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Research paper

Multiscale structure characterization of sawdust-waste water sludge extrudates dried in a pilot-scale fixed bed

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A B S T R A C T

Convective drying of waste water sludge and sawdust-sludge mixtures in a pilot-scale fixed bed was studied. Drying was performed in a cross-flow convective dryer using 500 g of wet material extruded through a disk with circular dies 12 mm in diameter. The structure of the bed mainly shows volume shrinkage and crack formation during drying. Several characterization techniques were used over a wide range of scales from nm up to mm. The overall bed structure was imaged with X-ray macrotomography, at a resolution of 0.36 mm per pixel. Single extrudates were scanned with X-ray microtomography, at a resolution of 41 μm per pixel. Pore structure of the dried samples were characterized by mercury porosimetry (7.5 nm $< d_p <$ 150 μm). Results show significant structural changes on all scales with increasing amounts of sawdust: shrinkage decreases, crack formation increases, and the pores become larger. This confirms the benefits of sawdust addition for sludge drying applications.

1. Introduction

As more waste water sludge is being produced year after year, its efficient disposal becomes increasingly important. The vast majority of sludge is disposed of through gasification and combustion in furnaces, but to do so water content must be lowered by drying, in order to reach a calorific value adapted to such thermochemical conversion process [1]. The amount of energy needed for drying such soft and pasty material is significant, therefore new approaches are sought to speed up the process. Adding sawdust to the sludge is a way of reinforcing the structure and help reduce drying time, thus lowering the amount of energy necessary for drying process [2]. On the basis of a previous study dealing with back mixing [3], where it was found that expansion of the sludge bed enhanced heat and mass transfer, the addition of sawdust in the sludge is a promising approach to achieve such expansion. The choice of sawdust is justified in that it is useful for gasification, and it is produced in large amount by the forest industry and also needs safe disposal solutions [4]. Typically, sawdust is used in the manufacture of compressed biofuels or wood boards [5], but new applications should be explored. A mixing machine can be added to the typical industrial sludge drying setup which consists of a sludge extruder and a belt dryer. The investment cost will slightly increase because of the addition of the mixing machine, but the overall cost may be considerably reduced with the reduction in drying time.

Shrinkage and crack formation are important phenomena in sludge drying: as the sample loses its moisture it exhibits volumetric reduction before the internal stresses exceed the tensile strength and leads to cracking [6,7]. Such structural changes during the drying process affect the paths of heat and mass transfer, which in turn affects the drying characteristics of the material [8]. This inter-coupled process governing the drying mechanism needs to be better understood in order to identify the final sludge properties, and this requires more thorough characterization of the structural changes that occur during drying.

X-ray tomography is a non-destructive 3D imaging technique that can provide such structural information. It works by performing a series of X-ray radiograms of the sample from different viewing angles, and algorithmically reconstructs a 3D X-ray attenuation map [9–11]. This imaging technique has many advantages, and it has applications in many research fields [12–16]. On the study of sludge drying, Léonard et al. [17–19] were the first to use X-ray microtomography for the quantification of volume,

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Abbreviation: WWTP, waste water treatment plant.
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shrinkage, crack, moisture profiles, and other characteristics of sludges. It was shown that different types of sludge exhibit a wide variety of behaviors when dried [17,19,20]. Mechanically dewatered sludge cakes were studied using X-ray microtomography by Tao et al. [21,22], and they found that crack formation can enhance the drying rate but volume shrinkage reduces it. However, their experiments were conducted in a microdrying set-up, i.e. at a small scale (their samples measured 26 mm in diameter), where boundary effects can be predominant. A typical industrial set-up is at a larger scale, with a belt dryer fed from an extrusion system. Our experiment aims to better reproduce this setting, using a pilot-scale fixed bed. The size of the bed (about 160 mm in diameter and 60 mm in height) is such that high X-ray energies and a larger scale of imaging must be used.

The first large-scale X-ray tomograph was built at the University of Liège at the end of the 1990s. Marchot et al. [23] designed a 160 kV fan beam system to image gas–liquid flow patterns in fixed beds filled with plastic packings. In 2005, the same group built the first European high energy (420 kV), large-scale (max. 0.45 m in diameter and max. 4 m in height) X-ray tomograph [24], initially for the same field of study [25–27]. The 420 kV source allows the scanning of highly absorbing materials, such as thin metallic objects, and was used to study the structure of a packed bed during drying with a resolution of 0.36 mm [3]. Three other research groups developed large-scale X-ray tomography systems for chemical engineering applications, namely those of R. B. Eldridge at the University of Texas at Austin [28–30], D. Mewes at the Leibniz University of Hannover [31–33], and T. Heindel at the Iowa State University [34–36]. Such a setup has already been used in the field of drying: Léonard et al. [3] studied the influence of back mixing on convective drying of residual sludges in a pilot-scale fixed bed. The effect of sawdust addition on convective drying of sludge was also investigated, and the shrinkage, diffusion coefficient, and mass transfer coefficient were obtained [2,20].

At a smaller scale, mercury porosimetry has been successfully used for pore space characterization in dried materials [37,38]. Porosity, defined as the ratio of the pore volume to the apparent volume of the material, is strongly dependent on the moisture content and drying conditions. Porosity appears when volume reduction is smaller than the theoretical reduction from water removal [39]. Karathanos et al. [40] used mercury porosimetry for measuring changes during drying of celery. Changes in pore size occurred immediately when drying began and continued until the end, reducing the average pore radius from 4 to 0.5 μm. Characteristics of pores in dried tuna (Thunnus tongol) processed by air-, vacuum-, and freeze-drying were measured with this technique by Rahman et al. [41]. They found that the porosity of freeze-dried samples was much higher than that of the air-dried and vacuum-dried ones.

The multiscale structure of waste water sludges dried in a convective rig was characterized by Léonard et al. [42]. They investigated the structure and porosity of two dried sludge rigs from lab-scale convective drying experiments, using SEM imaging, nitrogen absorption isotherms, mercury porosimetry, and X-ray microtomography. Results showed that both sludges had similar mesoporous and macroporous structures, yet had different shrinkage behaviors and crack ratios. Again, that study was performed at the lab scale, and to better represent the industrial context our present works will focus on samples from a pilot-scale fixed bed with a multiscale characterization and the effect of sawdust addition. The addition of sawdust, although beneficial for the drying characteristics, adds a level of complexity to the drying behavior. The structure of these beds were acquired by X-ray macrotomography (pixel size = 0.36 mm), single extrudates were scanned using X-ray microtomography (pixel size = 41 μm), the pore nanoscale structure of the dried samples were measured by mercury porosimetry ($7.5\ \text{nm} < d_p < 150\ \mu\text{m}$). Nitrogen absorption isotherms ($0.5\ \text{nm} < d_p < 50\ \text{nm}$) was not used in this study as several tests have shown almost no porosity at that scale was found.

2. Materials and methods

2.1. Materials

Activated sludge was collected after mechanical dewatering in a WWTP (Grosses Battes, Belgium) located near the University of Liège. The WWTP is situated in Angleur (Belgium) between the river Ourthe and the channel Ourthe. It is mainly a residential WWTP, and the waste water of this WWTP comes from the following districts: Chênée, Vaux, Grivegnée, Angleur, and Embourg (Belgium). Although there are primary, secondary, and tertiary treatments in this WWTP, the sludge used was from the secondary treatment. The initial moisture content was determined according to standard methods [43]. Before drying, the sludge was stored at a temperature of 4 °C, to preserve drying properties during storage [44]. Table 1 gives the physical and chemical characteristics of the sludge used.

Sawdust was collected in a wood pellet factory ('Industrie du bois', Vielsalm, Belgium), of which 96.5% was Épicéas commun (Picea abies) and 3.5% was Sapin de Douglas (Pseudotsuga menziesii). The average age of the trees is 55 years old and the initial wet basis moisture content of the sawdust was around 30%. Table 2 presents the dry basis elemental and thermal analyses of the sawdust and Table 3 gives the sawdust size distribution.

The drying behavior of several samples was studied: the original sludge, the mixed sludge (the original sludge after mixing without sawdust), and several sawdust-sludge mixtures (the original sludge after mixing with sawdust). A kitchen mixer (KM1000, PROline, Belgium) was used to prepare the mixed sludge and sawdust-sludge mixtures. The mass fraction of sawdust, expressed on a dry matter basis for both sludge and sawdust, were 10%, 20%, 30%, and 40%. In previous studies [3,45,46], it was found that back mixing and liming changed the rheological property of the sludge and the structure of the bed. The cohesion of sludge would decrease with longer mixing times and higher mixing velocities. Consequently, the rigidity of the extrudates also decreased, producing fixed beds with smaller exchange areas, and thus a lower drying rate. For these reasons we have chosen a low mixing rate (40 $\text{r}\cdot\text{min}^{-1}$) and a short mixing time (30 s). The same protocol was used to mix the sludge without sawdust. Before drying, these samples were extruded through a disk with circular dies 12 mm in diameter, forming a bed of extrudates on the dryer perforated grid. The initial mass of the extrudates bed was fixed at 500 g in all experiments. In this way it is assessed that the corresponding industrial belt dryer would operate at constant feeding rate, on a global mass basis.

In addition, a minor portion of sawdust was dried in an oven at 80 °C, in order to perform mercury porosimetry.

2.2. Pilot-scale dryer

Drying experiments were carried out in a discontinuous pilot-scale dryer reproducing most of operating conditions prevailing in a full-scale continuous belt dryer, as shown in Fig. 1. A fan (a) draws in ambient air that is heated to the required temperature by a set of resistors (b). If needed, the air is humidified after heating with a vapor generator. The hot air flows through the sludge extrudates (c) that lie on a perforated grid (d) linked to a scale (e). The inner diameter of the sample holder is 160 mm. Three operating parameters may be controlled: air temperature, superficial velocity,