RESEARCH ARTICLE

Effect of storage time on the properties of faeces [version 1; peer review: 1 approved, 1 approved with reservations]

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Abstract

Background: This short-term study was carried out in 2013 at the Pollution Research Group at the University of KwaZulu-Natal (Durban, South Africa) as an assignment of a final year Chemical engineering student project. It focussed on the effect of storage time on the properties of fresh human faeces, more particularly moisture content, thermal conductivity, volatile solids and chemical oxygen demand (COD). These properties are important as they are often used in the design of drying or thermal treatment technologies for faeces. A storage period of one week was hypothesised at room ambient temperature.

Methods: The samples were tested for chemical properties using standard operational procedures developed at the Pollution Research Group with parameters such as totals solids, moisture content, suspended solids, volatile solids, COD and thermal conductivity.

Results: The thermal conductivity on average was 0.44 W/m.K. The moisture content was observed to decrease by 16%, from 77% to 61%. Thermal conductivity was plotted over the range of moisture contents to observe any trends, varying from 0.074 W/m.K to 0.61 W/m.K for dry faeces and water respectively. The use of a composition weighted average model fitted the data well and was found to be within 95% of the confidence interval of the best fit line.

Conclusions: The effect of storage time was found to be negligible on COD and thermal conductivity however moisture content decreased as days progressed and the volatile solids increased with an increase in storage time. Examining the relationship between thermal conductivity and moisture content, it was found that thermal conductivity increase with an increase in moisture content.

Keywords

Chemical oxygen demand, faeces, Moisture content, storage time, Thermal conductivity
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Author roles: Pandarum S: Conceptualization, Formal Analysis, Methodology, Writing – Original Draft Preparation; Ramkalawan Y: Conceptualization, Writing – Original Draft Preparation; Velkushanova KV: Conceptualization, Project Administration, Resources, Supervision, Writing – Review & Editing; Jingxi EZ: Writing – Review & Editing; Foxon KM: Supervision

Competing interests: No competing interests were disclosed.

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Introduction

According to the World Health Organization & UNICEF (2010), 2.6 billion of the world population lack adequate sanitation facilities. According to WASHwatch.org, in South Africa in 2015, the total number of people lacking access to basic sanitation was 26.4%; 8.1% of the population was using ‘unimproved’ sanitation services and 2.25% of defecated in the open.

At the same time, the conventional flush toilet sanitation systems are water- and energy-intensive, and for water-scarce countries such as South Africa, systems that require less water are becoming desirable to relieve the water usage. In the search for innovative and more sustainable sanitation solutions, it is envisioned that energy is derived from the combustion of faeces. Fresh faeces is too high in moisture content to ignite, and therefore a drying step is required. Data on the thermal conductivity of faeces is required for the design of drying and thermal treatment systems of faeces. However, these data are limited and further studies are required in the field.

It is important to determine the effect of storage time as the faeces characteristics may change with storage time as stockpiling of faeces may be required in the design. This paper aims at assessing the effect of storage time on the characteristics of faeces through analysis of thermal conductivity, moisture content, volatile solids and chemical oxygen demand (COD). It also assessed the correlation between changes in thermal conductivity and moisture content within a given period.

Drying of solids

Drying occurs as a result of the vaporization of liquid by supplying heat to wet feedstock. Drying is categorised into: direct (convection), indirect or contact (conduction), radiant (radiation) and dielectric or microwave (radio frequency) drying (McCabe et al., 1993; Parikh, 2014). Heat and mass transfer are the dominant mechanisms of the drying process, as heat is transmitted to the product in order to evaporate liquid, and mass is transferred as a vapour into the surrounding gas. The factors that affect heat and mass transfer control the drying rate (Parikh, 2014).

Mass-transfer-dependent dryers are direct dryers and essentially pass a gas through the solids to facilitate the removal of moisture. These dryers are often called adiabatic dryers. Heat-transfer-dependent dryers are indirect dryers and remove moisture by vaporizing the water or liquid using heat. These dryers are therefore non-adiabatic dryers and limited by heat transfer, and depend on the thermal conductivity for their design. Many non-adiabatic designs are based solely on the consideration of heat transfer analysis alone (McCabe et al., 1993).

The correlation between moisture content & thermal conductivity

Similar research of organic material done in other investigations, suggests that there is strong dependence of thermal conductivity on the moisture content of the material. A study conducted by Nayyeri et al. (2009) on the thermal properties of dairy cattle manure suggests that the effect of moisture content on the thermal conductivity of cattle manure is even greater than the effect of temperature. However, data on the thermal conductivity of human waste are not very common and motivated this study.

Physico-chemical analysis of faeces characteristics

For this study, the COD, volatile solids and moisture content were measured to identify their correlation with the thermal conductivity of faeces. COD refers to the amount of oxygen required by a portion of sample such that it is completely oxidised, and is calculated using the Closed Reflux Titrametric Method. COD is thus indicative of the biodegradable material in a sample of material. Moisture makes up approximately 79.2% (Almeida et al., 1999) of fresh faeces. A proportion of the remaining solids (84.4% - quoted by Lopez Zavala et al., 2002) are made up of volatile solids, solids which ignite upon high temperatures.

Figure 1(a) illustrates a linear model of thermal conductivity and moisture content that was fitted to the experimental data obtained from cattle manure and in Figure 1 (b) four parameters model was fitted to the data and the experimental curves obtained over the 3 temperatures (9, 24 & 39°C). From the study it was observed that the temperature did not have significant effect on the thermal conductivity of separated manure solids over the 30°C range investigated.

Methods

Materials

The study was carried out in 2013 whereby individual samples of fresh faeces were collected at the Pollution Research Group Laboratory facilities at the University of KwaZulu-Natal (Durban, South Africa) based on anonymous donations and stored in a cold room at 4°C. The individual stool samples were homogenised together using a blender. The entire mixed sample was transferred into a bucket and closed with a lid. Holes were drilled into the side to allow movement of oxygen to the surface such that aerobic degradation could occur. The sample with the faecal mix was then stored in the laboratory for a one-week period at a room temperature of 24-25°C. The sample was sealed so to hinder accelerated moisture content loss as a result of unlimited air supply.

Thermal conductivity

The C-Therm TCi Thermal Conductivity Analyser was used for this study. It has an accuracy of 5% and a precision of 1%, and is able to measure substances of temperatures between -50°C and 200°C. It can also measure a wide range of thermal conductivities, varying from 0 to 120 W/m.K, see Figure 2 below. Samples of 1.88 mL were placed in the sampling sensor, as indicated by the standard operating procedure. C-Therm TCi 2.3 software was used, and the thermal conductivity was directly measured. The software has the ability to take multiple measurements on a single sample and samples were analysed in triplicate.

Moisture content volatile solids

Three samples of 10 g each were used to analyse the moisture content of the faeces in triplicate. The samples were placed in an oven (Gallenkamp) at 105°C and dried for 24 hours. The
mass evaporating and remaining represent the moisture content and total solids respectively. The samples remaining were then placed in a furnace (Furnace E160) at 550°C for 2 hours, after which the mass was measured. The mass loss on ignition was taken to be the volatile solids, and therefore the difference in mass from before and after being put in the furnace was measured. Moisture content and volatile solids was calculated using the following equations:

\[
MC = \frac{m_{\text{crucible}} + m_{\text{faeces}} - m_{\text{exit oven}}}{m_{\text{faeces}}} \quad (1)
\]

Where:
- MC = Moisture Content
- \( m_{\text{crucible}} \) = mass of a crucible
- \( m_{\text{faeces}} \) = mass of faeces
- \( m_{\text{exit oven}} \) = mass of crucible & faeces after drying in the oven

\[
VS = \frac{m_{\text{exit oven}} - m_{\text{exit furnace}}}{m_{\text{faeces}}} \quad (2)
\]

Where:
- VS = Volatile solids
- \( m_{\text{faeces}} \) = mass of faeces
- \( m_{\text{exit furnace}} \) = mass of faeces & crucible exiting the furnace

Figure 1. Thermal conductivity and moisture content correlations obtained from Bonhoff & Converse (1986). (a) Linear model; (b) four parameter model.

Figure 2. C-Therm TCI Thermal Conductivity Analyser.
The COD was calculated using the Standard Closed Reflux, Titrimetric Method. A sample with mass of 1 g was dissolved in 1 litre of distilled water. Triplicate samples of 5 mL were measured from the mixed solution. The samples were digested for 2 hours in an acidic dichromate solution in the Ethos One High Performance Microwave Digester. The digested mixture was then titrated with ferrous ammonium sulphate (FAS). The COD was then calculated from the standard equations:

\[
COD = \frac{(\text{Blank-Titration}) \times \text{molarity of FAS} \times 8000}{\text{Sample(mL)}}
\]

(3)

\[
COD (\text{gO}_2/\text{g sample}) = \frac{\text{COD (mg O}_2/\text{L)}}{\text{Dilution factor} \times 1000}
\]

(4)

Where:

FAS = ferrous ammonium sulphate

mg O₂/L = milli grams of Oxygen per litre

gO₂/g sample = grams of oxygen per gram of sample

Statistical analysis

Averages and standard deviations were calculated using the inbuilt functions of Microsoft Excel. Confidence interval testing was done for part 2, for data relating the thermal conductivity to the moisture content.

Confidence interval

The confidence interval was done on the slope and intercept of the data.

\[
Y = a + bX
\]

(7)

Where Y is represented by the thermal conductivity and X is represented by the moisture content. a and b represent the y-intercept and slope of the best fit line respectively. Values for a and b using a best fit line was 0.041 and 0.561 respectively. The following are the equations used to calculate the confidence intervals.

\[
a \pm t_{(\nu, N-2)} S_a \sqrt{\frac{\sum (X^2)}{NS_{XX}}}
\]

(8)

\[
b \pm t_{(\nu, N-2)} S_b \sqrt{S_{SXX}}
\]

(9)

Where the following variables are defined and calculated using table in appendix with \( \bar{X} = 0.42 \) and \( \bar{Y} = 0.28 \), and \( t = 2.228 \):

\[
S_{XX} = \sum (X_i - \bar{X})^2 = 1.15
\]

(10)

\[
S_{YY} = \sum (Y_i - \bar{Y})^2 = 0.38
\]

(11)

\[
S_b = \sqrt{\frac{S_{YY} - b^2S_{XX}}{N - 2}} = 0.049
\]

(12)

\[
(\bar{X})^2 = 2.82
\]

(13)

The confidence intervals for a and b were thus:

\[
a: 0.043 \pm 2.228 \times 0.049 \sqrt{\frac{2.82}{12 \times 1.15}} = 0.043 \pm 0.050
\]

\[
b: 0.56 \pm 2.228 \times 0.049 \sqrt{1.15} = 0.56 \pm 0.10
\]

Results and discussion

This section presents the results of the different analysis of the fresh faeces and assess the correlations between thermal conductivity, COD, volatile solids, total solids and moisture content.

Effect of storage time on thermal conductivity

Figure 3 illustrates effect of storage time on the thermal conductivity of the faeces. Thermal conductivity varied slightly over the course of the experiment. The initial average thermal conductivity was 0.46 W/m.K, then increased to 0.48 W/m.K and remained constants as days progressed; however, day 6 attributed to variation of material within the storage bucket, with an average thermal conductivity of 0.33 W/m.K (which is considered as an outlier). It needs to be noted that the rate of degradation and dehydration may not have been uniform throughout the sample as it was observed that the surface material was dryer and harder than the wetter, softer material beneath, resulting in inconsistencies with analytical tests. In a study aimed at determining the thermal properties of composting bulking materials such as saw dust, soil compost blend, beef manure and turkey litter by Ahn et al. (2009), thermal conductivity ranged from 0.12–0.81 W/m°C. Therefore, the thermal conductivity obtained in this study fell within the range reported by Ahn et al. (2009).

Effect of storage time on COD

Figure 4 illustrates the effect of storage time on the COD of the faeces. The COD did not present any significant trend over the period of investigation, and averaged at a value of 0.82 g O₂/g dry sample, suggesting that the amount of oxidizable material remained fairly constant. There was no significant relationship to suggest that storage time has an effect on the COD.

Effect of storage time on moisture content and volatile solids

Figure 5 illustrates average results of moisture content (Figure 5a) and volatile solids (Figure 5b) during the effect of storage time experiment. Over the same period of days, the
Figure 3. Thermal conductivity as a function of time.

Figure 4. Chemical oxygen demand as a function of time.

Figure 5. Effect of storage time on (a) moisture content and (b) volatile solids.
moisture content decreased by approximately 16%, from the initial moisture content of 77% to 61%. This can be expected due to the concentration gradient existing between the sample and the surrounding air and because of this gradient, mass transfer of water from the sample to the air occurs (McCabe et al., 1993).

The volatile solids resembled a trend inverse to that of the moisture content, consisting of (on average) 85% of the total solids, compared to 84.4% as quoted by Lopez Zavala et al. (2002). Two factors contributed to the volatile solids, (i) the distribution of particles within the homogenous mixture as it was difficult to completely homogenise a sludge mixture and (ii) the variation of degradation and dehydration of the faeces within the storage bucket. The rate of degradation and dehydration may not have been uniform throughout the sample as it was observed that the surface material was dryer and harder, as compared to the wetter, softer material beneath it.

Establishing a relationship between thermal conductivity & moisture content
For better understanding of the relationship between thermal conductivity and moisture content, the thermal conductivity of water (100% moisture content) and dry faeces (0% moisture content) were also measured. A simple model, using a composition-weighted average of the thermal conductivity of dry faeces and water was measured at 0.074 W/m.K and 0.61 W/m.K respectively, was proposed to predict the thermal conductivity of faeces (Figure 6). A 95% confidence interval test was performed on both the slope and intercept of the proposed model, and it was observed that both were well within range. The slope of the proposed model (0.54) was within the confidence interval of [0.45: 0.66] and the intercept of 0.074 was within the calculated range of [-0.0069 : 0.093]. When plotting the upper and lower confidence lines, it was evident that the model fitted well within the 95% confidence of the linear line of best fit. Thermal conductivity of water measured precisely to a literature value of 0.61 W/m.K (Ramires et al., 1995). Nayyeri et al. (2009) also carried out a study on thermal properties of dairy cattle manure and observed a linear relationship between thermal conductivity and moisture content where by increasing trend in the thermal conductivity of dairy cattle manure was also observed with the increase in moisture content, which was the same trend as observed by Yang et al., 2002.

**Conclusion**
Upon investigating the effect of storage time on the properties of fresh faeces, for the purpose of better design of the sanitation system and to produce potable water, fertiliser and energy through combustion process. On the investigating the effect of storage time, it was found that the thermal conductivity varied insignificantly as it remained fairly constant, with an average of 0.4 W/m.K. The moisture content decreased by 16%, from 77% to 61%, this behaviour is due to the rate of degradation and dehydration that may not have been uniform throughout the sample as it was observed that the surface material was dryer and harder, as compared to the wetter, softer material beneath it. The COD presented no significant trend, averaging at a value of 0.82 g/g dry sample. The effect of storage time was found to be negligible on COD and thermal conductivity however moisture content decreased as days progressed and the volatile solids increased with an increase in storage time. Examining the relationship between thermal conductivity and moisture content, it was found that thermal conductivity increase with an increase in moisture content, which is the same trend observed in literature.

For future studies it is important to evaluate operating conditions of storage, such as temperature, pressure and type of storage units. It is also important to investigate the correlation between

![Figure 6. Correlation between thermal conductivity and moisture content.](image)
thermal conductivity and temperature and to investigate the correlation between the rate of drying and moisture content.

**Data availability**

Open Science Framework: The effect of storage time on the properties of faeces. [https://doi.org/10.17605/OSF.IO/TMZD2](https://doi.org/10.17605/OSF.IO/TMZD2) (Velkushanova, 2019).

This project contains raw data on thermal conductivity, moisture content, volatile solids and chemical oxygen demand.

Data are available under the terms of the Creative Commons Attribution 4.0 International license (CC-BY 4.0).

**Grant information**

This study was funded by the Bill and Melinda Gates Foundation, project “Mechanical Properties of Faecal Sludge” (grant OPP1164136).

The funders had no role in study design, data collection and analysis, decision to publish, or preparation of the manuscript.

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Version 1

Reviewer Report 11 June 2019

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Marc Deshusses
Department of Civil and Environmental Engineering, Duke University, Durham, NC, USA

This is a limited scope study on the property of faeces, mostly the thermal conductivity as it may be affected by short term (7 day) drying. The study reports the results of a single experiment and lacks the depth and breadth needed to derive generic information on the process. In my opinion it is missing some key controls and results have limited usefulness given the relatively short time for storage and moderate physical/chemical changes during this time. Still it could be relevant to practitioners in the field. I would suggest to be careful extrapolating from what is basically one data point to other conditions.

Specific issues I recommend to address are below:

1. Abstract: Date and location of the study are not particularly important.

2. P3, left column, last paragraph: Explain why there is strong dependency of the thermal conductivity and moisture and what is the usual functionality reported.

3. P3, L10 right column: COD is not indicative of the biodegradable material in a substance (but BOD is). Correct.

4. P3, L12 right column: Given the variability in moisture content of faeces, 79.2% is far too many digits. A range seems more appropriate here.

5. Methods/Materials: The number or samples, how many stools, the mass, and how exactly they were processed needs to be specified. Also, it is unclear if the bucket content was mixed after sampling. If not, where was the sample taken? Near the surface? If it was mixed, what were the impacts of mixing? More explanations are needed.

6. Methods/Materials: I am having a hard time understanding exactly how the faeces were aged. Please edit the methods adding enough information that if they were given to someone reasonably skilled, they would be able to reproduce the experiment.
7. Methods/Materials: The last sentence is incomprehensible. If the sample is sealed, how can there be unlimited air supply? Also, the impact of storage is unclear and not discussed.

8. Methods/Thermal conductivity: the exact model of the device should be specified, and the sample size (last line) should be specified too.

9. Figure 2 with a picture of the device is useless, remove it.

10. Figure 1: I assume that theta is the moisture content. You might want to make it clear. Are the model correlations in the original source, or is this something you derived? This is unclear.

11. Bottom of page 4: Give units with all definitions.

12. These equations could be simplified a bit as some are trivial. There is also some inconsistency on how it is reported - why write the equation with the mass of the crucible for MC but not for VS? “mfaeces” should indicate that this is the mass of wet faeces.

13. P5, L4 left column: replace “dissolved” with “suspended”.

14. Equations 3-4 are trivial and do not need to be in the paper. Delete.

15. P5, left column: “values for a and b … 0.041 etc.” This is a result, not a method and should be in the results section.

16. P5 bottom left and top right equations for confidence intervals, etc.: This is standard definition and could be in an Appendix, while the actual values move to Results.

17. Results and discussion, first 3 lines: “This section…” this is not needed. Delete.

18. P5, right column L8 from bottom: Not the right words. It is the fraction of the dry weight that is oxidizable that remained constant (not the amount). Also, here and in other places, it is important to stress “over the duration of the study period” as I’d caution about extrapolation.

19. Add error bars to Figs 3 and 5 and also indicate what they are in the caption of Figs 3-5.

20. Fig 5: Indicate if the g sample is wet or dry (seems it is wet weight).

21. P7, left column 2rd paragraph: About the 84.4% vs 85%. This is a minute difference given the experimental uncertainties.

22. Conclusion: First sentence has no verb and needs to be fixed.

23. Conclusion: Second sentence says thermal conductivity varied insignificantly which is contradicting everything else in the paper including the last sentence of that paragraph. Fix.

24. Fig 6: It seems essential to me to place the experimental data on that figure, and clearly indicate the range of moisture contents that were investigated and what is extrapolated.

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?**
No

**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

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

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

**Reviewer Expertise:** Chemical and environmental engineering, sanitation, FSM

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.

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Reviewer Report 28 May 2019

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Sudhir Pillay
Water Research Commission (WRC), Pretoria, South Africa

Suggested modifications:

**Introduction:**
- Recommend StatsSA data instead of WASHwatch.org reference for South Africa.

**Materials**
- Add in size of collection bucket (l x b x h).
- Add how samples were taken from bucket? From the top? Were they mixed before?
- Clarify purpose of drilling holes and thereafter sealing in bucket. Was not clear of procedure. Was the lid on but holes still present?

**Results and discussion**
- Effect of storage time on COD.
- The text needs to match the figures presented. And the attached spreadsheet needs to match those inserted in paper. The authors would need to do this. For example, Day 1 had double COD
than Day 2. Was this an abnormality due to inconsistency of sample? I would recommend adding
std dev to average of of 0.82 g O₂/g dry sample, the word "significant relationship" could be
interpreted/usually interpreted in a statistical manner in academic journals. Consider revision or run
an ANOVA test if data is normally distributed and then state no significant change over time.

Conclusion

- I would add biodegradability tests as part of future research.

Overall Comments:

An interesting and much needed study. The scientific language style was good and presentation
appropriate. During my review, I could not find any spelling errors or missing references (suggestion to
change WASHwatch.org reference). I would recommend changes to the section on COD change with
time and have made recommendations. Please include graphs used in paper and have it placed in Excel
attached to paper (I did not see the graphs in excel - please complete).

Faecal sludge samples are known to be physically & chemically different to domestic wastewater sludges
and domestic wastewater samples, The sample handling and preparation will therefore be different. The
snapshot results from the authors have shown this. I would highly recommend the authors to mix samples
in the bucket and take samples from bucket to see if there is more consistency in results (COD, MC, TS,
VS) - there needs to be a follow-on on this paper. The research could form part of SOP for sludge
sampling and the pre-testing/sample preparation phase for analysis. So far, I have not seen this type of
data and is definitely a knowledge gap.

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

Is the study design appropriate and is the work technically sound?
Yes

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

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

Are all the source data underlying the results available to ensure full reproducibility?
Partly

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Faceal sludge management, Anaerobic Digestion, Membrane Filtration

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.