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X-RAY COMPUTED TOMOGRAPHY: AN OUTSTANDING VISUALISATION TOOL FOR DRYING RESEARCH – Feedback on the last 20 years at University of Liège

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Abstract
X-ray tomography refers to cross-sectional imaging of an object from transmission data collected by using an X-ray beam to irradiate the object under many different directions. It is a non-destructive evaluation technique giving access to characteristics of both the external and internal structure of an object such as dimensions, shape, internal defects,... Based on more than 20 years of expertise, this paper will show some beneficial uses of this technique in the context of drying research.

Keywords: *X-ray tomography, drying, shrinkage, cracking, structure*

1. Introduction

X-ray tomography (from greek: tomê, section and graphein, to describe) refers to cross-sectional imaging of an object from transmission data collected by using an X-ray beam to irradiate the object under many different directions. It is a powerful non-destructive evaluation technique giving access to characteristics of both the internal and the external structure of an object such as dimensions, shape, internal defects or density. Originally developed in the field of diagnostic medicine, this technique finds now numerous non-medical applications. The development of microfocus X-ray sources and high resolution CCD detectors led to the commercialisation of laboratory X-ray microtomographs with improved resolution (around a few microns) dedicated to the investigation of small samples. In this paper, we illustrate the potentialities of the technique regarding drying research, based on an expertise of more than 20 years. X-ray microtomography can be advantageously used to follow shrinkage and crack development, to determine internal moisture profiles and to characterize the microstructure of dried materials. Large systems can be used, for example, to characterise the structure of packed beds. For the time being, no X-ray tomograph has been built around a laboratory drying equipment. The follow-up of the drying process thus requires the interruption of the drying process to scan the sample being dried.

2. Equipments at ULièged

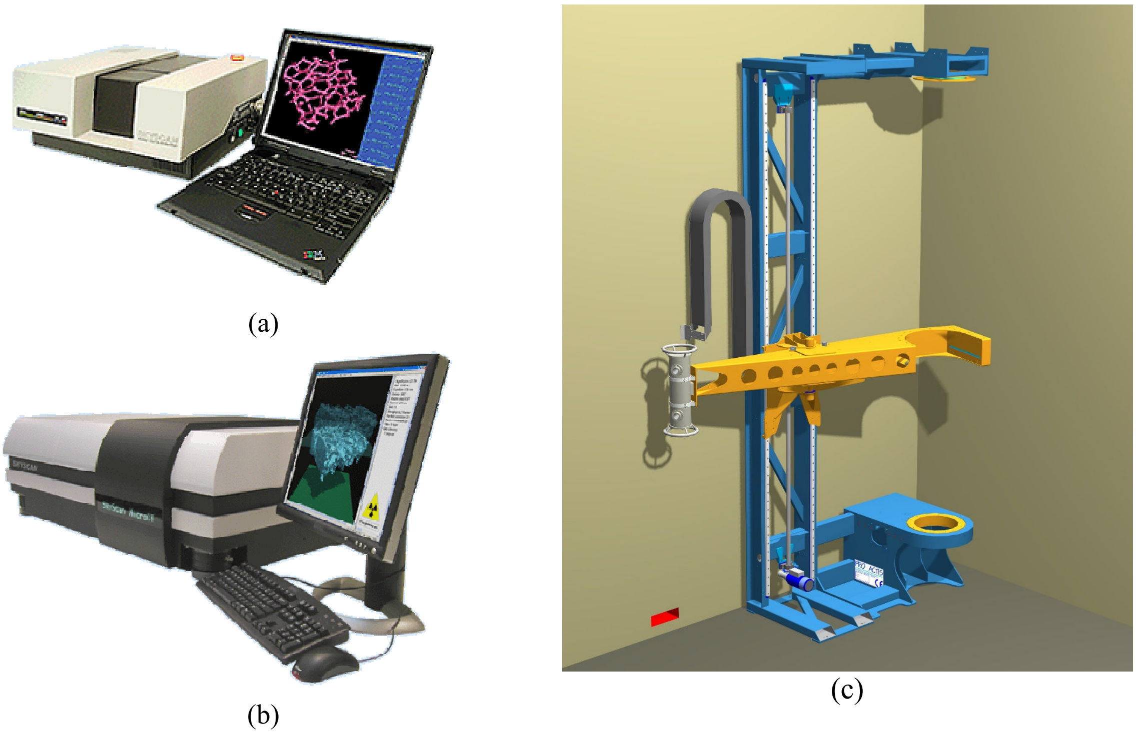
X-ray tomography is used in combination with experimental drying facilities. Three X-ray tomographic systems are available at the Chemical Engineering Research Unit, as well as three drying equipments.

The Skyscan 1074 X-ray scanner (Bruker, Belgium) was bought in 1999 (Fig. 1a). The cone beam source operates at 40 kV and 1 mA. The detector is a 2D, 768 pixels × 576 pixels, 8-bit X-ray CCD camera giving images with a fixed pixel size of 41 µm.

The Skyscan 1172A microtomograph (Kontich, Belgium) was obtained in 2006 (Fig. 1b). With its CCD pixel size of 11.5 µm, and its camera and detector displacement range, it can theoretically reach a pixel size of 0.7 µm per voxel. Nevertheless, the focal spot is at least 5 µm in diameter, thus adding strong diffusion in the radiograms (and hence a high amount of blurring in the reconstructions).

The macro-tomograph was built in 2005 (Fig. 1c), as one of first European high energy (420 kV), large-scale (max. 0.45 m in diameter and max. 4 m in height) X-ray tomograph (Toye et al., 2005).

The generator is a Baltograph CS450A (Balteau NDT, Belgium), which may be operated between 30 and 420 kV. The X-ray source is an oil-cooled, bipolar TSD420/0 tube (Comet and Balteau NDT), whose intensity may be varied between 2 and 8 mA depending on the voltage used. A lead collimator produces a 1 mm thick fan beam. The detector is an X-Scan 0.4f2-512-HE manufactured by Detection Technology (Finland). It consists of a linear array of 1280 photodiodes each coupled with a CdWO4 scintillator. The mechanical rig designed by Pro Actis, Belgium, consists of two parts, asource–detector arm and a rotating table on which the object to be scanned is fixed. This arm is embedded in a carriage that slides on two vertical high precision machined rails. The rig allows vertical movement up to 3780 mm, keeping verticality and horizontality errors within 1 mm.


(a)

(b)

(c)

Fig. 1. Skyscan 1074 X-ray scanner (a); Skyscan 1172A microtomograph (b): Macrotomograph.

The three convective dryers available allow to study the drying kinetics of both small or individual samples and fixed bed, either in air or superheated steam (Fig. 2).


Fig. 2. Dual cell convective dryer using either air or superheated steam

A lot of different materials have been tested using drying associated to tomography. It will be focused here on wastewater sludge, resorcinol formol gels, and clay.

3. Typical applications

The first study ever performed in the team coupling drying and tomography was the follow-up of convective drying of individual sludge extrudates. The methodology still used today was developed during a PhD thesis (Leonard, 2003) and needs to interrupt the drying operation to put the sample in the X-ray microtomograph (Leonard et al., 2002). Dedicated image analysis algorithms were developed in order to extract useful indicators such as shrinkage, cracks, moisture profiles and their evolution with the sample water content (Leonard et al., 2003, Leonard et al., 2004, Leonard et al., 2005). Fig. 3 illustrates, from left to right a radiography of the wet and dried sludge sample, a reconstructed grey scale cross section in the dried sample and the extraction of the cracks after segmentation. X-ray microtomography has also be used in order to better understand the influence on the sludge dewatering step, in particular the conditioning step, on further drying. The follow up of the product inner structure during drying allowed to observe the impact of the use of different coagulant/flocculant combinations, as illustrated in Fig. 4.

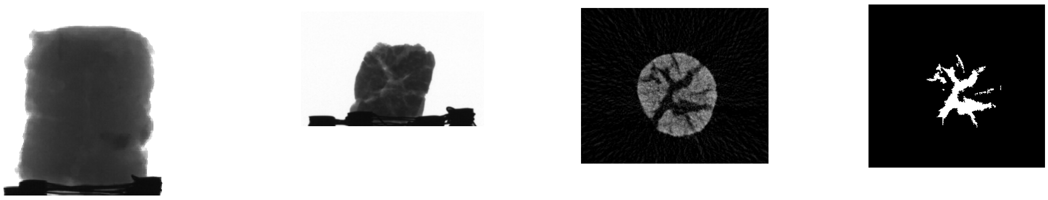


Fig. 3. Radiography of the wet and dried samples, cross sections in the dried sample, cracks extraction (from left to right)

Fig. 4. 2D cross section of dried sludge samples dewatered using different coagulant/flocculant combinations (from left to right) (Pambou, 2016)

This methodology has also been applied in order to optimize the convective drying of resorcinol-formol xerogels, keeping them free of cracks (Job et al., 2006). The determination of internal moisture profiles was also used to validate thermo-hygro-mechanical modelling of xerogel drying (Escalona et al., 2010). Fig. 5 shows the comparison between experimental and simulated moisture profiles for a typical synthesis condition expressed by the ratio between resorcinol and the so-called catalyst, i.e. sodium carbonate, R/C.

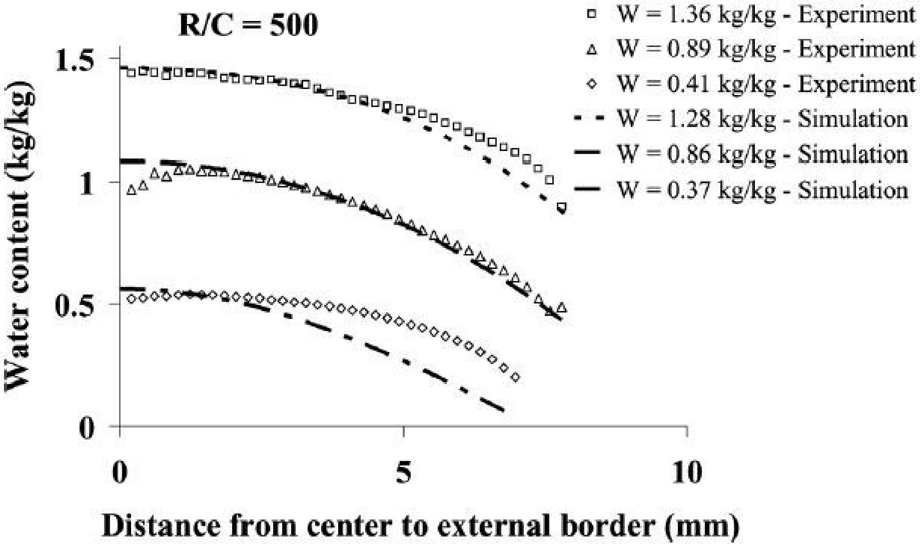
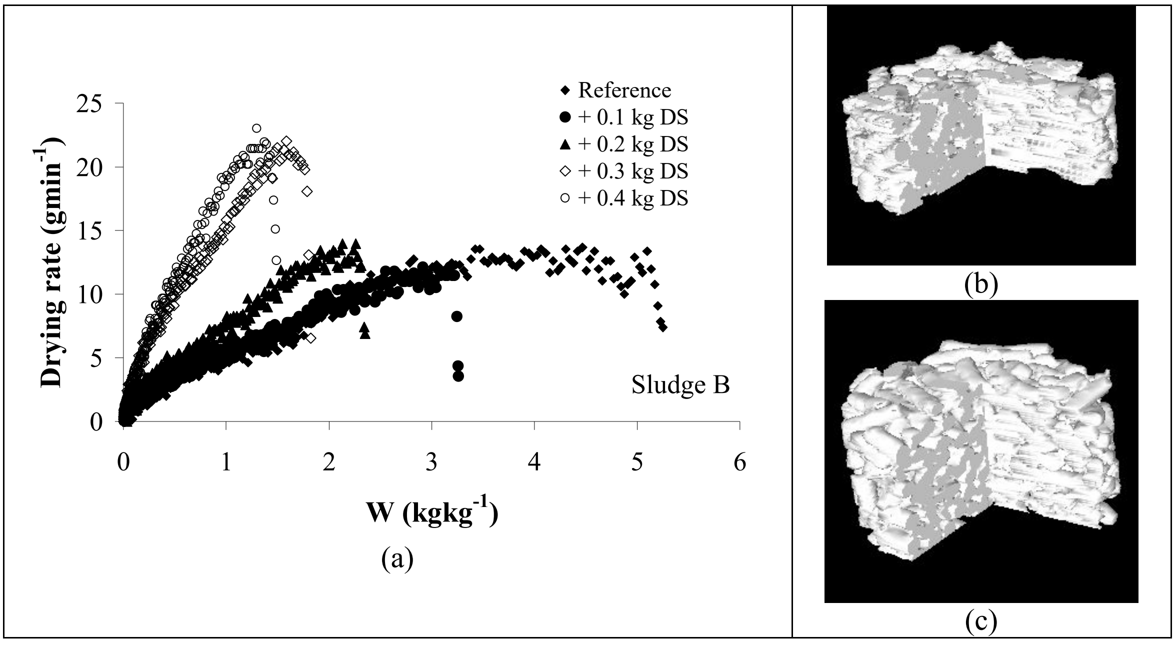


Fig. 5. Comparison of experimental and simulated internal moisture profiles: 80°C, 1m/s (Escalona et al., 2010)

Later on, X-ray macrotomography was used to study the impact of addition of dried product, i.e. backmixing, on sludge convective drying behaviour. Either the addition of dried sludge (Léonard et al., 2008b) or wood saw dust (Li et al., 2017, Li et al., 2016, Li et al., 2015) was investigated at the level of a fixed bed of extrudates of about 1 kg of product. Fig. 6a shows the positive impact of backmixing on sludge drying kinetics, from an addition of 0.3 kg of dry sludge (DS). This addition of dried product increases the permeability and the exchange area of the fixed bed (Fig. 6b and 6c).



(a)

(b)

(c)

Fig. 6. Drying rate for several backmixing levels (a); 3D images of the fixed bed in its initial state (b) and after addition of 0.4 kg DS (c)

Through a collaboration with colleagues of geomechanics, X-ray tomography coupled to drying has been used in the context of better understanding cracking phenomena under forced ventilated conditions. These studies were done in the context of geological storage of nuclear waste (Prime et al., 2016, Prime et al., 2015b, Prime et al., 2015a, Gerard et al., 2010, Hubert et al., 2018). Simulation models were developed in the LAGAMINE finite-element software, with a focus on the mechanical behaviour and the introduction of cracking criteria. Fig. 7 shows 3D views of a clay samples after increasing times. The colours show the evolution of the sample radius at any point on its surface, and therefore the heterogeneity of the shrinkage.

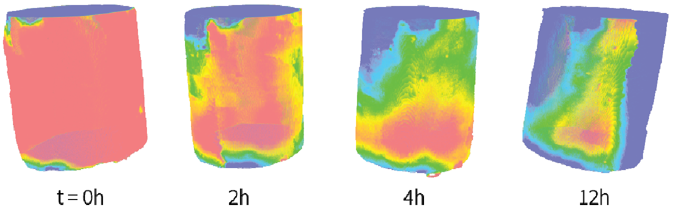


Fig. 7 3D view of a clay sample (1cm height) for increasing drying times

Soil scientists, namely colleagues of the Agro Tech faculty of Gembloux, were also interested by using this technique in order to better apprehend water and air exchange phenomena in the soil, in relation with agricultural practices or dessication/wetting cycles (Tran et al., 2019b, Tran et al., 2019a, Smet et al., 2018b, Smet et al., 2018a, Parvin et al., 2017).

A lot of more punctual collaborations were also made possible thanks to the combination of equipments available, associated with the expertise in image processing and analysis. One can cite studies about banana drying (Léonard et al., 2008a), yeast drying (Debaste et al., 2010), tomato drying (Khama et al., 2016), saw dust bed characterization (Parmentier et al., 2014), paper pulp sludge (Mäkelä et al., 2016). The story still continues, with tests currently done in 2019 on potato drying, in collaboration with a Mexican team.

4. Conclusions

X-ray tomography has been proved to be a powerful non destructive characterization technique with a lot of applications in the field of drying. While the association of tomography with drying was not usual at the end of nineties, the use of this technique has continuously increased since then. In 2018, 23 articles including drying in the title and tomography in the keywords or title or abstracts are referenced in scopus, against 1 in 2000. Hence other interesting studies using this technique can be highlighted: those of the group of Tsotsas in Magdeburg (Szadzińska et al., 2019, Pashminehazar et al., 2019, Pashminehazar et al., 2018, Sondej et al., 2016, Pashminehazar et al., 2016, Sun et al., 2015, Sondej et al., 2015, Wang et al., 2012), Carmeliet in Leuven (Prawiranto et al., 2019, Lal et al., 2018, Lal et al., 2017b, Lal et al., 2017a), Tao in Shanghai (Wu et al., 2010, Xiao et al., 2007b, Xiao et al., 2007a), or Pisano in Torino (Pisano et al., 2017)

In the future, drying facilities built within a tomograph should develop, in order to avoid drying interruptions and go deeper into the understanding of shrinkage, cracking, and other drying related phenomena.

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