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Feasibility study on using combined tomography and spectroscopy techniques to evaluate the physical and chemical characteristics of organo-mineral fertilisers

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Fertilisers play a key role in agriculture, providing key nutrients needed by crops to ensure a secure food supply. However, with increasing prices and rising environmental concerns, there is a growing need to rely on alternative and sustainable fertiliser sources, introducing the opportunity to use organic amendments to formulate organo-mineral fertilisers (OMF). Despite their environmental advantages, the inherent variability in composition of organic amendments within OMF poses a challenge for their standardization. This study aims to use OMF derived from anaerobic digestate and coupled with carbon capture technologies to analyze for its physical characteristics and chemical composition using neutron computed tomography (NCT), X-ray computed tomography (XCT) and Raman spectroscopy (RS). This is a feasibility study to assess using non-destructive techniques on OMF as previously this has not been explored. This work represents the first attempt to utilize a combination of imaging techniques to investigate on OMF and demonstrates their feasibility for measuring the variability between individual samples. This is a proof-of-concept study which shows that combining NCT and XCT can provide images on how uniformly packed each OMF pellet are. The use of RS is to characterize OMF is more challenging largely due to the high fluorescence background arising from its matrix. This study needs to be further developed to enable image-based analysis using machine learning algorithms to determine characteristics of large batches of OMF. Further development is needed building on this work to quantify OMF pellet characteristics so that it can be confidently used as novel fertilisers in agriculture.

KEYWORDS

organo-mineral fertilisers, soil nutrients, neutron computed tomography, Raman spectroscopy, X-ray computed tomography

Introduction

Agriculture is a major source of greenhouse gas (GHG) emissions, requiring urgent mitigation. In the UK, agriculture is responsible for 9% of all GHG emissions, and over 50% of these emissions are due to the manufacture and use of nitrogen (N) based fertilisers to maintain yields (Defra, 2020). Crop uptake of N is also suboptimal, with nutrient use efficiency limited to 50% (Oyetunji et al., 2022). One approach to address this is to use precision agricultural approaches using variable rate application to ensure fertiliser applications match crop requirement and supply from soils (Mirzaee and Nafchi, 2024). Another challenge in agricultural landbank is soil degradation due to over-extraction of nutrients from soil with minimal replenishment (Rickson et al., 2015). This challenge is further exacerbated with the growing price of natural gas causing the cost of fertiliser production to increase and potentially affecting food security. With mineral fertilisers causing close to 2% of global greenhouse gas emissions (Menegat et al., 2022), a more sustainable way forward is to utilize renewable sources without reducing crop yields and minimising environmental impacts such as diffuse pollution. Organo-mineral fertiliser (OMF), which combines organic amendments with reduced amounts of mineral fertiliser, can partly be a solution to tackle this challenge, as discussed further in the Strength-Weakness-Opportunities-Threats (SWOT) analysis in Sakrabani (2024).

A notable challenge associated with fertilisers produced using organic amendments is that their composition can vary significantly due to the inevitable variation of feedstock characteristics, unlike equivalent mineral fertilisers whose stoichiometry is more consistent. Thus, to ensure the traceability in production of OMFs, it is important to ensure that their characteristics can be assessed and that their variability between batches can be measured. Another aspect which is important to evaluate is the compactness of OMF pellets as when farmers spread using conventional spreaders along the tramline, the flight path across 24 to 36 m is important to ensure an equal spread of nutrients can be achieved. If the OMF pellets are not of adequate compactness then when it flows from the hopper to the spinning disc, it will shatter, resulting in a cloud and inhomogeneous spread of nutrients and patchy crop growth which is not desired. Thus, it is important to get an accurate measure of OMF pellet quality in terms of its structure and chemical composition for it to be reliably used in agriculture.

Léonard et al. (2020) have demonstrated that X-ray computed tomography (XCT) can accurately determine the closed porosity of ammonium nitrate prills. In this case XCT was more effective as a non-destructive technique compared to mercury porosimetry. Biessikirski et al. (2024) have used tomography techniques to distinguish between fertiliser grade and prill grade ammonium nitrate by showing porosity characteristics such as pore size and distribution patterns. Indore et al. (2022) summarized key applications of synchrotron tomography applications in agriculture where the use of this technique allowed accurate assessment of internal structures of seeds, parts of plants such as the leaves, stalk and pore space in soil when in proximity to root hair. This imaging approach allows accurate assessment of internal structures of plants enabling us to elucidate detailed mechanisms on transport of solute from soil to plants, transport of gas between vascular components of plants and potential weak spots for attack of pest and disease. Whilst the use of imaging techniques has been established in an agricultural context, there is a knowledge gap on its application in OMFs which led to the current study. The innovation of this study exists in terms of adapting these techniques to suit organic materials which are very variable and have an inherent moisture content. Techniques using neutron are very sensitive to moisture, so samples need to be pre-dried to make it compatible. The dark color of organic material can possibly cause fluorescence which can absorb light or electromagnetic radiation, influencing spectra of samples.

This works aims to exploit a novel approach for this application, using non-destructive combination of neutron and X-ray computed tomography (NCT and XCT) to assess the physical characteristics of fertiliser pellets such as their porosity distribution, as well as Raman spectroscopy (RS) to determine their chemical composition.

Materials and methods

Sample collection and preparation

The OMF samples that were used in this study were derived from anaerobic digestate using crop residue as feedstock followed by carbon dioxide (CO_2) capture as explained in Lake et al. (2019). There is a further addition of calcium nitrate and aqueous ammonia which blends with the captured CO_2 to form ammonium nitrate and calcium carbonate, as detailed in Lake et al. (2019). The resulting mixture is then pelleted, resulting in an OMF which both tackles the climate change aspect of fertiliser production and demonstrates an excellent example of a circular economy.

The samples in the form of pellets were stored in airtight sealed bags as only small quantities were needed for this study. Three samples were used in each case when dealing with NCT, XCT and RS. The pellets can swell by absorbing ambient moisture, hence airtight sealed bags were used.

Neutron computerized tomography and X-ray computed tomography

NCT was performed on the IMAT beamline (Burca et al., 2013; Burca et al., 2018) at the ISIS Neutron and Muon Source, while XCT was performed on the I12-JEEP beamline (Drakopoulos et al., 2015) at Diamond Light Source, representing the first attempt to study the structure of OMF pellets with combined neutron and X-ray imaging techniques.

The different interactions of X-rays and neutrons with matter enabled to exploit the complementary information provided by both modalities on these types of samples. X-rays primarily interact with the electrons of an atom, whereas neutrons are neutral particles that interact with atomic nuclei. While X-ray imaging provides information on pellet composition and structure, it offers limited contrast between water and other biological pellet constituents. Neutrons, however, provide a complementary perspective where water and biological matter can be clearly differentiated.

The basic principle of neutron/X-ray radiography involves a beam of neutrons/X-rays that is attenuated as it passes through a sample, and the intensity of the remaining beam is measured with a corresponding 2D spatially resolved detector. The resulting 2D "projection" image (or radiograph) does not, however, contain all the sample's 3D spatial information. By rotating the sample in front of the detector and collecting a set of projections at a range of sample rotation angles (Figures 1a,b), a 3D reconstruction of the object and its internal structure can be obtained.

Raman spectroscopy

The Raman spectra were acquired with the inVia Renishaw microscope available at the Centre Laser Facility (Omori et al., 2021; Robertson et al., 2023) using an 830 nm excitation wavelength to minimize fluorescence from the samples, a 20X objective and a 1 mW laser power. For each sample, 20 spectra were collected non-invasively at various locations on the fertiliser pellets, highlighting eventual heterogeneities within each sample as well as the differences in the chemical composition between samples. RS analyses the inelastically-scattered photons from a sample which undergo a small energy shift with respect to the incident laser radiation. This energy shift carries information about the vibrational energy levels that are related to the molecular composition of the samples. RS is highly chemical specific and non-invasive with no sample preparation but limited to point like analysis whilst imaging is time consuming.

RS provides high specificity due to its sensitivity to molecular fingerprint regions. Raman analysis workflows focus generally on spectral matching, which allows for confident identification, provided the spectral resolution is below 2 cm^{-1}. In this specific case the frequency found in the sample matched the reference Raman spectra in the RRUFF™ Project database (Lafuente et al., 2015).

Results

The neutron radiographies and the reconstructed NCT slices show that the distribution of the dense matter within a pellet is not uniform along the vertical axis (Figures 1b,d). Characterising such variations of material packing density within the pellet is important, as they can be linked to the heterogeneity of nutrient distribution when released into soil. Moreover, the high moisture content of the pellet could be revealed by the strong interaction of neutrons with hydrogen, as shown by the neutron measurements (Figures 1c,d). Additionally, XCT provides valuable information on the internal structure, texture, and porosity, which are important indicators of the pellet's quality and performance (Figures 1e,f) that NCT cannot provide, demonstrating that XCT and NCT are complimentary techniques with regards to pellet imaging.

The data obtained from RS, as shown in Figure 2, highlights a low variability in molecular compositions among different pellet samples, while also showing the presence of chemical heterogeneity within each sample. Due to their characteristic vibration modes, it was possible to identify the presence of magnesium nitrate ($MgNO_3$) with symmetric

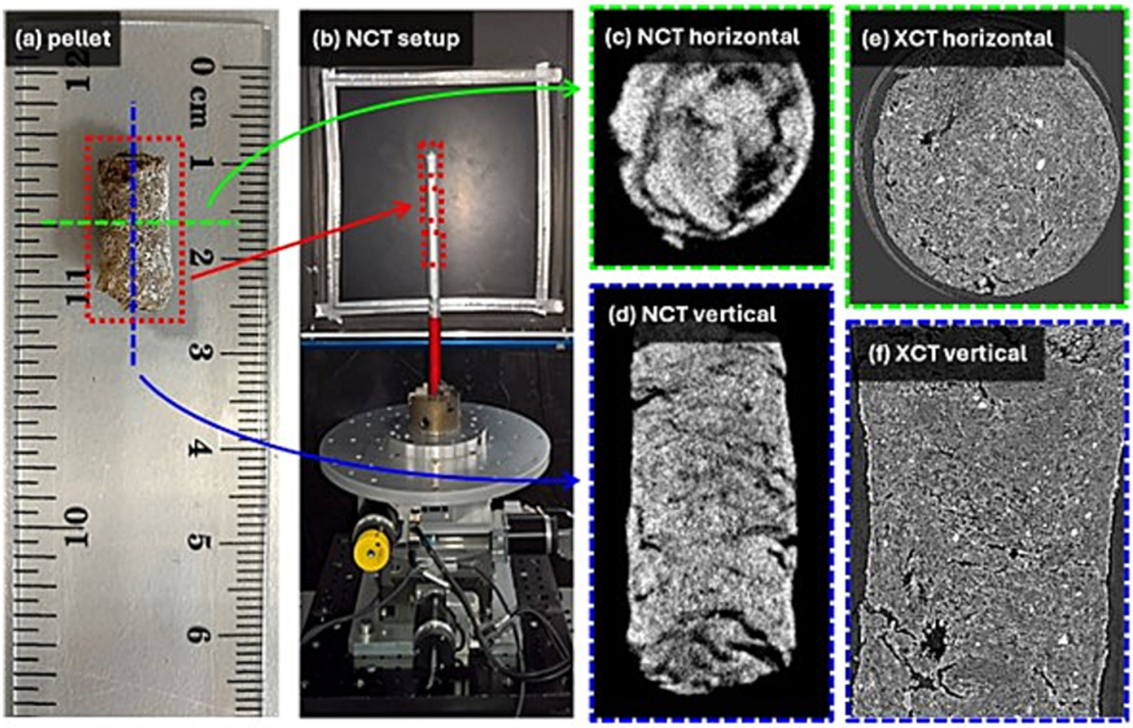

FIGURE 1
(a) Photograph of one of the imaged fertiliser pellets (ruler for scale), where the dashed lines indicate the NCT and XCT horizontal (green dashed line) and vertical (blue dashed lines) slicing planes; **(b)** Photograph of the NCT experimental setup, where the pellets (red dashed lines) are inserted into a thin aluminium tube and mounted onto a tomography stage in front of the imaging detector; **(c,d)** Horizontal (green) and vertical (blue) cross-sections taken from NCT reconstructions of the pellet in **(a)**, showing variations in packing density indicating the distribution of pellet constituents; **(e,f)** Horizontal (green) and vertical (blue) cross-sections taken from XCT data of the same pellet, showing detailed internal structure of the pellet's constituents.

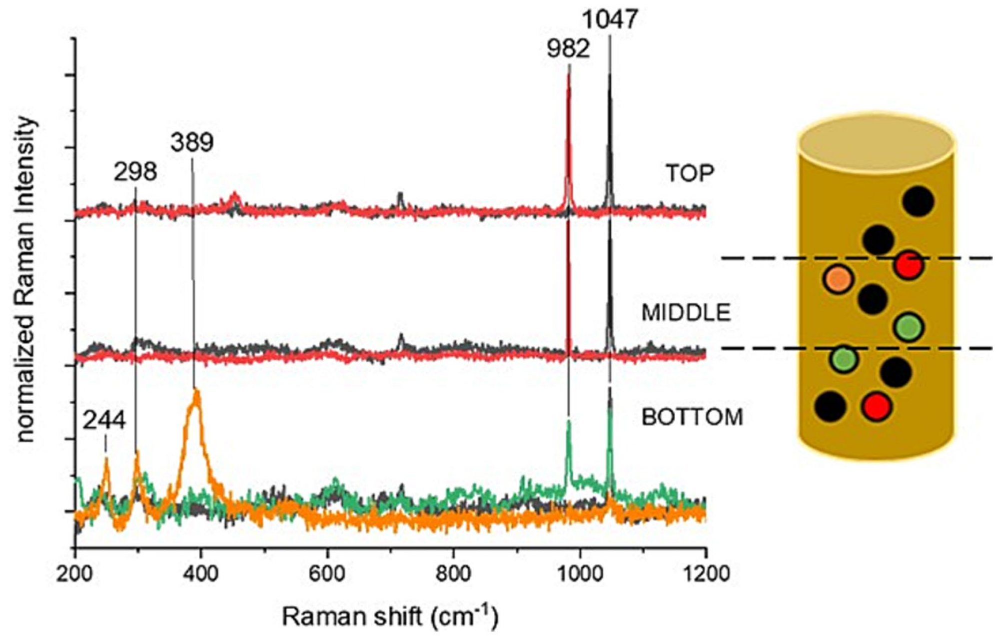

FIGURE 2
Raman spectra of the chemical compounds identified in three different pellets. Spectra are vertically shift for clarity according to the location of the pellet in which they were measured (i.e. bottom, middle, top), different colour line indicate different location points within the area. Number on each peak indicate the Raman frequency used for identification. A schematic of the pellet is shown on the right, horizontal line indicate the 3 different area and different circles highlight different locations probed with Raman instrument.

stretching at 1046 cm^{-1}, magnesium sulphate ($MgSO_4$) with a vibration mode at 982 cm^{-1}, and a localized presence of iron oxide [α-FeO(OH) goethite] in the fertiliser pellet matrix characterized by the main Raman bands centred at 244 cm^{-1}, 298 cm^{-1}, and 389 cm^{-1}.

Discussion

Burak and Sakrabani (2023) reported in a field study using carbon captured based OMF that crop yield was not compromised when compared to mineral fertilisers. One of the potential explanations for this is in field scale observations when OMFs were applied on the soil surface, the presence of organic matter within it allows to absorb soil moisture enabling it to swell and release nutrients. Information when using NCT on how densely packed each OMF pellet is important to determine potential for ingress of soil moisture. A very densely packed OMF will be more resistant to ingress of soil moisture than a tightly packed one. Whilst in the field scale context, swelling of OMF pellet is vital for physical breakdown to release nutrients is desirable, the opposite is the case when storing and applying OMF. In the context of OMF storage, it is housed in a conventional farm barn exposed to ambient weather conditions. Thus, if the packaging material where OMF pellets are kept is not optimum, then ambient moisture ingress will cause detrimental effect to the quality. If each OMF pellet swells within a bag, then it can fuse and 'cake' which makes spreading using conventional spreaders not feasible. So, NCT allows a non-destructive approach to quantify the distribution of the dense matter within each pellet as shown Figures 1c,d. XCT provides complimentary information on distribution of porosity which is linked to moisture ingress and its implications as explained earlier on. Information on distribution porosity of OMF pellets can influence the flight trajectory when spread from a tramline. An OMF pellet which has high porosity can be challenging to spread as it can potentially easily shatter when it hits the spinning disc. On the other hand, a highly porous OMF pellet when uniformly applied to the soil surface, can absorb moisture to swell and release nutrients. Consequently, a trade-off is required on the physical quality of OMF pellet which can provided when using NCT in conjunction with XCT.

The challenges associated with acquiring and analysing RS pellet data were largely due to the high fluorescence background arising from the OMF matrix and the presence of impurities. For this reason, a near-infrared excitation wavelength was selected to minimize this effect. Moreover, due to its point-like nature, it was necessary to obtain several measurements at various positions on the sample to identify the presence of all chemical compounds within the pellet. This required the additional optimization of measurement parameters such as the power density on the sample, acquisition time, and the number of measurement positions. Overall, it was more challenging to measure using RS on OMF pellets due to the nature of the organic material. Nevertheless, it provided information on the presence of Mg and Fe and its distribution within each pellet. This is an important consideration as when the pellets will be applied from a spinning disc spreader along a tramline, its nutrient distribution in its intact form is very relevant using NCT, XCT and RS to achieve homogenous spread. When compared to conventional wet chemistry techniques such as acid digestion followed by measurement of metals using ICP-MS or ICP-OES, the determination is for a bulk sample rather than distribution at various depths within each pellet. The use of acid digestion causes sample destruction to provide total metal concentration unlike using non-conventional methods which use intact samples.

This feasibility study provided an opportunity to test the use of non-destructive techniques such as NCT, XCT and RS to characterize physical and chemical composition of OMF pellets. Further work is needed to fine tune the approaches when using these techniques and adapt it to OMF pellets. When this has been achieved, there is potential for using machine learning techniques to process several images of OMF pellets using XCT and NCT to enumerate packing density of material and porosity within batches. In terms of future research, benchmark products using specific feedstock can be determined and using machine learning algorithms we can utilize this to standardize across a range of products. This will be vital to ensure that a suitable and reliable product can be used by farmers to promote sustainability in food production whilst ensuring soil health is not compromised.

Conclusion

This study highlights the potential of using techniques such as NCT, XCT, and RS for measuring the variability between OMF samples. In the future, further fine-tuning of these approaches is needed to ensure that this proof-of-concept can be used as a method of ensuring traceability during the manufacture of OMF as a novel green fertiliser to be used in sustainable intensification of agriculture. In practical terms, as organic feedstock components of OMF are heterogenous, use of novel approaches as in this study can be used to quantify variability between batches of fertilisers.

Data availability statement

Data supporting this study are openly available from the CORD repository at https://doi.org/10.57996/cran.ceres-2762.

Author contributions

RS: Conceptualization, Funding acquisition, Project administration, Resources, Supervision, Writing – original draft, Writing – review & editing. SM: Data curation, Investigation, Methodology, Visualization, Writing – review & editing. AL: Writing – review and editing, Visualization, Formal analysis. GB: Conceptualization, Investigation, Methodology, Data curation, Resources, Supervision, Project administration, Funding acquisition, Writing – review and editing.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Generative AI statement

The authors declare that no Gen AI was used in the creation of this manuscript.

Publisher's note

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