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Comparison of near-infrared spectroscopy and DEM simulations for tracing spatial dispersion of water during mixing

Roland Hohl*, Nicolas Heigl, Daniel Koller, Charles A. Radeke

Research Center Pharmaceutical Engineering GmbH, Inffeldgasse 13, 8010 Graz, Austria

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ABSTRACT

The aim of this work was to validate a wet mixing process, in which a liquid spray is used to impregnate particles during mixing. The experimental results obtained using a bladed-mixer with a near-infrared sensor were compared with the results obtained using a 1:1 discrete element method simulation. The porous particles used in both cases absorbed the sprayed liquid for a process time of about 18 min. Multiple sensors attached to the mixer wall continuously monitored the liquid contents of passing particles. The sensors were modeled in the simulation and the resulting signals were analyzed and compared with the experimental results. We show that the algorithms used for spray and liquid absorption can be used to predict the moisture distribution inside granular materials in chemical and pharmaceutical processes. Such simulations can help to save money, e.g., in resource-intensive experimental plans and equipment design studies, and by varying material parameters.

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Introduction

Liquid spraying is frequently used in the processing of active pharmaceutical ingredients and excipients, e.g., during wet granulation, blending, coating, or fluidized bed granulation, in which dispersion of the liquid phase is a critical quality attribute (Corredor, Bu, & Both, 2011). There is a growing tendency to use water as a solvent rather than organic solvents because it is environmentally friendly and eliminates explosion risks; therefore monitoring of the water content has become an important task. Water can have a pronounced impact on the chemical reactivity and stability of a formulation and on the final product quality and shelf life (Corredor et al., 2011; Dalton & Hancock, 1997). Traditionally, Karl–Fischer titration and loss on drying (LOD) are used for residual water detection, but both methods have considerable drawbacks such as long measurement times, invasive analysis, and no on-line capabilities (Roggo et al., 2007).

Good progress, based on various principles, is being made in enabling on-line determination of the water contents of granular materials in the biomass, food, and pharmaceutical industries. A robust method uses capacitance or impedance measurements, based on the high dielectric constant of water (Moura, Berbert,

Berbert-Molina, & Oliveira, 2013; Wang, Senior, Mann, & Yang, 2009). These methods also have the advantage that with some computational effort a tomographic reconstruction (i.e., a soft model) of the water distribution is possible (Beck & Williams, 1996).

The attenuation and phase shift of microwave radiation can be used for moisture determination (Austin, Rodriguez, Sung, & Harris, 2013). Triboelectric probes have also been reported, and are used for determining moisture content during granulation (Portoghese, Berruti, & Briens, 2008).

Near-infrared (NIR) radiation is strongly absorbed by water, giving two distinct absorption bands in the NIR region, i.e., a combination of bending and asymmetric stretching of O–H ($\nu_3 + \nu_2$) from 5100 to 5300 cm^{-1} , and a combination of symmetric and asymmetric stretching of O–H ($\nu_1 + \nu_3$) from 6800 to 7100 cm^{-1} . Numerous NIR-based applications have been reported (Corredor et al., 2011; De Beer et al., 2011; Koller et al., 2011; Mantanus et al., 2010; Rantanen et al., 2000; Reich, 2005).

NIR spectroscopy offers the possibility of fast and real-time measurements, and can be coupled with simultaneous material identification or concentration estimation. However, compared with microwave absorption, NIR radiation has the drawback of a limited penetration depth into the sample, although it enables easy access to sealed environments via fiber optics.

In this study, we used a laboratory-scale stainless steel reactor equipped with a spray nozzle and a four-bladed impeller for moisturizing and blending Al_2O_3 spheres. On-line multipoint NIR

* Corresponding author.

E-mail address: rolandh@sbox.tugraz.at (R. Hohl).

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spectroscopy with fiber-optic probes at different locations was used to monitor water dispersion. Partial least squares regression (PLSR) with LOD as the reference technique was used to develop a linear multivariate model to predict the percentage LOD of the processed Al_2O_3 spheres. Furthermore, discrete element method (DEM) simulation was used for virtual quantification of the amount of fluid sprayed onto the spheres as they passed through the spraying zone. The DEM simulation data were compared with the experimental NIR spectroscopic data.

In contrast to the locally restricted short-range NIR sensor analysis, DEM enables observation of the liquid distribution for each individual particle in a simulation over the whole process time. We used this capability to model and analyze a 1:1 simulation of the mixing device and granules.

Classical DEM simulation tools cannot model liquid spraying and liquid spreading out into powders or particle beds. We therefore used an in-house code with appropriate extensions to model the use of a spray nozzle for continuous wetting of a powder during mixing.

Materials and methods

Experimental part

Al_2O_3

Al_2O_3 spheres (SA 52124, UniSpheres[®]) were purchased from Saint-Gobain NorPro (New Jersey, USA). The median sphere diameter was 1.3 mm, the surface area $5.00\text{ m}^2/\text{g}$, the median pore diameter $0.15\ \mu\text{m}$, the total pore volume (Hg) $0.30\text{ cm}^3/\text{g}$, the packing density $1040\text{ kg}/\text{m}^3$, the chemical impurity $<0.1\%\ \text{SiO}_2$, and the maximum water absorption level 30 wt%. We used $\alpha\text{-Al}_2\text{O}_3$, which is insoluble in acids or bases and has adequate mechanical stability.

Experimental setup

The mixing experiments were performed using a laboratory-scale reactor made of stainless steel (i.d. = 10 cm) with a controllable four-bladed steel impeller (Heidolph RZR 21021; blade diameter = 95 mm, blade width = 25 mm, blade angle = 45°). The rotational

speed was 4 rpm in a counter-clockwise direction. Six different ports, i.e., vision panels made of NIR-penetrable sapphire glass, were integrated to connect single fiber-optic probes (Fig. 1). The blending behavior of this setup has been described by Scheibelhofer, Balak, Koller, and Khinast (2013).

NIR instrumentation

NIR spectra were recorded using an FT-NIR400 spectrometer (PerkinElmer, Brunn am Gebirge, Austria), which was connected to a fiber switch box (FSM2 1×6 , Piezosystem Jena GmbH, Jena, Germany), enabling successive recording of spectra at six different locations. The spectral range was $4100\text{--}10,000\text{ cm}^{-1}$ and the spectral resolution was 16 cm^{-1} . Recording of a full-range spectrum per fiber-optic probe, or channel, was achieved in about 0.35 s. Twelve scans were averaged to gain an adequate signal-to-noise ratio, leading to a total measurement time of 4.2 s per spectrum and channel. Accordingly, the total measurement time for the six successively activated channels was 25.2 s.

NIR calibration

PLSR with LOD (MLS-N moisture analyzer, Kern & Sohn GmbH, Balingen-Frommern, Germany) as a reference method was used to develop a linear multivariate model (SIMCA P+; Umetrics, Umeå, Sweden) for quantification of moisture in the Al_2O_3 spheres. Calibration standards were prepared by filling 100 mL plastic bottles with 20 g of dry Al_2O_3 and adding predefined amounts of distilled water (TKA, MicroPure UV Reinstwassersystem, Niederelbert, Germany). The moisturized samples were then mixed for 2 min to distribute the H_2O equally (Turbula[®] T2F; Willy A. Bachofen GmbH, MuttENZ, Switzerland). The calibration range was limited to 0–26 wt% H_2O because higher moisture contents caused Al_2O_3 agglomeration. The weight percentages of sprayed H_2O and the associated regained percentage LOD values for the calibration standards are listed in Table 1.

In the next step, standard materials were distributed homogeneously on a rotating table (Sensor Turn, WLP-TEC GmbH, Göttingen, Germany), which was turned at 42 rpm. The six fiber-optic probes were fixed on an acrylic glass disk, which was then

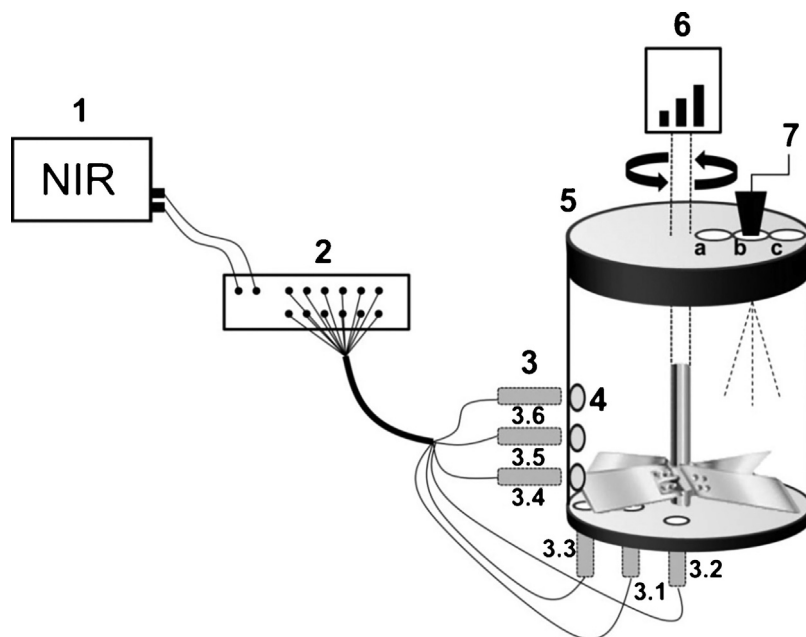


Fig. 1. Schematic diagram of experimental setup: (1) NIR spectrometer, (2) fiber switch box, (3.1–3.6) single-fiber-optic probes, (4) NIR transparent sapphire glass windows, (5) mixing reactor, (6) controllable mixing device with four-bladed impeller, and (7) spray nozzle, which can be installed at three different positions: a: inner, b: middle, and c: outer spray nozzle positions.

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