Gates Open Research 2020, 4:67 Last updated: 13 AUG 2020



RESEARCH ARTICLE

# Isothermal drying characteristics and kinetics of human faecal sludges [version 1; peer review: 1 approved with reservations]

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**v1** **First published:** 25 Jun 2020, **4**:67
https://doi.org/10.12688/gatesopenres.13137.1
**Latest published:** 25 Jun 2020, **4**:67
https://doi.org/10.12688/gatesopenres.13137.1

## Abstract

**Background:** Drying is an important step for the thermochemical conversion of solid fuels, but it is energy-intensive for treating highly moist materials.
**Methods:** To inform the thermal treatment of faecal sludge (FS), this study investigated the drying characteristics and kinetics of various faecal wastes using thermogravimetric analysis and isothermal heating conditions.
**Results:** The findings show that FS from anaerobic baffled reactor (ABR) and ventilated improved pit (VIP) latrines exhibit similar drying characteristics, with maximum drying rates at 0.04 mg/min during a constant rate period that is followed by a distinct falling rate period. On the contrary, fresh human faeces (HF) and FS from urine-diverting dry toilets (UDDT) exhibited a falling rate period regime with no prior or intermittent constant rate periods. The absence of constant rate period in these samples suggested limited amounts of unbound water that can be removed by dewatering and vice versa for VIP and ABR faecal sludges. The activation energies and effective moisture diffusivity for the sludges varied from 20 to 30 kJ/mol and 3□10$^{-7}$ to 1□10$^{-5}$ $m^2$/s at 55°C and sludge thickness of 3mm. The Page model was consistent in modelling the different sludges across all temperatures.
**Conclusions:** These results presented in this study can inform the design and development of innovative drying methods for FS treatment.

## Keywords

Thermogravimetric Study, Biomass Conversion, Sanitation Intervention, Kinetic Behaviour, Sludge Treatment

### Open Peer Review

**Reviewer Status** ?

| | Invited Reviewers |
|---|---|
| | **1** |
| **version 1** 25 Jun 2020 | ? report |

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Any reports and responses or comments on the article can be found at the end of the article.

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**Author roles: Somorin T**: Conceptualization, Formal Analysis, Investigation, Writing – Original Draft Preparation, Writing – Review & Editing; **Getahun S**: Conceptualization, Formal Analysis, Investigation, Methodology, Resources, Writing – Review & Editing; **Septien S**: Conceptualization, Funding Acquisition, Methodology, Resources, Supervision, Writing – Review & Editing; **Mabbet I**: Investigation, Supervision; **Kolios A**: Funding Acquisition, Resources, Supervision, Writing – Review & Editing; **Buckley C**: Funding Acquisition, Resources, Supervision

**Competing interests:** No competing interests were disclosed.

**Grant information:** This work was supported by the Bill and Melinda Gates Foundation [OPP1164143] as part of the research project "Characterization of faecal material behaviour during drying", a collaboration between Pollution Research Group, University of KwaZulu-Natal, Durban, Cranfield University, UK, and Swansea University, UK.
*The funders had no role in study design, data collection and analysis, decision to publish, or preparation of the manuscript.*



**How to cite this article:** Somorin T, Getahun S, Septien S *et al.* **Isothermal drying characteristics and kinetics of human faecal sludges [version 1; peer review: 1 approved with reservations]** Gates Open Research 2020, **4**:67 https://doi.org/10.12688/gatesopenres.13137.1

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## Introduction

More than one-third of the world's population are without access to modern sanitation, a situation that disproportionately affects low-income countries, particularly rural dwellers[1]. To eradicate poor sanitation, on-site sanitation facilities are being developed in many parts of the world. This includes the development of i) ecological toilets e.g. ventilated improved pit (VIP) latrines, composting toilets, and urine-diverting dry toilets to safely collect and convert human waste to an environmentally-friendly form (e.g. compost) and for recovery of useful products such as fuel and energy[2], and ii) advanced waste-to-energy technologies to convert human waste to fuel, heat and/or electricity, without putting undue pressure on natural resources[3–4]. For safe handling, transportation and environmental quality of faecal waste streams, processes such as dewatering, drying and pasteurisation are recommended[5].

Thermal drying is of specific interest because it can reduce waste volumes and improve the longevity and quality of end-products[6]. The integration of heat can eliminate pathogens and odour with potential health and environmental gains, but the scale of benefits in faecal sludge management will depend on material characteristics and process conditions[7–8]. To eliminate pathogens in waste streams, sufficiently high temperature and residence time need to be reached[9], which can come at a cost when large volumes of waste are treated. If the dryer is not appropriately designed, drying could reduce the energy quality of the feedstock. Material characteristics can change, causing properties such as stickiness, viscosity and shrinkage to initiate wear and tear of moving parts of internal combustion engines. Auto-ignition can occur, which might lead to an event of a fire[10]. Thus, an appropriate drying method is important for the safe removal of moisture. In this regard, thermogravimetric techniques can provide insights into the drying characteristics of faecal sludges and kinetic processes governing internal mass transfer.

Thermogravimetric analysis (TGA) enables material characterisation because it measures the change in mass of a material with respect to time or temperature, as the material is subjected to controlled temperature and heating changes[11–12]. Based on TGA techniques, drying rates are shown to vary from one sludge to another, depending on sample composition, origin, treatment method and retention time[13–14]. Here, intrinsic material properties such as porosity, density, water-solubility, thermal conductivity and viscosity affect drying rates, alongside with environmental factors (e.g. air velocity, temperature and humidity). The rate of loss of moisture differs in treated and untreated sludges and the volume of shrinkage, energy requirement and drying kinetics have changed with the type of additive used[15–16]. Studies by Zhang *et al.*[17] showed that municipal sewage sludge (MSS) has multiple drying profiles with characteristic apparent activation energies and effective moisture diffusivities. In the study conducted by Qian *et al.*[18], multiple falling rates were not reported but drying mechanisms follow a shrinkage core model with drying temperature and sample mass influencing drying kinetics. The multiple drying profiles observed by Reyes *et al.*[19] for MSS in a drying tunnel with parallel airflow at various temperatures and air velocities exhibited a relatively long constant rate period. Drying curves were modelled using Fick's second law equation and quasi-stationary methods. Among the nine different mathematical models employed by Zhang *et al.*[16], the Midilli model outperformed others, with respect to the prediction of the moisture content evolution during drying in the tested sludge, and in comparison, to experimental data. Thermogravimetric methods and kinetic models are, thus, proven tools for understanding and modelling the drying behaviour of sludge.

Several studies that have evaluated drying behaviour of sludge materials have focused on materials such as sewage sludge[20], pulp wastes[21] using different dryer configurations, contact methods (direct or indirect) and operating conditions to identify and optimise process efficiency[21,22] but limited information exists on FS, which differ in material composition[23]. A few studies that have attempted to understand FS drying have given focus to the development of physical processes[24–26]. To accommodate the unusual proprieties of FS and minimise capital and operating costs in off-grid systems, low-cost dewatering approaches such as drying beds, geobags, Imhoff tanks, membrane envelopes, were often cited. In this respect, Cofie *et al.*[24] described drying beds as an effective method for dewatering and removing helminth eggs from FS but results varied and depended on the quality of the filtering medium, degree of stabilisation, loading rates, bed height and on external conditions (e.g. rainfall and ambient temperature). Long residence times, which are in the order of 1 – 8 weeks, high land space requirement and the need to treat the resulting effluent (percolate) further limited their application. Seck *et al.* [25a] showed that drying rates can be improved in drying beds by mixing FS during loading but covering the bed (e.g. using greenhouse structure) provided no significant additional benefits apart from providing rain shield. Other treatment methods involving geobags and Imhoff tanks had reduced land requirements, but pathogen loads were only slightly reduced and post-treatment was needed to sanitise solid and effluent waste streams[2,27]. Thermal processes, e.g. "LaDePa" (Latrine Dehydration Pasteurization) infrared dryer and solar dryers, have been proposed and are being developed[28] but further information is needed to improve dryer performance, decrease area footprint and to reduce energy consumption.

This study presents the drying characteristics and kinetics of onsite sanitary wastes under controlled isothermal heating conditions. Temperatures between 55°C and 205°C were considered in this study to model low heating and pre-treatment conditions. FS from different sources were investigated to account for compositional changes. Kinetic parameters associated with drying were determined using mathematical models (Page, Newton, Logarithmic and Henderson). The results presented in this study can inform the design of innovative drying methods for FS treatment. The kinetic data can serve as reliable process model inputs for thermal drying treatment of FS.

## Methods

### Sample preparation & characterisation

On-site sanitary wastes were received from the Pollution Research Group, at the University of KwaZulu-Natal, South Africa. The sludges were collected from the following sources: a) anaerobic baffled reactor (ABR) at a decentralized wastewater treatment system (DEWATS); b) VIP latrines and c) urine-diverting dry toilet (UDDT). The DEWATS is a mixture of black water, greywater and human faecal sludges and it receives effluent from neighbouring households and communal ablution blocks in Fraser's informal settlement, Durban. Due to the limited size of the inlet of the DEWATS, samples were collected during pit emptying and using a vacuum truck. For representative sampling of the DEWATS, samples were collected from three zones (top, middle and bottom) of the settling tank (first compartment). The VIP samples were collected from a pit in Bester informal settlement, located 25 km north of Durban. Due to inaccessibility of the pit, FS was collected directly from a vacuum truck during pit emptying; however, this process involves water dilution for suction. The FS sample from the UDDT was collected from Kwamashu, 20 km north of Durban, a facility that serves a single household. FS samples from the UDDT were collected manually using spades and forks, with large household waste (clothing, sanitary material, paper, etc.) found in the sludge removed onsite. All samples were screened for materials larger than 5mm (using a 5mm sieve) and then stored at 4°C at the laboratory of the Pollution Research Group.

The collection and analysis of FS for this investigation were approved by the Biomedical Research Ethical Committee from the University of KwaZulu-Natal (Ethical Clearance Reference: EXM005/18). The samples were couriered to Cranfield University in sealed plastic bottles to avoid moisture loss, wrapped in zip lock bags and contained in a box with ice blocks between the secondary and outer packaging for continuous preservation of samples at 4°C. This was under the authorization of the Health Department of the Republic of South Africa (export permit: J1/2/4/2). At Cranfield University, fresh human faeces (HF) was collected from a volunteer as part of the Nano Membrane Toilet Sampling Collection Campaign. This sample collection process involves: a) campaign for voluntary donation of human faeces at Cranfield University from staff and students, b) preparation and provision of sample collection kits (including cardboard bowl, pair of gloves, black plastic bags (for collection and disposal of gloves), zip ties as well as information and instruction sheet, volunteer consent sheet, pen and labelling paper in designated sampling toilets, c) anonymous donation of sample in a cardboard collection bowl contained in a black plastic bag with zip ties and held in a plastic box, d) collection of samples from designated sampling toilets, e) sample storage at -85°C in a designated freezer. This sampling protocol is approved by the Cranfield Research Ethics Committee Approval (CURES/2310/2017) and consent is agreed in written form by completing a volunteer consent form. Due to the focus on drying, the HF sample was stored at 4°C and brought to room temperature before analysis. To determine the initial moisture content of the samples, 5g of the samples was weighed and dried at 105 ± 5°C in a hot air oven. Table 1 highlights the initial moisture content of the samples as received from the University of KwaZulu-Natal and Cranfield University.

### Thermogravimetric analysis

Under isothermal drying conditions, 40 mg of sample was thinly spread in a cylindrical aluminium crucible to a diameter of ~3 mm and weighed to an accuracy of ± 0.5 mg. Samples were subjected to controlled temperatures of 55, 85, 105, 155, 205°C in a thermogravimetric analyser (Model: *PerkinElmer TGA 8000*™). All experiments were carried out using an airflow rate of 40 mL/min. Prior to isothermal temperature, samples were raised from 30°C to the specified temperature at a rapid heating rate of 100°C/min to minimise drying during the heating-up stage. For repeatability, each test was conducted at least in duplicates.

### Kinetic analysis: isothermal drying

The moisture ratio (MR), which shows the extent to which drying has taken place in the samples at a given time, was determined using Equation 1.

$$MR = {}^{m - m_e}\!\big/_{m_o - m_e} \qquad \text{Eqn. (1)}$$

where $m_o$, $m$ and $m_e$ are initial moisture, the moisture of the sample at a time (t) and final moisture of the sample, respectively. The $m_e$ represents the point at which the sample weight is constant with time after drying has stopped after reaching the thermodynamic equilibrium. In this study, the sample was considered to be completely dried at the end of the experiment, so $m_e$ was equal to 0. The drying curves were fitted with widely used drying models – see Table 2 using MATLAB R2019a Curve Fitting Toolbox (open source alternatives such as GNU Octave could also be used for this purpose).

**Table 1. Initial moisture content of samples (as received basis).**

| | ABR | HF | UDDT | VIP |
|---|---|---|---|---|
| Initial moisture (wt. %) | 83 | 62 | 48 | 94 |

ABR, anaerobic baffled reactor; HF, human faeces; UDDT, urine-diverting dry toilet; VIP, ventilated improved pit.

**Table 2. Models for isothermal drying.**

| Drying model | Isothermal models |
|---|---|
| Page | $MR = exp(-kt^n)$ |
| Newton | $MR = exp(-kt)$ |
| Logarithmic | $MR = a + b\ exp(-kt)$ |
| Henderson | $MR = a\ exp(-kt)$ |

The effective moisture diffusivity of the samples was determined at different temperatures, using Fick's second law of diffusion in Equation 2–Equation 6. These equations and derivatives are well-known for describing the internal mass transfer mechanisms, especially during the falling rate period. The equations assume that there is a uniform distribution of moisture, negligible external resistance and shrinkage, and constant diffusivity of moisture across each sample. Note that the effective moisture diffusivity is a value that lumps the contribution of several internal mass transfer phenomena, such as gas and liquid molecular diffusivity, capillary movements, flow due to gradients of pressure, among other phenomena.

$$\frac{\partial MR}{\partial t} = \nabla\left[D_{eff}\left(\nabla MR\right)\right] \qquad \text{Eqn. (2)}$$

$$MR = \frac{8}{\Pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}exp\left[-\frac{(2n+1)\Pi^2 D_{eff} t}{4L^2}\right] \qquad \text{Eqn. (3)}$$

where $D_{eff}$, $L$ and $t$ are effective moisture diffusivity ($m^2/s$), the thickness of sample in the crucible (m) and drying time (s), respectively. Equation 2 can be simplified to a straight-line through mathematical manipulations, as displayed in Equation 3. As such, a plot of *ln MR* against *t*, as shown in Equation 4, provides the slope of the line, $k_0$ (Equation 5), from which the $D_{eff}$ was derived.

$$ln(MR) = ln\left[\frac{8}{\Pi^2}\right] - \left[\frac{\Pi^2 D_{eff}}{4L^2}t\right] \qquad \text{Eqn. (4)}$$

$$k_0 = \frac{\Pi^2 D_{eff}}{4L^2} \qquad \text{Eqn. (5)}$$

Considering the Arrhenius equation in Equation 6 that describes the temperature dependence of $D_{eff}$, the activation energy could be obtained from the plot of $ln\,(D_{eff})$ against $1/T(K)$.

$$D_{eff} = D_0 exp\left[\frac{E_a}{RT}\right] \qquad \text{Eqn. (6)}$$

where $E_a$ is activation energy of the drying process (kJ/mol), $D_o$ is a pre-exponential factor ($m^2/s$), $T$ is drying temperature (K) and $R$ is universal gas constant (J/mol K).

### Statistical analysis of drying models

The drying models in Table 2 was fitted to the experimental data obtained for the different conditions. Statistical methods such as chi-square ($X^2$) were computed using Equation 7 to determine the goodness of fit of the predicted values in comparison to the experimental data.

$$X^2 = \frac{\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2}{N-n} \qquad \text{Eqn. (7)}$$

where $MR_{pre,i}$ are the predicted moisture ratios, $MR_{exp,i}$ are the experimental moisture ratios, $N$ is the number of observations and $n$ is the number of drying constants.

## Results and discussion

### Isothermal drying behaviour

The drying profiles of the ABR, HF, UDDT and VIP at a drying temperature of 55°C are shown in Figure 1 by means of a) MR vs time and b) drying rate vs moisture content (wet basis).

The results in Figure 1a show that MR decreased with time for all the sample types but the drying profiles for some of the samples differed from one another (Figure 1b). For example, the drying rates for the ABR and VIP samples were relatively constant at ~0.04 mg/min until moisture levels of about 20 wt.% when drying rates started to decrease. However, the drying curves for the HF and UDDT exhibited a falling rate regime with no prior or intermittent constant rate period, with the rate of moisture loss differing across stages. While the ABR sample had a single falling rate period, the HF had multiple falling rates[29]. These different drying profiles suggest a different physical state of water inside the sludge and internal moisture transport processes.

Typically in sludges, moisture is said to be present as free, interstitial, surface and/or intracellular water[30]. These moisture forms exhibit different drying profiles and are affected by the type and strength of chemical bonds in water molecules. According to Erdincler *et al.*[31], only the free water and a part of the interstitial water can be removed by conventional dewatering

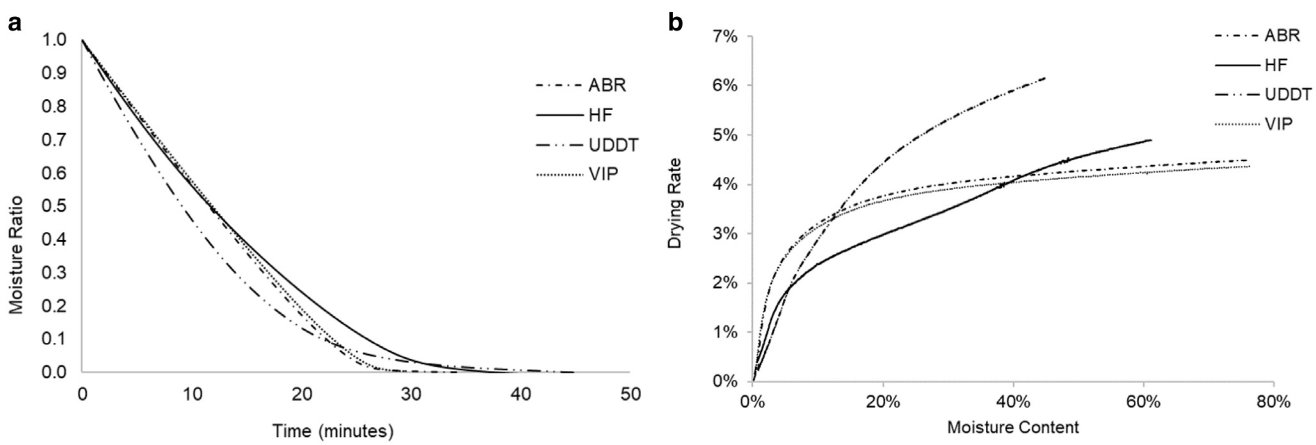


**Figure 1. The isothermal drying curves of ABR, HF, UDDT and VIP at 55°C. a**) moisture ratio vs time, **b**) moisture ratio derivative (drying rate) vs moisture content. ABR, anaerobic baffled reactor; HF, human faeces; UDDT, urine-diverting dry toilet; VIP, ventilated improved pit.

processes, the rest require drying. Free water is considered to have no or loose bonds with particles[30,31]; hence, moves freely and can be separated relatively easy by mechanical separation methods[32]. The interstitial water is said to be bound by active capillary forces within the sludge particles, particularly by microbial flocs that aggregate and form complex links. These can be separated relatively easy by agitation or other mechanical methods such as centrifugation[10]. The surface water is bound by adhesive forces to particles, with no free movement and includes water that is bound within exopolysaccharides of microbial cells. According to Rose *et al.*[33], about 50 wt.% of the moisture in HF are bound in bacterial cells and in complex biofilm matrix – mainly exopolysaccharides. The intracellular water is deemed as bound water, along with surface water, as such, not readily removed by simple solid separation techniques[34]. These drying concepts suggest that the process by which moisture is being transported and removed from the FS samples are different, although the exact mechanisms for these internal transport processes are not known.

One important material property that is vital for understanding the drying profiles of materials is the critical moisture content ($X_C$), which can serve as an indicator of the amount of unbound and bound moisture in the sludge at a low drying temperature. This property indicates the magnitude of the heat and mass transfer resistances in solids[35] and changes with material property (e.g. thickness) and factors that affect drying rate (e.g. temperature). In practice, drying will occur at a constant rate if the moisture content is above the critical moisture content and the moisture removed during this period will be considered mainly as unbound. In contrast, below the $X_C$, the drying rate will exhibit a falling rate regime and the remaining moisture to remove from the sludge will be considered mostly as bound. Indeed, the $X_C$ is an indicator of the amount of unbound and bound moisture in the material. In this study, the $X_C$ for ABR and VIP samples was about 20 wt.%, whereas no $Xc$ was observed for the UDDT. For the HF, there was also no prior or intermittent constant rate period, but the multiple falling rates indicate a lower critical moisture content at about 34 wt.%. The high moisture content in VIP and ABR samples and the constant rate period exhibited during drying up to 20 wt.% suggest that unbound or weakly bounded moisture are largely present and part of it can be removed by dewatering. In the case of HF and UDDT, only thermal drying can be applied to remove moisture, because of the likely limited amounts of unbound moisture in the samples. These aspects have implications for the design, development and optimisation of treatment systems. Further work is needed to ascertain the mechanisms by which moisture is held freely in open pores, trapped, locked or bound to organic materials and removed from solids. This can be achieved using advanced imaging techniques and computational methods. Note that at 55ºC, drying occurred in a similar way between the VIP and ABR samples, whereas the drying curves were slightly different for the UDT and HF samples. The UDT sample exhibited the fastest drying, whereas the HF samples the lowest drying (Figure 1).

## Influence of drying temperature

The faecal sludges (ABR, HF, UDDT and VIP) were subjected to low-temperature isothermal drying conditions (temperatures: 55 to 205°C). Results are illustrated in Figure 2 – Figure 5 as a plot of a) MR versus time and b) drying rate versus moisture content.

The plot of MR against time shows that drying times reduced significantly with an increase in drying temperature (Figure 2a–Figure 5a). For complete removal of moisture in all the samples, drying times reduced by more than 90% at 205°C, between 64 and 77% at 105°C and up to 62% at 85°C, with respect to drying at 55°C. The drying rates differed across samples, although drying rates increased with increasing temperature. For ABR and VIP samples at a drying temperature of 105°C (Figure 2b and Figure 5b), the drying rates were relatively constant at 0.18 mg/min until a moisture content of ~20 wt.% (critical moisture content), after which the drying rate started to decrease. A similar pattern was followed at 155°C, but with a higher maximum drying rate (~0.42 mg/min) and shorter constant rate period. At 205°C, drying presented a short constant rate period, and most of the transformation occurred in a falling rate period. Indeed, it can be noted that the constant rate period was shortened as temperature increased. This can be attributed to the effect of temperature on the transport

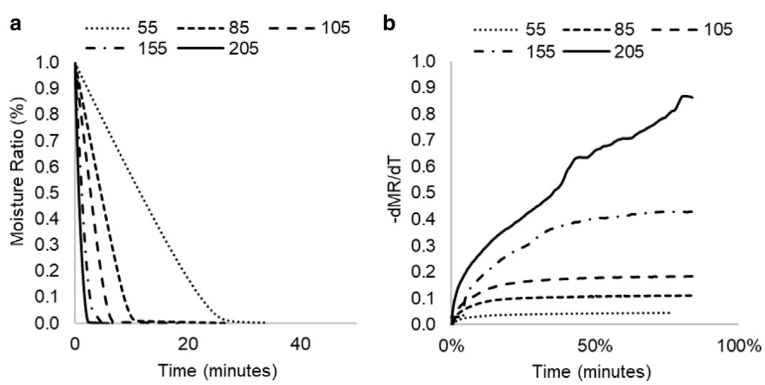


**Figure 2. Isothermal drying curves of ABR at 55 – 205°C. a**) MR versus time and **b**) drying rate versus moisture content. ABR, anaerobic baffled reactor.

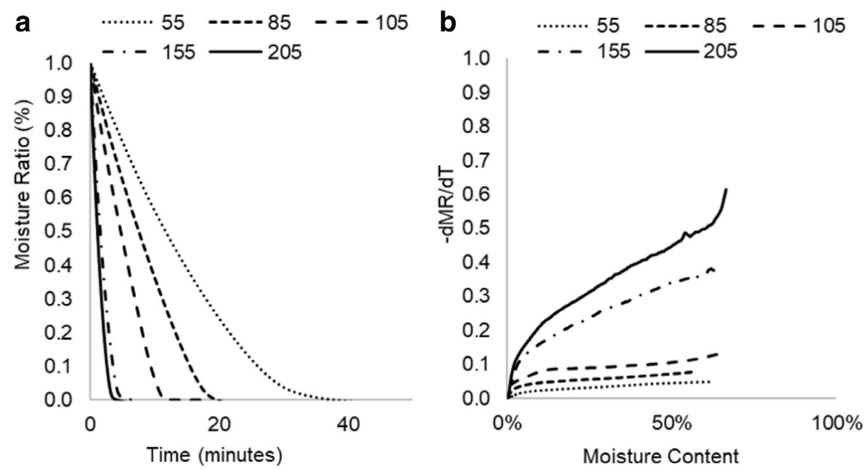


**Figure 3. Isothermal drying curves of HF at 55 – 205°C. a**) MR versus time and **b**) drying rate versus moisture content. HF, human faeces.

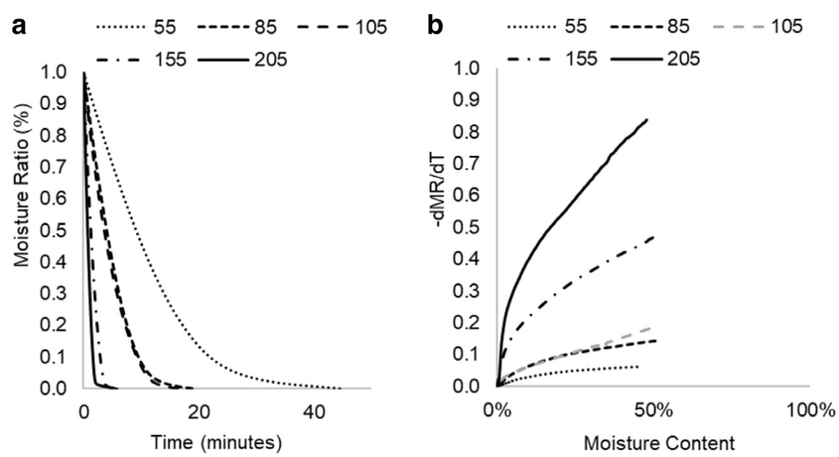


**Figure 4. Isothermal drying curves of UDDT at 55 – 205°C. a**) MR versus time, **b**) drying rate versus moisture content. UDDT, urine-diverting dry toilet.

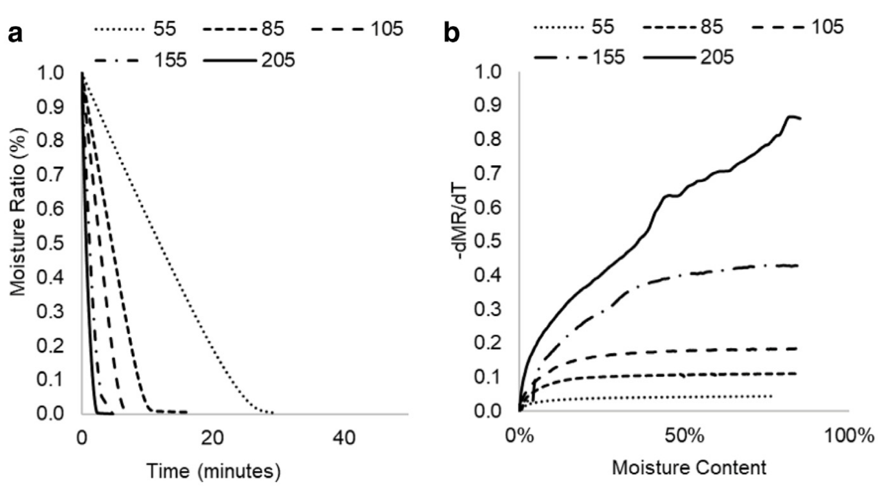


**Figure 5. Isothermal drying curves of VIP at 55 – 205°C. a**) MR versus time, **b**) drying rate versus moisture content. VIP, ventilated improved pit.

mechanisms. At lower temperature, there is gradual and effective heat transfer into the internal part of the solids, which favours evaporated moisture at the surface of the sludge, maintaining the constant rate period during which the surface of the sludge is completely saturated in moisture. However, at high temperature, transport mechanisms are overtaken by thermal events. Here, the evaporation of the moisture at the sludge surface occurs faster than its replacement with moisture from the core, leading to the decline of the drying rate. In the case of HF and UDDT samples (Figure 3b and Figure 4b), drying rates also increased and drying times reduced with increasing temperature, but no constant rate period was observed, as commented in the previous section. For all the samples, the falling rate period was the time-consuming step. These results give valuable information for the design and operation of drying systems.

### Kinetic analysis

The plots of moisture ratio versus time were fitted into Page, Newton, Logarithmic and Henderson models as these models best describe mathematically the mechanisms in the drying process. The mathematical models were assessed using statistical methods e.g. root mean square error (RMSE), $X^2$, $R^2$ and sum of squared estimate of errors (SSE) for the measure of fitness of the predicted values to the experimental data. The results, which are summarised in Table 3–Table 6, show that

**Table 3. Statistical data for isothermal drying of anaerobic baffled reactor (ABR).**

| Model name | Temp. (°C) | ABR | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | a | b | k | n | SSE | $R^2$ | RMSE | COV |
| Henderson | 55 | 1.15 | - | 0.09 | - | 9.97 | 0.95 | 0.07 | 2 |
| | 85 | 1.16 | - | 0.22 | - | 4.45 | 0.97 | 0.05 | 2 |
| | 105 | 1.07 | - | 0.23 | - | 2.58 | 0.97 | 0.05 | 2 |
| Logarithmic | 55 | -0.39 | 1.45 | 0.04 | - | 2.12 | 0.99 | 0.03 | 3 |
| | 85 | -0.04 | 1.18 | 0.20 | - | 3.63 | 0.97 | 0.05 | 3 |
| | 105 | -0.03 | 1.17 | 0.34 | - | 2.17 | 0.97 | 0.04 | 3 |
| Newton | 55 | - | - | 0.08 | - | 14.20 | 0.93 | 0.08 | 1 |
| | 85 | - | - | 0.20 | - | 6.52 | 0.95 | 0.06 | 1 |
| | 105 | - | - | 0.33 | - | 3.80 | 0.96 | 0.06 | 1 |
| Page | 55 | - | - | 0.02 | 1.60 | 2.04 | 0.99 | 0.03 | 2 |
| | 85 | - | - | 0.06 | 1.62 | 0.80 | 0.99 | 0.02 | 2 |
| | 105 | - | - | 0.15 | 1.60 | 0.44 | 0.99 | 0.02 | 2 |

SSE, sum of squared estimate of errors; RMSE, root mean square error; COV, coefficient of variation.

**Table 4. Statistical data for isothermal drying of human faeces (HF).**

| Model name | Temp. (°C) | HF | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | a | b | k | n | SSE | $R^2$ | RMSE | COV |
| Henderson | 55 | 1.10 | - | 0.08 | - | 4.58 | 0.97 | 0.05 | 2 |
| | 85 | 1.12 | - | 0.13 | - | 5.64 | 0.96 | 0.06 | 2 |
| | 105 | 1.14 | - | 0.21 | - | 4.25 | 0.96 | 0.06 | 2 |
| Logarithmic | 55 | -0.38 | 1.39 | 0.04 | - | 0.18 | 1.00 | 0.01 | 3 |
| | 85 | -0.40 | 1.43 | 0.06 | - | 0.64 | 1.00 | 0.02 | 3 |
| | 105 | -0.17 | 1.24 | 0.14 | - | 1.73 | 0.98 | 0.04 | 3 |
| Newton | 55 | - | - | 0.07 | - | 6.49 | 0.96 | 0.06 | 1 |
| | 85 | - | - | 0.11 | - | 7.65 | 0.94 | 0.07 | 1 |
| | 105 | - | - | 0.19 | - | 5.77 | 0.94 | 0.07 | 1 |
| Page | 55 | - | - | 0.03 | 1.35 | 1.23 | 0.99 | 0.02 | 2 |
| | 85 | - | - | 0.04 | 1.49 | 1.59 | 0.99 | 0.03 | 2 |
| | 105 | - | - | 0.07 | 1.53 | 1.10 | 0.99 | 0.03 | 2 |

SSE, sum of squared estimate of errors; RMSE, root mean square error; COV, coefficient of variation.

**Table 5. Statistical data for isothermal drying of urine-diverting dry toilet (UDDT).**

| Model name | Temp. (°C) | UDDT | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | a | b | k | n | SSE | $R^2$ | RMSE | COV |
| Henderson | 55 | 1.10 | - | 0.10 | - | 2.39 | 0.99 | 0.03 | 2 |
| | 85 | 1.10 | - | 0.22 | - | 1.28 | 0.99 | 0.03 | 2 |
| | 105 | 1.07 | - | 0.23 | - | 0.92 | 0.99 | 0.03 | 2 |
| Logarithmic | 55 | -0.05 | 1.12 | 0.08 | - | 1.18 | 0.99 | 0.02 | 3 |
| | 85 | -0.06 | 1.12 | 0.19 | - | 0.59 | 0.99 | 0.02 | 3 |
| | 105 | -0.07 | 1.10 | 0.19 | - | 0.23 | 1.00 | 0.02 | 3 |
| Newton | 55 | - | - | 0.09 | - | 4.01 | 0.98 | 0.04 | 1 |
| | 85 | - | - | 0.20 | - | 2.03 | 0.98 | 0.04 | 1 |
| | 105 | - | - | 0.22 | - | 1.26 | 0.98 | 0.03 | 1 |
| Page | 55 | - | - | 0.04 | 1.29 | 0.22 | 1.00 | 0.01 | 2 |
| | 85 | - | - | 0.12 | 1.31 | 0.17 | 1.00 | 0.01 | 2 |
| | 105 | - | - | 0.15 | 1.22 | 0.27 | 1.00 | 0.02 | 2 |

SSE, sum of squared estimate of errors; RMSE, root mean square error; COV, coefficient of variation.

**Table 6. Statistical data for isothermal drying of ventilated improved pit (VIP).**

| Model name | Temp. (°C) | VIP | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | a | b | k | n | SSE | $R^2$ | RMSE | COV |
| Henderson | 55 | 1.13 | - | 0.08 | - | 8.46 | 0.95 | 0.07 | 2 |
| | 85 | 1.16 | - | 0.22 | - | 4.38 | 0.96 | 0.07 | 2 |
| | 105 | 1.13 | - | 0.34 | - | 1.76 | 0.95 | 0.07 | 2 |
| Logarithmic | 55 | -1.00 | 2.02 | 0.03 | - | 0.45 | 1.00 | 0.02 | 3 |
| | 85 | -0.16 | 1.25 | 0.16 | - | 2.12 | 0.98 | 0.05 | 3 |
| | 105 | -1.09 | 2.11 | 0.10 | - | 0.08 | 1.00 | 0.01 | 3 |
| Newton | 55 | - | - | 0.07 | - | 11.92 | 0.93 | 0.08 | 1 |
| | 85 | - | - | 0.20 | - | 6.36 | 0.94 | 0.08 | 1 |
| | 105 | - | - | 0.30 | - | 2.52 | 0.93 | 0.08 | 1 |
| Page | 55 | - | - | 0.02 | 1.57 | 1.95 | 0.99 | 0.03 | 2 |
| | 85 | - | - | 0.06 | 1.62 | 0.80 | 0.99 | 0.03 | 2 |
| | 105 | - | - | 0.15 | 1.56 | 0.36 | 0.99 | 0.03 | 2 |

SSE, sum of squared estimate of errors; RMSE, root mean square error; COV, coefficient of variation.

the Page model best describes the drying profiles of the various sludges, particularly for ABR and UDDT samples. The data points from the Page model had the highest values of $R^2$, which exceeded 0.99 in most cases, as well as the lowest values of SSE and RMSE. The experimentally determined and predicted data points are illustrated in Figure 6. For the HF and VIP samples, the logarithmic model best described the drying profile of HF at 55°C and 85°C and at 55°C and 105°C for VIP samples. Here, the logarithmic model had values of SSE and RMSE in between 0.9922 and 0.9990, and 0.01 and 0.03, respectively.

Table 7 displays the effective moisture diffusivities calculated from the experimental data. The $D_{eff}$ for the sludges increased by increasing temperature and reduced with initial moisture content. The values varied between $3.4 \cdot 10^{-7}$ and $7.6 \cdot 10^{6}$ m²/s at 55°C and $3.2 \cdot 10^{6}$ and $7.0 \cdot 10^{6}$ m²/s at 205°C, for a sludge thickness of 3 mm. *Ea* values were 28 kJ/mol (ABR), 30

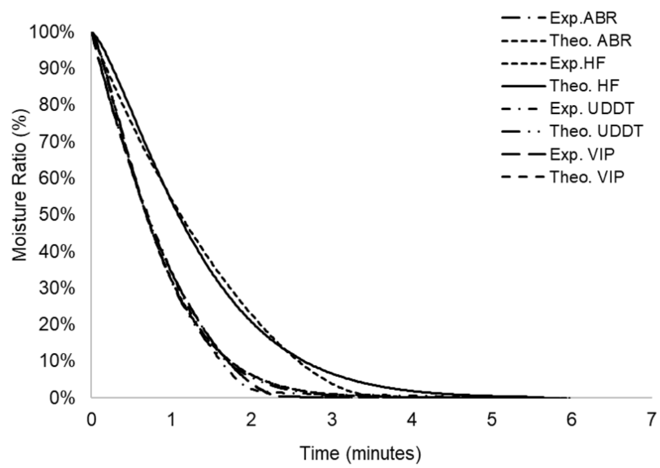


**Figure 6. Predicted MR (using Page model) in comparison to experimentally determined values for different faecal sludges (ABR, HF, UDDT and VIP).** ABR, anaerobic baffled reactor; HF, human faeces; UDDT, urine-diverting dry toilet; VIP, ventilated improved pit.

**Table 7. Isothermal drying kinetic parameters of faecal sludges.**

| Temperature (°C) | Effective moisture diffusivity ($m^2/s$) | | | |
|---|---|---|---|---|
| | ABR | HF | UDDT | VIP |
| 55 | $3.4 \cdot 10^{-7}$ | $3.8 \cdot 10^{-7}$ | $5.1 \cdot 10^{-7}$ | $3.2 \cdot 10^{-7}$ |
| 85 | $7.9 \cdot 10^{-7}$ | $5.5 \cdot 10^{-7}$ | $1.3 \cdot 10^{-6}$ | $7.9 \cdot 10^{-7}$ |
| 105 | $1.3 \cdot 10^{-6}$ | $1.1 \cdot 10^{-6}$ | $1.5 \cdot 10^{-6}$ | $1.2 \cdot 10^{-6}$ |
| 155 | $4.2 \cdot 10^{-6}$ | $4.6 \cdot 10^{-6}$ | $5.0 \cdot 10^{-6}$ | $3.9 \cdot 10^{-6}$ |
| 205 | $7.6 \cdot 10^{-6}$ | $9.9 \cdot 10^{-6}$ | $4.8 \cdot 10^{-6}$ | $7.0 \cdot 10^{-6}$ |
| Activation energy (kJ/mol) | 28 | 30 | 21 | 27 |

ABR, anaerobic baffled reactor; HF, human faeces; UDDT, urine-diverting dry toilet; VIP, ventilated improved pit.

kJ/mol (HF), 21 kJ/mol (UDDT) and 27 kJ/mol (VIP). The relative low values of the activation energy demonstrate that drying is a process kinetically controlled by physical phenomena, namely heat and mass transfer. A considerably higher activation energy would be expected for a process controlled by chemical phenomena. The effective moisture diffusivities for the samples in this study were higher compared to those from sewage sludge drying[36], which were are in the order of $3.9 \cdot 10^{-10}$ to $4.4 \cdot 10^{-10}$ $m^2/s$ at a thickness of 2–8 mm.

## Conclusions

Drying characteristics and kinetics of various faecal sludges were examined using thermogravimetric analysis under isothermal conditions. The results from this investigation suggested a high level of boundedness of the moisture in the FS samples, particularly for the HF and UDDT sludge, which would lead to a high energy consumption for drying. The absence of constant rate period in this sample and UDDT suggests that the HF and UDDT sludges may contain limited unbound water that could be removed by dewatering. On the other hand, mechanical dewatering could be applied for the VIP and ABR FS to reduce the energy consumption for moisture removal. Drying could be improved by increasing the drying temperature, as this should favour the removal of bound moisture and enhance the mass transfer phenomena, leading to a fast process and a reduction in the drying time. The different drying behaviours of the samples suggest that the internal moisture transport phenomena occur differently as a function of the type of sludge. For all samples, the values of the activation energy ranged from 20–30 kJ/mol, which reflects a process controlled by transfer phenomena, and the effective diffusivity was in the order $10^{-7}$–$10^{-8}$ $m^2/s$, which was higher than values from sewage sludge drying. The Page model described the drying kinetics with the best fit, so this type of model could be used for the design and operation of faecal sludge drying systems.

## Data availability

### Underlying data

Figshare: DATA-DRYING-ISOTHERM-FAECAL-SLUDGES.xlsx. https://doi.org/10.6084/m9.figshare.12349622.v1[29].



## Disclaimer

The work was completed at Cranfield University and findings and conclusions contained within are those of the authors and do not necessarily reflect positions or policies of the Bill and Melinda Gates Foundation.

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# Open Peer Review

## Current Peer Review Status: ?

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Reviewer Report 13 August 2020

https://doi.org/10.21956/gatesopenres.14325.r28987



?

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**General comments:**
In this paper, samples of faecal sludge/human faeces were obtained from four sources: anaerobic baffled reactor, VIP latrines, an ablution block, and direct donations from volunteers. The drying kinetics of test masses from collected FS samples at varying temperatures were obtained using TGA, and the results were analyzed by fitting to four mathematical expressions. Then a mechanistic model based on Fickian diffusion was used to extract values for effective diffusion coefficients for each faecal sludge sample and at each temperature, and a value for activation energy was then obtained.
The topic of this manuscript is important. Motivation, methods, results and analysis are, for the most part, clearly presented and easy to understand. The authors are to be commended for their experimental work as well as the effort to extract mechanistic information from the data. I have a number of significant comments and questions that I have attempted to organize into logical categories below. I hope my comments will contribute to an improved paper and make this important work more useful to the community. Note that my comments are based on the paper, I did not examine the primary data.

**A Transferability of results and conclusions to drying operations:**
As the authors point out, drying is important in faecal sludge transport and processing and was the motivation for the study. It would help the reader relate the results from this study to potential drying operations if the authors can clarify the following:

1. How many latrines/toilets/ablution blocks were the faecal sludge samples collected from? For example, are the reported VIP latrine data from samples collected a single time from a single latrine? Or multiple times from the same latrine? Similarly, are the human faeces samples from a single donor, or was it a sample from agglomerated donations from multiple volunteers? Whatever the answers are, can the authors comment on how representative their samples are of each broad category of faecal sludge?

2. Were the collected samples homogeneous? If not, were they homogenized before the 40

mg TGA test masses were taken? If so, how was this done?

3. How do the sample preparation methods between the time of collection and TGA kinetics analysis compare to how faecal sludge would be prepared in drying operations? Methods of preparation can affect the nature of the matrix or medium through which water diffuses.

4. Can the authors compare typical faecal sludge thickness in drying operations vs TGA sample thickness? Thickness is an important parameter in drying kinetics.

**B TGA analysis and results:**

1. Air flow rate of 40 mL/min was used for TGA drying experiments. Was the convective mass transfer resistance under these conditions negligible compared to internal diffusive resistance?

2. What was the relative humidity in the atmosphere for the drying experiments? This is useful to know because it gives the sink condition for drying.

3. The equilibrium moisture content was assumed to be zero in equation 1. The equilibrium moisture content should be a function of the temperature and relative humidity, but at low RH can be a small enough number that it can be assumed to be zero. A comparison of the TGA drying data to the measured initial moisture content should be presented to indicate whether this is a good assumption.

4. Each test was conducted at least in duplicate. Does this mean at least two 40 mg test masses were removed from different parts of each faecal sludge sample and run at the specified temperature? Or that there were multiple collected faecal sludge samples? (Same question as A1.) How much variation was there in the results among replicate masses? Can standard deviations be reported on extracted quantities such as diffusion coefficient and activation energy?

5. Table 1. The moisture content for HF and UDDT are significantly lower than in healthy faeces. Presumably, the UDDT samples dry in the pit before collection. What is the explanation for the low moisture content (62%) of human faeces collected without further storage or processing?

6. Moisture in the manuscript is reported in units of %, but not defined. Is it mass of water divided by total mass of faecal sludge, ie the mass basis concentration of water in the faecal sludge matrix?

7. The x-axis in Figure 2b should be labelled as moisture content, not time.

8. 8. In Figure 1b, the drying rate axis is labelled as %. In Figures 2b, 3b , 4b and 5b. the drying rate axes are labelled as -dMR/dT, with numbers between 0 and 1 on the axis but no units.
(a) I believe the y-axis labels in Figures 2b to 5b are drying rates with units of mg/min. Please confirm and label with the proper units.
(b) What does the % in the y-axis in Figure 1b represent? And why is it presented differently than Figures 2b, 3b, 4b and 5b? A time component is needed for drying rate.
(c) To be consistent with the use of t for time, and T for temperature in other parts of the

manuscript (eg equations 2 to 6), the drying rate symbol on the y-axis should be dMR/dt.

**C Models and data analysis**
The following comments relate to data analysis using the four fitting models and the Fickian diffusion model:

1. Small inconsistency: equation 7 gives the definition of $X^2$ for goodness of fit, but statistical analysis shown in Tables 3 to 6 used other statistical quantities SSE, R2, and RMSE to conclude that the Page model gave the best fit for most conditions. The short section on statistical analysis of drying models needs to be edited to reflect what was actually done.

2. The coefficient of variation numbers in Tables 3 to 5 are mistakenly listed. The numbers listed for COV are 1, 2 or 3, corresponding to the number of fitting parameters in each of the models.

3. My analysis of the four models and the fitting results - as outlined below - raises questions about the way the statistical analysis was done that the authors should address. Even though the models are only loosely mechanistically based, the parameters obtained from fitting should still make sense, but the results of the logarithmic model and the Page model are inconsistent with inferred mechanisms.

   (a) The Newton model MR(t) = exp (-kt) is the same as the Henderson model MR(t) =a exp (-kt) but with the pre-exponential factor forced to take on a value of one. Since MR(t =0) = 1, it makes some sense to choose the Newton model. The fit of the Newton model is not quite as good as the Henderson Model, but that is to be expected since there is only one fitting parameter. Also, note that the fitted values for "a" in the Henderson model are all close to 1 - ranging from 1:07 to 1:016; so the two models give very similar results.
   The form of these two models is consistent with the simplified Fickian diffusion model (equation 4) which gives a pre-exponential factor of $8/\pi^2$ = 0:81 rather than 1 due to the truncation of all but the first term of the infinite series. The good fit of the Henderson and Newton models suggest that Fickian diffusion is a key determinant of drying kinetics.

   (b) The logarithmic model MR(t) = a + b exp (-kt) is similar to the Henderson model but with a component of water that cannot be removed: the parameter "a" in the logarithmic model would represent an irreversibly bound amount of water, while the drying of the removable fraction is consistent with Fickian diffusion. Given this physical interpretation, the negative values of "a" obtained from fitting, ranging from -0:04 to -1:09, is perplexing and suggests that either something has been reported in error or that the statistical analysis was done incorrectly. Clarification is required.

   (c) The Page model MR(t) = exp ($-kt^n$) is unique among the four fitting models due to the $t^n$ term. The Fickian diffusion model assumes that $D_{eff}$ is constant throughout the drying process, but the reality is that the matrix through which water must be transported changes as the sludge dries, and Deff should decrease with time. Having a $t^n$ terms provides a way to account for such time dependent changes in the intrinsic nature of the medium, and makes the Page model attractive. However, since we expect $D_{eff}$ to decrease with time, the exponent n should be less than 1. So it is perplexing that the best fit values of n shown in Tables 3 to 6 are all greater than 1 - ranging from 1:29 to 1:60. Some clarification or re-checking of the statistical analysis should be done.

(d) Given the questions raised above about the logarithmic model and the Page model, the conclusion that the Page model gives the best fit should be reviewed.

(e) In addition, given the relatively small number of samples, are the relatively small differences in goodness of fit of the models statistically significant?

4. The authors use the simplified result of the Fickian model to extract Deff from the data, and then to compute an activation energy. There is some internal inconsistency since they had concluded the Page model to be the best fit. Nevertheless, the fitting results of the Henderson/Newton models make sense and can justify interpretation using the Fickian model.
   A big assumption in Fick's model is that the effective diffusion coefficient is a constant throughout the drying process. This assumption fails as the water concentration changes over a wide range of values - eg MR ranging from 1:0 down to 0 means the water concentration changes from 92% to zero for faecal sludge from VIP toilets, and from 48% to zero for UDDT latrines. The medium in which water diffuses would change significantly over the course of each drying kinetics experiment, and therefore Deff would change significantly - most likely decreasing over time. It would be useful to see the ln (MR) vs ln (t) plots to see how good the fit is to a straight line. I would anticipate deviations from linearity over time.
   Another possible reason the Fickian model would deviate from ideality over time is that depending on the relative kinetics of internal diffusion and evaporation at the surface, a skin can form at the surface of the matrix. The skin would act as a barrier membrane and slow down evaporation in the latter part of the drying experiment. This is a commonly observed phenomenon in the drying of foods and would also lead to a decrease in apparent Deff over time. Note that skin formation would be expected to impact drying kinetics more significantly in thicker sludge - such as would be expected in drying operations.

5. It would be useful to show the diffusivities and activation energies in Table 7 with confidence intervals so the reader can see if the differences for different sludges are statistically significant.

6. Assuming the differences in diffusivities are statistically significant, can the authors explain some of the observed differences? For example, why is the diffusivity in UDDT at several of the temperatures higher than the other sludges even though it is has the lowest initial water content?

7. A point of precision: I think of diffusion as the phenomenon of a chemical species moving in a medium due to molecular level motion. So it is water as the chemical species - not moisture – that diffuses in faecal sludge. Accordingly, $D_{eff}$ is the effective diffusion coefficient of water, not of moisture.

**D Interpretation and conclusions:**
The authors attribute a great deal of significance to bound water vs free water, and present interpretations of varying regions of drying curves to the critical moisture content. I agree that there it is important to distinguish between the two types of water which helps to understand when mechanical dewatering processes can be used vs when heat addition to facilitate

evaporation/drying is necessary. There are two additional points to make:

1. The presence and extent of bound water can be more easily determined by more direct methods - particularly measurements of the thermodynamic activity of water in the sludge at varying stages of drying, and measurements of boiling points. Bound water would have a slightly higher boiling point than free water that would be observable by differential scanning calorimetry. Using drying kinetics curves to infer bound vs free water is indirect, and other phenomena can complicate data interpretation.

2. In broad terms, transport rates are related to the driving force divided by resistance. The free water/bound water argument addresses only the driving force component of transport rate - when all the free water has been removed, the thermodynamic activity falls below one and the driving force decreases. However, resistance is also increasing as the matrix becomes dryer or a skin is formed and can also impact drying kinetics.

**Is the work clearly and accurately presented and does it cite the current literature?**
Partly

**Is the study design appropriate and is the work technically sound?**
Partly

**Are sufficient details of methods and analysis provided to allow replication by others?**
Partly

**If applicable, is the statistical analysis and its interpretation appropriate?**
Partly

**Are all the source data underlying the results available to ensure full reproducibility?**
No source data required

**Are the conclusions drawn adequately supported by the results?**
Partly

***Competing Interests:*** No competing interests were disclosed.

**I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.**