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# Moisture distribution and hydrodynamics of wet granules during fluidized-bed drying characterized with volumetric electrical capacitance tomography

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ABSTRACT

Fluidized-bed drying of wet granules is a common process in many industrial sectors, and monitoring of the moisture and the hydrodynamics of the granules during drying is important in order to ensure the quality of the final product. Electrical capacitance tomography (ECT) is an electrical imaging modality in which the permittivity distribution inside an object is computed using capacitive measurements from the boundary of the object and mathematical algorithms. Here, three-dimensional moisture distributions were computed through an experimentally determined relationship between granule moisture and relative permittivity. Estimates for the average moisture content were calculated and evaluated. The hydrodynamics of the wet granules were characterized with respect to air velocity and granule moisture.

## 1. Introduction

Wet granulation is a commonly used process in the pharmaceutical, chemical, agricultural and food industries. In wet granulation, powders and binder liquids are mixed in a granulator. After the process, the wet granules are dried, and the drying is usually performed in a fluidized-bed dryer. In the dryer, moisture is transferred from the wet granules to a heated stream of air that is injected through the material. Often, it is desired to obtain granules with a certain moisture content. Furthermore, the hydrodynamics (or the fluid dynamics) of the granules during drying will affect the quality of the final product. For example, a too high superficial gas velocity of the air stream may result in granule attrition and entrainment of fine particles, and a too low velocity in non-homogeneous mixing and inefficient drying.

Traditionally the temperature of the outlet air has been used in monitoring of fluidized-bed drying. However, this is not applicable at high moisture contents and it does not provide information about the hydrodynamics of the process. Other methods include near infra-red (NIR) detectors (Morris et al., 2000), microwave resonance technology (Buschmüller et al., 2008) and triboelectric probes (Portoghese et al., 2008). Even though, these methods are sensitive to moisture, there are also some drawbacks: for example, the methods analyze the moisture in a pointwise manner, i.e. the location of the sensor plays an important role in the measurements, and they do not provide information about the hydrodynamic properties. Furthermore, microwave resonance sensors and triboelectric probes need to be installed inside the product bowl which is not always feasible, and NIR detectors work only if the observation window remains clean.

The most common technique to assess the hydrodynamics of fluidized-beds is pressure measurements, and those have also been used in few studies to investigate the hydrodynamics during drying (Chaplin et al., 2004; de Martin et al., 2011; Vervloet et al., 2010; Wormsbecker and Pugsley, 2008; Wormsbecker et al., 2009). However, it seems that there are several different opinions on the number of the needed pressure sensors, the suitable locations of the sensors, the appropriate data processing methods and the correct interpretations of the results.

In this study, electrical capacitance tomography (ECT) was utilized for monitoring the three-dimensional (3D) moisture distribution of pharmaceutical granules during the drying process. In ECT, the internal permittivity distribution of the target is reconstructed with the help of measured capacitances and mathematical algorithms. Volumetric ECT is a 3D counterpart to the two-dimensional (2D) electrical capacitance tomography.[1]

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[1] Three-dimensional electrical capacitance tomography is sometimes referred to as electrical capacitance volume tomography. Furthermore, if measurements

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The 2D-ECT occasionally suffers from unwanted fringe effects due to the fact that the applied electric field which is 3D by nature is approximated with a 2D electric field. Furthermore, the reconstructed 2D-tomograms utilize the (often unrealistic) assumption that the permittivity distribution is homogeneous with respect to the third spatial dimension. In 3D-ECT, the potential field is treated as being 3D which improves the accuracy of the method, and in addition, since the result is a 3D volumetric tomogram no spatial assumptions need to be made.

For process monitoring purposes, ECT represents an attractive option since it is non-intrusive, non-invasive and it is an in-line technique. The ECT sensing electrodes can be installed around the outer-boundaries of the product bowl, thus the electrodes are not in touch with the processed materials. In-line measurements enable real-time controlling. In addition, the method provides both local pointwise information from the target through the reconstructed tomograms and overall information through characteristic measures that can be computed using the whole tomogram. This means that ECT enables both the characterization of the moisture content and the hydrodynamics of the wet granules.

ECT has previously been used for monitoring of various fluidized-bed experiments (for example, Halow and Nicoletti, 1992; Makkawi and Wright, 2002a; Tanfara et al., 2002; Wang et al., 1995, 2010; Warsito and Fan, 2003; Wiens and Pugsley, 2006), reference techniques have been used to confirm the ECT results (Chaplin et al., 2005; Holland et al., 2009; Pugsley et al., 2003; Wang et al., 2008), and general guidelines on how to carry out the experiments have been proposed (Makkawi and Wright, 2002b, 2004). However, there are fewer studies on ECT monitoring of fluidized-bed drying. Previously, capacitance data related to a packed bed of wet granules have been associated with moisture, and comparisons between the radial density profiles determined from 2D-ECT and x-ray tomograms have been made (Chaplin et al., 2005). *S*-statistic analyses of ECT data and 2D-tomograms have been used to interpret hydrodynamic changes occurring during drying (Chaplin and Pugsley, 2005). Mathematical models and CFD simulations have been compared to ECT results, capacitance measurements related to packed bed and minimum fluidization of wet granules have been associated with granule moisture, and 2D-tomograms illustrating the density of solids have been presented (Wang et al., 2008). An online method for controlling of air flow rate during drying based on ECT measurements and a mathematical model has been demonstrated (Wang et al., 2009). The effects of the excitation signal frequency and the data normalization method to the ECT results during drying have been investigated (Wang and Yang, 2010).

The way this present study differs from the above mentioned ones is that this is the first time when the geometry of the conical product bowl has been properly taken into account and the drying processes are being monitored with 3D tomography to obtain volumetric tomograms of the moisture distributions. The correct geometry and the volumetric tomography are needed because the fluidized-bed processes have a very chaotic nature and therefore it cannot be assumed that the moisture distribution would be homogenous in the vertical spatial direction. Moreover, absolute moisture values are reconstructed rather than normalized permittivity values or densities.

In this study, a Finite Element Method (FEM)-based reconstruction algorithm was developed. Previously, similar FEM-based techniques have been used in Wajman et al. (2006), Soleimani et al. (2009), and Banasiak et al. (2010). In the inverse problem (the imaging problem), the reconstruction algorithm used in this study was based on the difference reconstruction method. Other descriptions of ECT image reconstruction algorithms can be found elsewhere (Soleimani et al., 2009; Wajman et al., 2006; Warsito et al., 2007a; Watzenig and Fox, 2009; Xie et al., 1992; Yang and Peng, 2003).

In this study, the applicability of the volumetric ECT was first examined with simple demonstration tests. Subsequently the relationship between the moisture content of the wet granules as a function of relative permittivity was experimentally determined. Finally, three different drying experiments were conducted, and analyzed with the help of ECT imaging as well as via analysis of small samples taken during the experiments. Various estimates for the average moisture of granules based on ECT data and reconstructed images are presented and compared. Furthermore, the bed hydrodynamics with respect to air velocity and moisture content were characterized with the help of tomograms and normalized moisture curves.

## 2. Theory

This section describes briefly the theory behind the ECT and the general mathematical principles used in the estimation of the permittivity distribution and the moisture distribution with the help of the difference reconstruction method. A more detailed derivation of the difference reconstruction method can be found elsewhere (Rimpiläinen et al., 2011).

### 2.1. Estimation of the permittivity distribution

Here, the computational domain is denoted with $\Omega$ and the permittivity at $\mathbf{x} \in \Omega$ is defined as

$$\epsilon(\mathbf{x}) = \epsilon_{\mathrm{vac}}\epsilon_r(\mathbf{x}), \tag{1}$$

where $\epsilon_{\mathrm{vac}}$ is the vacuum permittivity ($\epsilon_{\mathrm{vac}} \approx 8.8542 \times 10^{-12}$ F m$^{-1}$) and $\epsilon_r(\mathbf{x}) \geq 1$ the relative permittivity of the medium. In the inverse problem (i.e. the imaging problem), a forward model that connects the ECT-measurements with the electric potential distribution $u(\mathbf{x})$ and the electric charges $q_l$ at the electrodes needs to be formulated. First, the potential distribution can be solved from the equation

$$\nabla \cdot \epsilon(\mathbf{x})\nabla u(\mathbf{x}) = 0, \quad \mathbf{x} \in \Omega \tag{2}$$

with the boundary conditions

$$u = 0, \quad \mathbf{x} \in \partial\Omega_{\mathrm{sc}} \bigcup_{l=1}^{N_e} e_l \backslash e_{\mathrm{ex}}, \tag{3}$$

$$u = V, \quad \mathbf{x} \in e_{\mathrm{ex}}, \tag{4}$$

$$\epsilon(\mathbf{x})\frac{\partial u}{\partial \boldsymbol{\nu}} = 0, \quad \mathbf{x} \in \partial\Omega \backslash \left\{ \partial\Omega_{\mathrm{sc}} \bigcup_{l=1}^{N_e} e_l \right\}. \tag{5}$$

Here, $\partial\Omega$ is the boundary of the domain, $\partial\Omega_{\mathrm{sc}}$ denotes the boundaries of the electrically grounded screens, $e_l$ the $l$th electrode, $N_e$ the number of electrodes, $e_{\mathrm{ex}}$ the excitation electrode, $V$ is the excitation voltage, and $\partial u(\mathbf{x})/\partial \boldsymbol{\nu}$ is the derivative of the potential in the direction of the outward unit normal vector $\boldsymbol{\nu}$ (outwards from the electrode surface). The condition (3) is valid at the sensing electrodes and at the electrically grounded screens, and the condition (4) is valid at the excitation electrode. The condition (5) is valid at the boundaries that are not made of metal and therefore are not at any specific potential. The condition means that the electric displacement field is zero in the direction of the unit normal at the boundary.

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(*footnote continued*)
are carried out with respect to time the method is sometimes referred to as four-dimensional (4D) ECT.