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Experimental Determination of Moisture Sorption Isotherm of Fecal Sludge

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Abstract: Dewatering and drying of fecal sludge (FS) is a key treatment objective in fecal sludge management as it reduces volume (thereby reducing emptying frequency and associated transportation costs), inactivates pathogens, and is beneficial and/or necessary to resource recovery activities such as composting and combustion as fuel. However, studies on dewatering performances of FS are limited. The physical water distribution of such matrices is not fully understood, limiting the progress in the development and optimization of FS dewatering technologies. The objective of this study is to present a gravimetric method intended to assess the dewatering characteristics and associated modelling of FS through moisture sorption isotherms. Samples were placed in airtight jars containing different saturated salt (NaOH, $CaCl_2$, NaCl, KCl, K_2SO_4) solutions to reproduce a range of relative humidity values (6 to 97%). Results confirmed the achievement of characteristic sigma-shaped moisture sorption isotherms with increasing moisture adsorption at higher values of relative humidity. Furthermore, experimental data best fit the three-parameter Guggenheim–Anderson–de Boer (GAB) model. This method can be replicated to contribute critical data about the characterization of fecal sludge, a seriously under-researched matrix.

Keywords: desiccation; drying; fecal sludge management; moisture sorption isotherms; sanitation

1. Introduction

In low-income countries, the most common forms for excreta disposal are on-site, non-sewered sanitation systems (NSS) [1]. If safely managed, these sanitation facilities can provide a hygienic and affordable method for excreta disposal, meaning that the fecal sludge (FS) is safely contained, collected, treated, and disposed of. Such systems would contribute to the Sustainability Development Goals technology spectrum for *safely managed sanitation services* [2]. However, in many regions of the world, current FS management practices and approaches are insufficient and often unsafe. Many municipalities have not yet put the necessary strategies, policies, and budgets in place to maintain these NSS systems (e.g., desludging and treatment), resulting in the contamination of the environment [3] and spread of diarrheal diseases [4]. It is estimated that only 26% of urban and 34% of rural sanitation services worldwide effectively prevent human contact with excreta along the entire sanitation chain and can therefore be considered safely managed [5]. Additionally, inadequately managed sanitation facilities have degraded water quality in most rivers across Africa, Asia, and Latin America, directly affecting quality of life, working capacity of the inhabitants, education, and economies [6]. Better methods for the safe management of FS from NSS are urgently needed.

The moisture content of fresh faeces ranges from 63 to 86% and the fecal sludge collected by vacuum trucks in urban areas can contain more than 95% water [7,8]. As FS has a high water content, dewatering processes (i.e., drying beds, thermal drying, centrifuges, etc.) for the safe and cost-effective transportation of FS are advantageous. These techniques can reduce water content, resulting in benefits such as reductions in associated transport costs, as well as potentially enhanced pathogen inactivation [9,10], which can reduce the health risks of handling the fecal matter. However, studies on the dewatering of FS are relatively limited given its importance. The physical water mobility of FS is not fully understood, possibly limiting the progress in the development and optimization of FS dewatering technologies. Future research is needed to develop predictive models of sludge characteristics on dewatering, based on a fundamental understanding of FS dewatering mechanisms [8].

Moisture sorption isotherms (MSIs) are a graphical representation describing the sorption process of water molecules into a specific material at a specific temperature. They illustrate where water molecules are progressively and reversibly released from hygroscopic forces in biological material as a result of mainly capillary effects and direct bonding [11]. This method is largely used in the food industry [12], and has been successfully used to describe the sorption behavior of activated sludge [13,14]. However, direct transfer of moisture distribution characteristics from wastewater sludge to FS is not possible due to considerable differences in composition (e.g., organics, total solids, etc.) [3].

Given the lack of desiccation-related data on FS and the potential for MSIs, our aim was to test the potential of an experimental method to produce moisture sorption isotherm data for FS and to then evaluate various mathematical models to determine which best describes the datasets.

2. Materials and Methods

A static gravimetric method was adapted [15] to determine the sorption characteristics of the FS samples. Two initial masses of FS (1.5 g and 5.0 g) were tested to study the effect of the mass of the samples on the water sorption isotherm. Three commonly used mathematical models were then evaluated for best fit with the experimental results.

2.1. Fecal Sludge Sampling and Initial Characterization

Surficial (i.e., top 5 to 10 cm exposed to air) FS was collected from a decentralized sanitation system in Quebec City (QC) that has urine separation and separate toilet paper disposal. The age of the FS was estimated to be 24 h or less. Once collected, the samples were transported in a closed plastic container and characterized for their total solids, volatile solids, and water content using methods 2540G from the Standard Methods [16]. The time between sampling and analysis was no more than 3 h.

2.2. Preparation of Saturated Salt Solution

Five saturated salt solutions were used to reproduce a range of relative humidity (RH) values between 0.06 and 0.97 (Table 1) [17]. Each American Chemical Society (ACS) grade salt was mixed with distilled water (approximately 100 mL) until a solution with excess crystals was formed. The saturated salt solutions were poured into Mason jars to a depth of 1 cm (Figure 1).

Table 1. Relative humidity of saturated salt solutions at 35 °C [17].

Salt	Relative Humidity (%)
Sodium hydroxide ($NaOH$)	6
Calcium chloride ($CaCl_2$)	24
Sodium chloride ($NaCl$)	75
Potassium chloride (KCl)	82
Potassium sulphate (K_2SO_4)	97

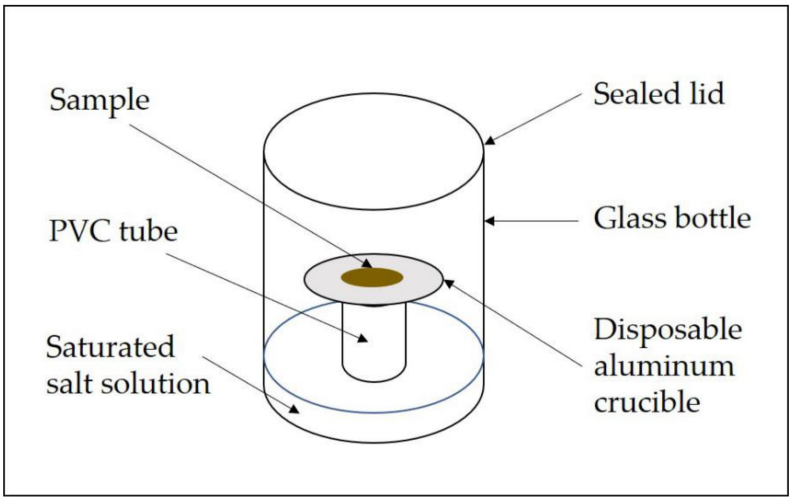

Figure 1. Experimental set-up for the moisture sorption isotherm (MSI) determination of fecal sludge (FS).

2.3. Determination of Sorption Isotherms

The FS samples were manually homogenized using a stainless-steel spatula. One drop of thymol was brushed onto the bottom of each aluminum crucible to prevent mold growth. Then, the samples were placed and weighed in the aluminum crucibles. Samples were spread to have approximately the same circular shape with a radius of 0.5 or 1 cm for the 1.5 g and 5.0 g samples, respectively. Each crucible was then placed on a PVC tube fixed to an airtight Mason jar with a saturated salt solution to control the relative humidity (Figure 1). These Mason jar desiccators were incubated at 35 ± 1 °C. Samples were weighed at regular intervals (24 h) until their mass varied by less than 2% W/W, the adopted operational threshold for equilibrium moisture content determination (approximately two weeks). It was ensured to limit the opening time of the jars to avoid humidity loss or gain. Finally, the equilibrium water content was calculated from the dry mass providing a point of the isotherm of sorption when coupled with RH values.

2.4. Mathematical Models of Sorption Isotherms

Table 2 lists the mathematical isotherm models used in this study for comparison to the empirical data. Models were selected based on their effectiveness for describing isotherms of high moisture content of biological materials (e.g., municipal sludge, food, plants). Nonlinear data analysis and curve fitting were executed in the R development core system (2013) [18]. The quality of each model was assessed with regard to the standard error of estimate and the residual standard error. Additionally, the Student's test was applied on regression coefficient for significance (p-Value < 0.05).

Table 2. Isotherm models used for fitting experimental data.

Isotherms	Equation	Parameters
Brunauer–Emmett–Teller (BET)	$M = \frac{M_0 C a_w}{(1-a_w)(1+Ca_w-a_w)}$	M = Moisture content (MC) C = constant M_0 = Monolayer MC
Guggenheim–Anderson–de Boer (GAB)	$M = \frac{M_0 C K' a_w}{(1-K'a_w)(1-K'a_w+Ca_wK')}$	M = Moisture content (MC) C, K' = constants M_0 = Monolayer MC
Flory–Huggins (FH)	$M = A \exp(Ba_w)$	A, B = constants

3. Results and Discussion

3.1. Moisture Sorption Isotherm from Two Different Sample Masses

Figure 2 shows representative sorption isotherm results for 1.5 g and 5.0 g samples. The boxplots present data in quartiles, allowing comparisons between the different RH results. Overall trends for both sample masses indicate achievement of sigma-shaped moisture sorption isotherms characterized by two characteristic bends. The first bend occurs at an RH of 0 to 6% and the second one at around 75 to 82% for both sample masses. According to Labuza (1984) [19], this is caused by the additive effects of Raoult's law, capillary effects, and surface water interaction. More precisely, RH values between 0 and 6% are suggested to result from the Van der Waals forces on water molecules and constitutes a molecular monolayer on the surface of the product [15]. Then, between 6% and 75% RH, water molecules are adsorbed on the saturated monolayer, resulting in the creation of more layers [15]. In this region, water is held in the solid matrix by capillary condensation. This water is thus available as a solvent for low-molecular-weight solutes and for some biochemical reactions [12]. Finally, between 75 and 97% RH, water is suggested to be bonded due to macro-capillary forces or as part of the fluid phase in high-moisture materials. This shows early all the properties of bulk water, and thus can act as a solvent [12]. Microbial growth becomes a major deteriorative reaction at this zone as water is available for bacteria (pathogens) [12]. Finally, for RH = 100%, the curve tends to an asymptote which corresponds to free water [14]. These sigma-shape isotherms that FS sample seem to correspond to have been reported for most food products with high moisture content [19]. This is as expected since the composition of FS is like most food in terms of molecules present, namely carbohydrates, lipids, and proteins [7].

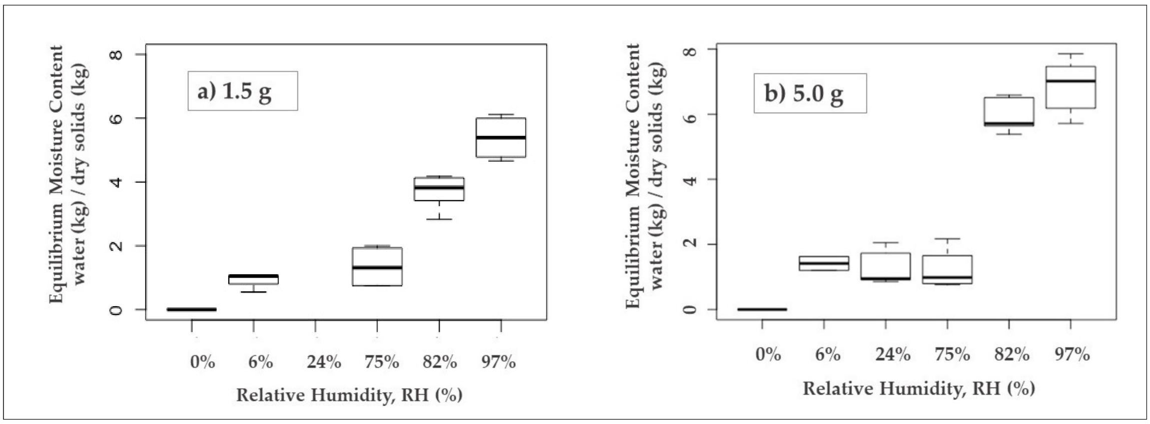

Figure 2. Box and whisker plots (min/max, lower/upper quartiles, and median) of equilibrium moisture content data for each given relative humidity at a temperature of 35 °C and an initial FS sample with masses of (**a**) 1.5 g and (**b**) 5.0 g.

A qualitative comparison of the experimental data at 35 °C, shown in Figure 2, suggests that the equilibrium moisture content reached by the 5.0 g sample mass is higher than that reached by the 1.5 g sample mass. Quantitatively, the sample mass has a significant impact on equilibrium moisture content at a given relative humidity ($\alpha < 0.001$).

3.2. Modelling of Isotherm

Figure 3 depicts experimental data fit with Brunauer–Emmett–Teller (BET), Guggenheim–Anderson–de Boer (GAB), and Flory–Huggins (FH) models at 35 °C and for both 1.5 g and 5.0 g initial sample masses. Table 3 summarizes the parameter estimates, standard error, and the residual standard error (RSE) to determine the best data fit for the model. The FH model was rejected for both sample masses, and the BET model was rejected for the 1.5 g sample mass, since model parameters (i.e., constant) failed the Student's test for significance (p-Value < 0.05). The GAB and BET models are both valid for the 5.0 g sample mass since the equation parameters (C, M_0, K′) were satisfactory (Pr (>|t|) = 0 to 0.05). For BET, M_0 is associated with the moisture content (dry basis) corresponding to an adsorbed monolayer (BET) and C and K′ are constants related to the temperature effect. Further testing should be performed across the range of humidity values to improve the calibration of the models and provide better assessment of the most suitable one for FS.

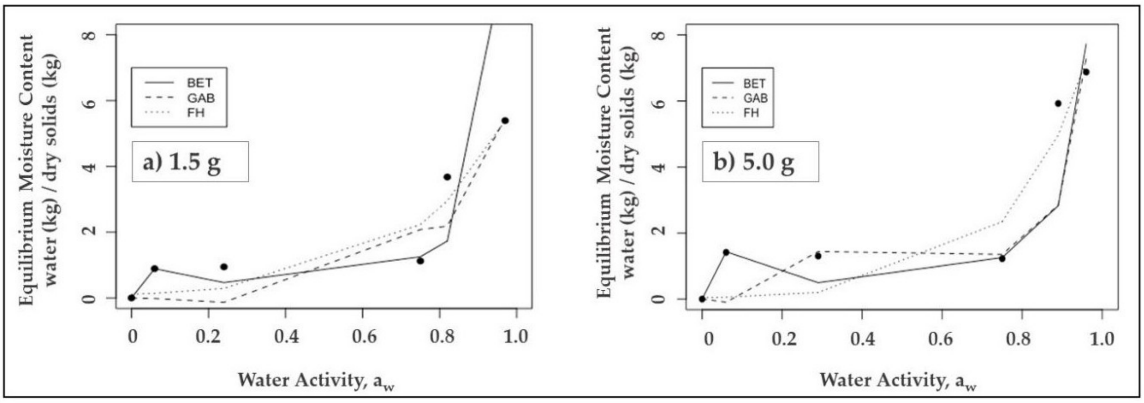

Figure 3. Experimental data (average and predicted values; Brunauer–Emmett–Teller (BET) (—), Guggenheim–Anderson–de Boer (GAB) (- - -), and Flory–Huggins (FH) (···) model predicted adsorption at 35 °C for (**a**) 1.5 g and (**b**) 5.0 g FS as initial mass.

Table 3. Predicted value analysis for selected models for 1.5 g and 5.0 g of fecal sludge (FS) as initial mass.

Model	Sample Mass	Parameters	Value	Standard Error	t-Value	Pr (>|t|)	RSE
BET	1.5 g	M_0	0.309	-	-	-	2.533 on 18 DoF
		C	−24.800	23.690	−1.047	0.308	
	5.0 g	M_0	0.309	-	-	-	2.837 on 22 DoF
		C	19.904	5.711	−3.485	0.002 **	
GAB	1.5 g	M_0	0.309	-	-	-	1.302 on 18 DoF
		C	−4.997	1.658	−3.013	0.00746 **	
		K′	0.973	0.007	142.140	$< 2 \times 10^{-16}$ ***	
	5.0 g	M_0	0.309	-	-	-	2.361 on 22 DoF
		C	−4.519	1.497	−3.017	0.00747 **	
		K′	0.985	0.006	157.107	$< 2 \times 10^{-16}$ ***	
FH	1.5 g	A	0.144	0.091	1.582	0.131	0.8884 on 18 DoF
		B	3.739	0.696	5.373	4.17×10^{-5} ***	
	5.0 g	A	0.403	0.234	1.725	0.0986	1.46 on 22 DoF
		B	2.942	0.639	4.602	000139 ***	

Signif. codes: 0 '***' 0.001 '**' 0.01 '*'; DoF: degrees of freedom.

4. Conclusions

The preliminary assessment of the moisture sorption isotherm of fecal sludge was investigated at 35 °C using a static gravimetric method. Overall moisture sorption isotherm trends indicated achievement of a sigma shape characterized by two characteristic bends and an increase of moisture adsorption at higher values of relative humidity. This means that, in this study, water in fresh faeces samples seems to be more easily extracted until a water activity of approximately 75%; more energy will be required to lower the water activity below this point. This sigma shape that fecal sludge isotherms seem to correspond to has also been reported for most food products with high moisture content (water activity—a_w over 0.9) [19]. This is as expected, since the composition of faeces will be like most food in terms of molecules present, namely carbohydrates, lipids, and protein. Furthermore, the effect of the mass of the sample and water activity on moisture content conforms to what was expected, namely lower moisture content with decreasing mass of the sample used, as can be seen when comparing Figure 2a,b. Experimental data seemed to follow the three-parameter Guggenheim–Anderson–de Boer model with a reasonable fit (p-Value < 0.05). However, further tests should be realized at lower relative humidity values to improve the calibration of the model.

This study validated the potential suitability of the gravimetric method for the characterization and modelling of the moisture sorption isotherms of fecal sludge. The results obtained represent one of the few datasets on dewatering properties for this type of matrix, and it is hoped that this method can be replicated to improve our understanding of fecal sludge desiccation properties. Future work using this approach includes determining the heat of sorption (or the solid–liquid bond strength); applying the approach to more samples from varied sources; analyzing the potential impact of chemical oxygen demand, pH, and total volatile solids on the equilibrium moisture content of fecal sludge to determine whether there are any universalities in the physical water distribution; comparing the physical water distribution of fresh faeces to that of fecal sludge; and investigating the impact of ventilation rates on drying rates. This could serve as a basis for the design of on-site sanitation systems for the in situ desiccation of fecal sludge intended for solids reduction and pathogen inactivation.

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