying that they could be potentially dewatered at lower MC. The methods to determine the limit of unbound-bound moisture differ slightly but the difference remain acceptable (below 10%).
- The capillary (or interstitial) moisture is the more abundant type of bound moisture. It can be found approximately between around 70 to 20% MC. It is responsible of the lumpy consistency and high stickiness behaviour exhibited by faecal sludge in this MC range (observed by a peak of the cohesion and adhesion forces). Capillary moisture can removed by dewatering until a certain extent, but it will probably require high intensity mechanical force for this. Thermal drying is required for the complete removal of the capillary moisture.
- Below 30% MC, the remaining moisture is mostly in the form of vicinal moisture (adsorbed at the surface of the solid particles by polylayers) and internal moisture (being part of the chemical structure of the solid material and biological bodies). The removal of this type of moisture can only be achieved by thermal drying, but this will lead to an exponential increase of the energy consumption (because the energy to remove the vicinal and internal moisture is significantly higher than the latent heat of water vaporization). Nonetheless, drying could be stopped below 30% MC, and might not require the removal of the vicinal and internal moisture. Indeed, the sludge has the form of a granular solid at this point and could be considered safe in terms of pathogens since the remaining moisture is too bound for most of the microbes development and survival.
- Below 10% MC, the residual moisture can be polywaters found as a monolayer adsorbed at the surface of particles and as vicinal moisture. High amount of thermal energy would require to reduce the MC to this level.
- The temperature influences mainly the vicinal and internal moisture distribution. Increasing the temperature allows to reduce slightly the fraction of this type of moisture.
- In general, faecal sludge drying requires a considerably higher thermal energy input than the latent heat of water vaporization because of the moisture boundness. In order to lead to energy savings, drying could be stopped at around 30% MC where the sludge could be considered safe, i.e. after removing the unbound and capillary moisture.

- The removal of the unbound and capillary moisture leads to significant changes in the physicochemical and mechanical properties of the faecal material, by converting it into a granular solid from a slurry or viscoelastic consistency and passing through an intermediary sticky lumpy phase. These changes must be considered in the design of dewatering and thermal drying units.
- The moisture boundness between the different types of faecal sludge differ slightly, but a more marked difference was observed with respect to fresh faeces. The average fresh faeces (type III and IV in the Bristol Stool Chart) could be more difficult to dewater and dry than faecal sludge due to a higher moisture boundness.

Once the missing work will be achieved, this will allow to differentiate the different types of capillary moisture based on the capillaries characteristics and to understand how the physiochemical and structural properties of faecal sludge can explain the moisture boundness.

5.1.2 Mathematical modelling

The results from the sorption isotherms were modelled. Through the followed modelling approach, a more integral predictive model could be developed. This would be very useful to predict the final MC that can be obtained at certain air conditions and the level of moisture boundness achieved, which will provide valuable information about the properties of the material such as the consistency and disinfection level.

5.2 CAPACITY BUILDING

The project involved several students of varied ethnic origin, nationality, group age and age, demonstrating its inclusiveness. The details of the involved students are summarized in Table 5.1.

Table 5.1. Details of the students involved in the project

Name	Gender	Age	Race	Nationality	Degree
Arunkumar Rayavellore Suryakumar[1]	Male	44	Indian	India	PhD student
Larona Malope[1]	Female	26	Black	Botswana	MSc student
Tanaka Chatema[1]	Female		Black	Zimbabwe	graduated MSc
Sherilee Megan Pillay[2]	Female	24	Indian	South Africa	BSc
Rahul Raghunanan[2]	Male	23	Indian	South Africa	BSc
Sinenhlanhla Ntombenhle Mavundla[2]	Female	23	Black	South Africa	BSc
Samkelisiwe Ndlovu[2]	Female	24	Black	South Africa	BSc
Farida Gitonga[3]	Female	28	Black	Kenya	MSc student
Lone Morubisi[3]	Male	31	Black	Botswana	MSc student

Affiliations
[1] WASH R&D Centre, UKZN
[2] Chemical Engineering, UKZN
[3] Pan-African University of Water and Energy Science (PAUWES)

5.3 KNOWLEDGE DISSEMINATION

The work from this project has been presented at the following conferences:

- Oral presentation of the PhD student at the UKZN Postgraduate Research and Innovation Symposium (PRIS) 2021, held virtually from the 9th and 10th December 2021;
- Poster presentation at the 42nd Water Engineering and Development Centre 2021 Conference, held virtually from the 13th and 15th September 2021;

- Oral presentation and conference paper at the 22nd International Drying Symposium 2022, held during the 25th to 27th June 2022 in Worcester, United States;
- Poster presentation at the DevRes 2022 Conference, held from the 22nd and 24th August 2022, in Uppsala, Sweden;
- Oral presentation at the Water Institute of Southern Africa 2022 Biennial Conference and Exhibition, held from the 28th to 30th September 2022 in Pretoria, South Africa;
- Oral presentation at the IHE Annual PhD Symposium, held as a hybrid event during the 13th and 14th October 2022;
- Flash presentation at the UKZN PRIS, held virtually during the 8th and 9th December 2022;
- Oral presentation at the FSM (faecal sludge management) 7 Conference, held from the 19th to the 23rd February 2022 in Abidjan, Ivory Coast;
- MSc dissertation from Lone Morubisi and Farida Gitonga (students from PAUWES).

The MSc dissertation of Larona Malope is expected to be available within the half of 2023, whereas the PhD dissertation of Arunkumar Rayavellore Suryakumar may be ready by the end of 2023. Three to four papers issued from the experimental work of this project are intended to be prepared and published in peer-review journals within the next two years (about the dewaterability of different sludges matrixes, sorption isotherms, hydraulic properties and characterization of the bound moisture).
The access to the Conference material, dissertations and papers can be found in the Supplementary Material section. It is important to note that the future dissertations and papers related to this project will be shared once they will published and will be added to the library.

5.4 WAY FORWARD

Not all the targets set in the initial proposal will be accomplished in this project, among which:

- Determination of the moisture boundness in fresh faeces;
- Study of methods to disrupt the moisture boundness for the improvement of the dewatering and drying processes (i.e. methods to decrease the moisture boundness and increase the proportion of unbound moisture).

These targets were discarded from the project program due to the lack of time for their completion before the end of the project. The major reason of this is the overall of the delays accumulated by the COVID-19 restrictions and negative effects in the labour dynamics. Moreover, the collection of fresh faeces from donors was very difficult in the period of sanitary restrictions.

A new proposal was submitted during the WRC 2022 open call to carry on the research initiated in this project and complete the unachieved targets. The proposal results were successful and the project was approved for funding starting from April 2023 to March 2026 (project number 2023/2024-01441). The focus of the proposed project is to find methods to improve the dewatering and drying process by altering the moisture boundness in faecal sludge and fresh faeces. The proposed project will also look at methods to mitigate sludge stickiness, which is a property directly related to moisture boundness. Stickiness is a serious problem encountered in various treatment process, causing fouling and clogging issues.

The new project will include collaboration with Cranfield University and Melbourne University, which already have shown interest in this topic during the reference group meeting. Currently, Cranfield University is exploring pre-treatment methods to improve the anaerobic digestion of sewage sludge. We suspect that these methods could also decrease moisture boundness, leading to a dewatering and drying potential improvement, so that they could be incorporated in the research plan of the proposed project. The University of Melbourne has a valuable understanding of the dewatering of the sludge, which could be useful for the project.

The current collaboration with the University of Birmingham could be continued in the new project, as they can bring their expertise in material science and surface properties to find methods to reduce the stickiness of faecal sludge.

6 APPENDIX: HYDRAULIC TESTS

The characterisation of the hydraulic properties of faecal sludge allows to understand the mobility of moisture within the material, leading to a better understanding of moisture boundness. This section describes the experimental setup and methodology of the hydraulic tests that will be conducted, and the preliminary results. This work was intended to be completed before the end of the project but this was not possible due to several challenges and difficulties. Once this work will conducted, the results and outcomes from the hydraulic tests will be presented in the dissertation of the PhD student and they will be the subject of a paper to publish in a peer review journal. These documents could be accessed from the link shown in the Supporting Documents section (expected to be available by the end of 2023 or beginning of 2024). An updated version of this report with the missing results might also be uploaded in the library.

6.1 INTRODUCTION

This research aims to develop and test a laboratory technique to determine bound and unbound moisture in faecal sludge from different OSS systems using water retention curves. The method to characterise the water retention properties will be adapted from soil science to faecal sludge. A hydraulic pressure cell, where compressed air is introduced to sludge in an airtight cell to displace capillary moisture from sludge, will be utilised to determine the hydraulic conductivity and capillary pressure. Like soil, faecal sludge contains physically and chemically bound, capillary, film and intracellular moisture (Volarovich and Churaev, 1968). However, unlike most soils, faecal sludge has a high organic content that decreases dewaterability. Therefore, sludge is predicted to have low hydraulic conductivities and high capillary pressure analogous to organically rich peat soils (Wong et al., 2009).

The soil's hydraulic properties influence water contained in unsaturated soils, i.e. the water content depends on the suction exerted in the soil (Majdeddin Mir Mohammad Hosseini et al., 2011, Miller et al., 2002). Therefore, soil water content is a function of soil matric potential (Ψ_m), and the relationship between the two parameters can be expressed by plotting volumetric moisture content (MC) against matric potential. The soil water retention curves (SWRC) describe the amount of water retained in the soil at given suction, as Figure 6.1 illustrates for different soil types. The curves predict soil water storage and reflect the ability of soil to prevent water from being expelled or released from its structure (Majdeddin Mir Mohammad Hosseini et al., 2011). This hydraulic property is crucial in managing water and predicting solute and contaminant movement in the environment as well as the availability of water to plants. The SWRC can be obtained using pressure filtration in the laboratory.

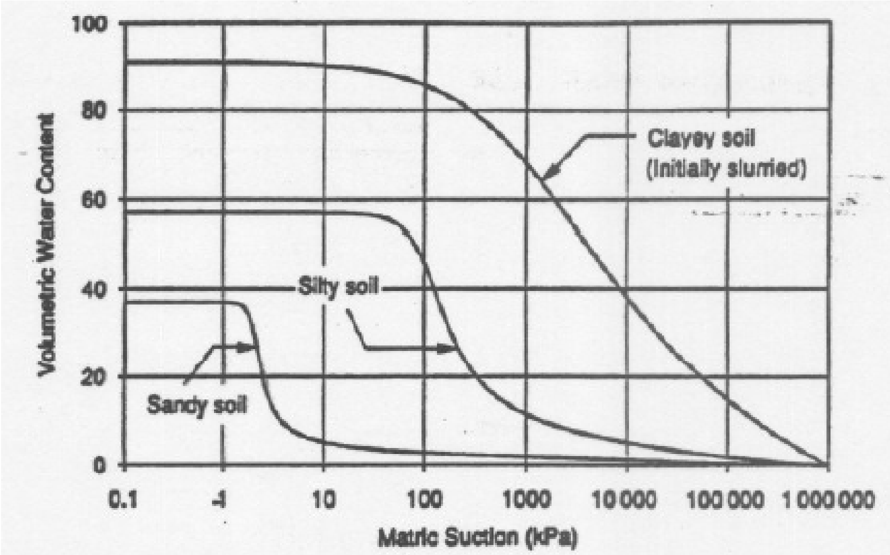


Figure 6.1. Soil-Water Characteristics Curves for different soil types (Leong and Rahardjo, 1997)

Water retention curves will be determined for different types of faecal sludge samples to characterise the moisture boundness in faecal sludge. This data will be instrumental in characterising the capillary moisture in faecal sludge and determining the distribution of capillary moisture as a function of the capillary pressure. This section describes the method that will be employed to determine the hydraulic conductivity in faecal sludge.

6.2 MATERIAL AND METHODS

6.2.1 Feedstock

Faecal sludge samples used in the present work were sourced from different onsite sanitation (OSS) systems in KwaZulu-Natal, South Africa. The list included ventilated improved pit (VIPs) latrines, urine-diverting dehydrating toilets (UDDTs) and septic tanks (STs). VIP and UDDT sludge was collected from the outskirts of Durban and the septage from Pietermaritzburg. The experimental work will use composite faecal sludge mixtures from each OSS system.

6.2.2 Hydraulic conductivity experimental rig setup

Figure 6.2 shows the experimental setup for the filtration test. Air from a compressor is regulated to desired values and flows into the pressure cell, which exerts pressure on the sludge sample in the core. Since air cannot flow past the sample, it exerts a physical force on the sludge that forces water within the sludge out through the ceramic filter and up the burette, which records volume change at each pressure value. The different components of the hydraulic conductivity experimental test rig are shown in Figure 6.2.

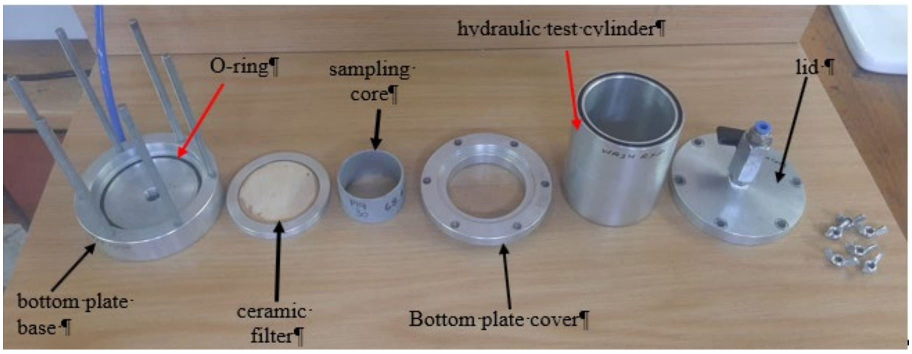


Figure 6.2. The different components that make up the pressure cell

Pressure gauges were installed on each pressure cell (Figure 6.3) to measure the actual working pressure inside the pressure cell to reduce data inaccuracy. This is to adjust the primary air supply to maintain accurate pressure in the system in case of any pressure drops during testing.

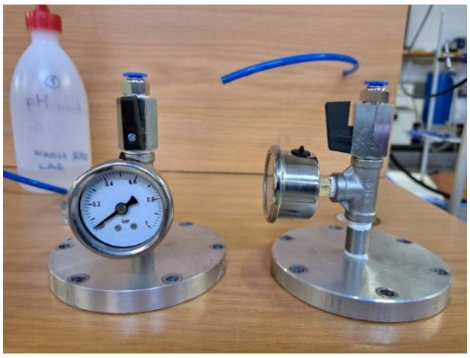


Figure 6.3. Pressure gauges fixed on pressure cell lids

The hydraulic system underwent further modification to include three (Figure 6.4) (instead of two) pressure cells that enable simultaneous triplicate measurements, as the equilibration time at given pressure commonly exceeds two weeks in soils.

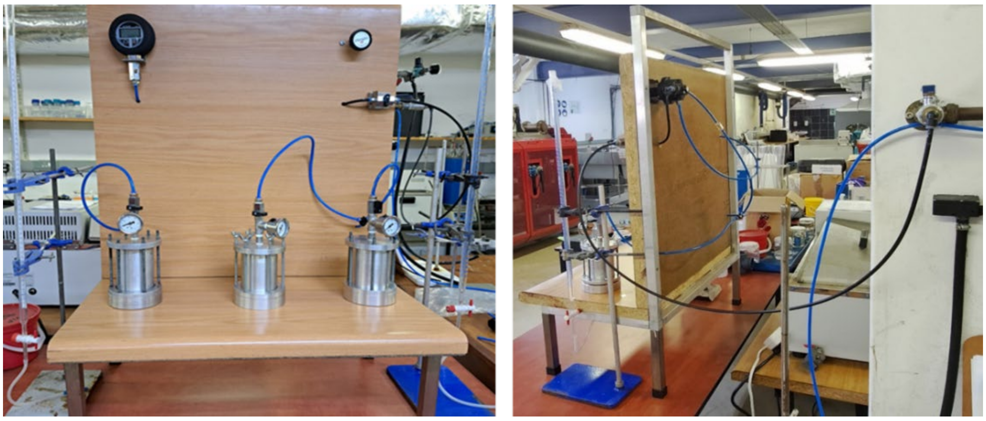


Figure 6.4. The Hydraulic system: modified to accommodate three pressure cells connected to the air supply at the top and burettes at the bottom (left). The air supply line setup that connects the main supply from the compressor to the cells

6.2.3 Methodology

The pressurised hydraulic cell experiments will provide data to plot water retention curves for the faecal sludge which will be used to classify the capillary-bound water fraction in faecal sludge. This lab technique was adapted from Lorentz et al. (2001) and is based on introducing air at increasing pressure in the flow (pressure) cell to expel water from faecal sludge. The increments in air pressure are only applied when the sample MC achieves equilibrium.

The pressure cell bottom plate is connected to a burette which will measure moisture lost by the sample (through water volume change) for each test pressure. Then the saturated sample is placed in the bottom plate (Figure 6.5) and secured by the bottom plate cover and nuts. A hydraulic cylinder is then placed on top of the bottom plate cover, covered by the lid, and secured by wingnuts. Each component has O-rings that must be greased to prevent air leaks from the pressure cell during testing. The piping from the air supply (compressor) is inserted in the fitting in the lid. The piping is connected to a regulator (to adjust and control the pressure going to the cells) and a central pressure gauge to read the regulated air pressure introduced to the hydraulic system. The test begins by opening the valves on the regulator and the pressure cells noting the pressures.

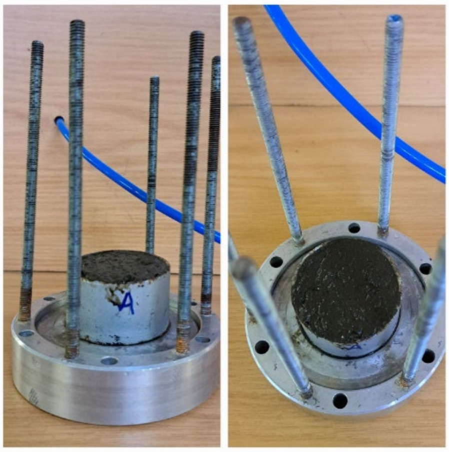


Figure 6.5. Saturated faecal sample placed in the bottom plate

6.2.3.1 Sample preparation

Faecal sludge is packed into the core holder sitting on a saturated ceramic filter, and saturated with distilled water. Saturation serves to eliminate air pockets in the sample before testing. If there is no air in the soil, capillary and film water becomes free water without a surplus bond energy (Andriesse, 1988). The sample is saturated by placing it in a closed container with water to prevent evaporation (Figure 6.6).

Faecal sludge has a high MC of about 78%, close to its saturation point. Comparably, saturated peats have MC ranging from 71-91% and high organic matter content (Andriesse, 1988), which suggests the suitability of methods of measurement of water retention characteristics in peat soils to faecal sludge application.

Figure 6.6. Faecal sludge in a core undergoing saturation in water via a saturated ceramic filter (the container lid is absent for more clarity)

- Sample saturation

Since sample saturation MC is an essential parameter in hydraulic property testing, it has to be done and determined accurately. However, with raw faecal sludge MC close to saturation values, the best conditions for faecal sludge saturation have to be determined. Therefore, samples were saturated for 12, 24 and 72 h, and the MC compared with the initial MC. The results in Table 6.1 indicate that raw sludge moisture did not deviate from the saturation MC. Also, there is no variation in the MC regardless of duration of saturation. Nonetheless, it is essential to standardise the method so samples will be saturated for 24 hrs before being transferred to pressure cells.

Table 6.1. MC of faecal sludge before and after saturation

Raw sludge MC (%)	12-h saturation MC (%)	24-h saturation MC (%)	72-h saturation MC (%)
79	77	79	79
76.4	76.8	78.7	-
79.5	-	79.21	78.0

6.2.3.2 Hydraulic rig testing procedure

The hydraulic properties are determined from simultaneous measurements of the faecal sludge water pressure head and volumetric water content (Majdeddin Mir Mohammad Hosseini et al., 2011). The MC of the sludge sample is determined at various matric suctions to make up a water retention curve (Leong and Rahardjo, 1997). The MC at saturation is determined for each sample type by drying saturated samples in the oven for 24 hrs at 105°C. The MC, intermediate and residual volumetric water contents (θ_r) are determined through calculations based on the volume of the moisture lost from the sample overall after pressure filtration. In this study, pressure will be stepped up in 0.05 or 0.1 bar intervals until 1 bar and at a lower resolution above this range (at 1 to 7 bar pressure). The pressure is only increased when the volume collected at a particular matric suction is constant and no longer increasing.

The obtained data is then used to calculate the volumetric MC, the pressure head and predetermined van Genuchten parameters to plot a matric pressure head (cm) against volumetric MC (cm^3). The relationship between pressure head (h) and volumetric MC (Ɵ) is given in equation (1) (van Genuchten, 1980) :

$$\theta = \theta_r + \frac{(\theta_s - \theta_r)}{[1+(\alpha h)^n]^m}$$ **Equation** (1)

Where:
Ɵ – dimensionless volumetric MC
θ_s – MC of a saturated sample
θ_r – residual MC in a sample
α, n and m – independent parameters estimated from the water retention curve

6.2.3.3 Measurement of the porosity

A high organic matter content introduces instability of the pore structure upon drying, which may lead to inaccuracies in determining hydraulic properties. These changes also apply to peat soils commonly having organic matter content > 75% (Andriesse, 1988). Similarly, faecal sludge consists mainly of organic matter, approximately 80%, which causes shrinkage of the sample upon drying and changes in the sample porosity. Porosity indicates the percentage of the sample volume occupied by pores and is determined using pequation 2.

$$Soil\ porosity = [1 - (Bulk\ density/Particle\ density)] \times 100$$ **Equation (2)**

Methods of determining the dry bulk density and particle density were adapted from a soil science manual by Bowen (2022) and are being investigated. The faecal sample is dried at (i) 105°C in the lab oven for 24 hrs and (ii) air dried (around 40°C) for 72 hrs and ground to a relatively homogenous matrix.

- Determination of bulk density

The powder is added and compacted in a spatula of known volume. The mass of the raw faecal sludge that fits in the spatula is then measured using an analytical mass balance. Table 6.2 shows that case (i) bulk density ranged between 0.47-0.57 g/cm^3, averaging at 0.50 g/cm^3. Tests on air-dried samples are yet to be conducted.

Table 6.2. Bulk density of oven-dried raw faecal sludge at 105°C for 24 hrs

	Volume (cm^3)	**Measured mass (g)**	**Bulk density (g/cm^3)**
	5	2,8621	0,5724
	5	2,5529	0,5106
	5	2,4118	0,4824
	5	2,3680	0,4736
Average	**5**	**2,5487**	**0,5097**

- Determination of particle density

A predetermined mass of raw faecal sludge is added to graduated cylinders containing water and left to sink to the bottom. The displaced volume is recorded, and the density is determined through calculation. The particle density ranged from 2.5-5.0 g/cm^3 regardless of the drying method, as evidenced in Table 6.3.

Table 6.3 Particle density determination using water volume displacement

	Drying temp ◦C (duration)	Replicate	Solvent volume (cm^3)	FS mass (g)	Final volume (cm^3)	Displaced volume (cm^3)	FS density (g/cm^3)
	105 (24h)	a	50	5,0116	51	1	5,0
	105 (24h)	b	50	5,0143	52	2	2,5
	105 (24h)	c	50	5,0120	51	1	5,0
Average	**105 (24h)**		**50**	**5,0126**	**51**	**1**	**4,2**
	40 (72h)	a	50	5,0057	51	1	5,0
	40 (72h)	b	50	5,0102	52	2	2,5
Average	**40 (72h)**		**50**	**5,0080**	**51,5**	**1,5**	**3,8**

Typically, soil is air-dried to determine dry bulk density in soil science. However, high organic content materials like faecal sludge experience progressive shrinkage when drying, unlike typical soils making an accurate determination of bulk density and particle density of dry faecal sludge challenging. Air-drying is assumed to have less altering effects on sample structure, unlike standard oven drying at 105°C. Therefore, more bulk density and particle density tests still need to be conducted to establish the duration of air drying, which gives comparable results to the standard method of completely drying faecal sludge. This will ensure conclusive values for bulk density and particle density to calculate the faecal sludge porosity accurately. Additionally, dried and repacked samples are likely to show better repeatability. However, the best sample preparation protocol will be decided based on the air drying test results.

6.3 PRELIMINARY HYDRAULIC PROPERTY TEST RESULTS

All preliminary tests detailed in the report made use of faecal sludge from VIP toilets. The preliminary Table 6.4 is an example of data obtained from a trial run for a 44.82 g faecal sludge sample with an initial MC of 76.82%.

Table 6.4. Results from a trial hydraulic property testing run

Pressure (bar)	Cumulative time (hrs)	Final moisture (%)
0.00	–	76.82
0.10	100.6	63.95
0.15	125.5	61.69
0.20	144.8	59.61
0.25	166.0	56.57
0.30	239.5	52.39
0.35	266.2	51.27
0.40	266.2	49.62
0.45	289.4	47.05
0.50	359.9	45.66

Table 6.5 shows results for a duplicate test trial currently in progress for a faecal sludge with an initial MC of 79.52%. Testing has been done from 0.05 to 0.20 bar with each pressure value set for an average of 7 days, save for 0.20 bar which has been running for 16 days. The plan is to increase the filtration pressure till reaching a maximum value of 0.9 bar. Results point to 47.37 g of faecal sludge reducing from an initial MC of 79,52% to 68,06%, whereas the 49.28 g sample had its moisture decline to 63.41%. N/B: Test progression has been gradual because of intermittent power supply interruptions affecting the air supply.

Table 6.5 Duplicate hydraulic property test trial

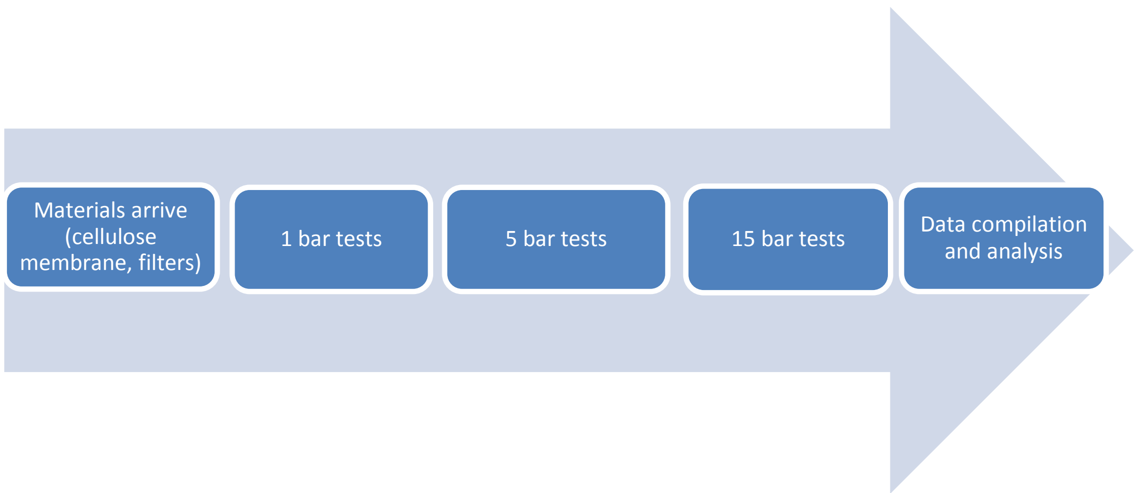
Pressure (bar)	Hydraulic test cell 1	Hydraulic test cell 2
	Sample mass = 47.37 g	Sample mass = 49.28 g
	Final moisture (%)	**(Final moisture %)**
0.00	79,52	79,52
0.05	77,53	77,46
0.10	74,31	72,63
0.20	68,06	63,41

6.4 IMPROVEMENT ASPECTS OF THE PRESSURE CELL RIG SETUP

Currently, testing pressure goes up to 1 bar (equipment limitation). However, this is insufficient to have a large spectrum of water retention curve, so we may miss important data above 1 bar. Therefore, additional equipment is needed to operate at higher pressure. Ceramic filters of 5 and 15-bar air entry value are commonly used in soil science to determine the WRC from saturation to near-dry conditions. Therefore, the hydraulic system with three pressure cells set up will allow reliable determination of the van Genuchten parameters (α, n and m) from the water retention curve.

Moreover, colloids in faecal sludge have been observed in the collected liquid, possibly causing ceramic filter blockage, thus compromising the efficiency of the filters. To overcome this issue, semi-permeable cellulose membrane will be used to prevent colloids from entering the ceramic filter. Procuring more ceramic filters and cellulose membranes has been difficult due to the lack of suppliers, financial restrictions, and the unique design of the hydraulic cells. However, the order has been placed with a local manufacturer, and the 5- and 15-bar filters will be custom-made to fit the experimental rig (estimated delivery in May).

6.5 SCHEMATIC FOR UPCOMING PROJECT ACTIVITIES



6.6 REFERENCES

ANDRIESSE, J. 1988. *Nature and management of tropical peat soils*, Food & Agriculture Org.
BOWEN, M. W. 2022. Principles of Soil Science Exercise Manual. *In:* OSHKOSH, U. O. W. (ed.).
FLAGA, A. Sludge drying. Proceedings of Polish-Swedish seminars, Integration and optimisation of urban sanitation systems. Cracow March, 2005. 17-18.

LEONG, E. C. & RAHARDJO, H. 1997. Review of soil-water characteristic curve equations. *Journal of geotechnical and geoenvironmental engineering,* 123**,** 1106-1117.

LORENTZ, S., GODA, P. & PRETORIUS, J. 2001. *Hydrological process research: experiments and measurements of soil hydraulic characteristics*, Water Research Commission.

MAJDEDDIN MIR MOHAMMAD HOSSEINI, S., GANJIAN, N. & PASHANG PISHEH, Y. 2011. Estimation of the water retention curve for unsaturated clay. *Canadian Journal of Soil Science,* 91**,** 543-549.

MILLER, C. J., YESILLER, N., YALDO, K. & MERAYYAN, S. 2002. Impact of soil type and compaction conditions on soil water characteristic. *Journal of Geotechnical and Geoenvironmental Engineering,* 128**,** 733.

VAN GENUCHTEN, M. T. 1980. A closed-form equation for predicting the hydraulic conductivity of unsaturated soils. *Soil science society of America journal,* 44**,** 892-898.

VOLAROVICH, M. & CHURAEV, N. Application of the methods of physics and physical chemistry to the study of peat. The 2nd International Peat Congress, Leningrad. Ed. by RA Robertson, 1968. 819-831.

WONG, L. S., HASHIM, R. & ALI, F. 2009. A review on hydraulic conductivity and compressibility of peat. *Journal of Applied Sciences,* 9**,** 3207-3218.

SUPPORTING DOCUMENTS

Previous Deliverables from this project

Conference material related to this project

Publications related to this project (it is important to note that the future dissertations and papers related to this project will be uploaded in this shared folder, as well as the updates to this report)