



In-line monitoring of durum wheat semolina wet agglomeration by near infrared spectroscopy for different water supply conditions and water addition levels



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ABSTRACT

The aim of this work is (i) to study the ability of near infrared (NIR) spectroscopy to follow in-line the agglomeration process of durum wheat semolina using mechanical mixer according to two different water supply conditions (i.e. high versus low water flow rates) and to four different water addition levels, and (ii) to describe the associated structural changes of agglomerates. The results showed that analyses of spectral variations can be useful to discriminate different agglomeration processes, which are associated to different kinetic changes in terms of agglomerate size distribution and water content per size fraction. Principal component analysis (PCA) has been used to qualitatively describe physical and chemical variations occurring during the wet agglomeration mechanisms and to identify specific kinetics and characteristic times. Whatever the water supply condition, kinetic variations of principal components (PCs) on raw and second derivative NIR spectra are observed. Characteristic times have been identified on PC scores and an attempt to link these times to changes of agglomerates physical and chemical properties have been carried out, mainly based on the analysis of associated loading spectra.

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1. Introduction

The wet agglomeration process is used to increase the size of particles by adding a wetting liquid and mechanical or pneumatic energy to promote collisions and adhesion of wetted particles (Iverson et al., 2001). This process is broadly used in a wide range of industrial applications (e.g. pharmaceutical industry, food processing, civil engineering, etc.) and presents many interacting variables, partly responsible for the process complexity. The principal challenge of the wet agglomeration process when conducted inside a mixer bowl, is to correctly disperse water in the powder bed and to ensure homogeneous growth mechanisms in order to obtain wet agglomerates with desired properties. The management of the agglomeration process is still largely conducted based on empirical knowledge and technical know-how of operators, mainly by visual monitoring or sampling procedures. After sampling at different mixing times and/or hydration levels, many physical and hydro-textural characteristics (e.g. particle size, water content, or compactness) of agglomerates can be measured. The off-line measurement methods (e.g. sieving, infrared dryers for water content determination, etc.) are still consuming long time justifying

the recent development of methods for in-line process control (Roggo et al., 2007). In-line monitoring of the agglomeration process has been considered by using specific analytical methods and in situ sensors or probes, such as power and torque measurements to evaluate the cohesion properties of the agglomerated wet mass, near infrared spectroscopy, acoustics and vibration, imaging, microwave resonance technology (Betz et al., 2004; Burggraeve et al., 2012; Holm et al., 2001; Lourenço et al., 2011; Miwa and Makado, 2009; Papp et al., 2008; Watano, 2001).

Near infrared (NIR) spectroscopy appears as a useful non-invasive tool to extract physical and chemical information of food products during an on-going manufacturing process (Aït-Kaddour and Cuq, 2009; Alcalà et al., 2009; Bertrand and Dufour, 2000; Dowell and Maghirang, 2002; Frake et al., 1997; Miller, 1979; Osborne, 2000, 2006; Rantanen and Yliruusi, 1998; Robert et al., 1999; Wellner et al., 2005). Part of the studies about the use of NIR spectroscopy to in-line monitor food processing concerned bread dough mixing. These studies conducted to the determination of NIR mixing curves and the identification of optimum mixing time to produce the best dough development (Aït-Kaddour et al., 2007; Alava et al., 2001; Huang et al., 2001; Millar et al., 2000; Olewnik et al., 2004; Psotka et al., 1999; Wesley et al., 1998). Works carried out to evaluate the ability of NIR spectroscopy to in-line monitor powder agglomeration mainly concern the pharmaceutical

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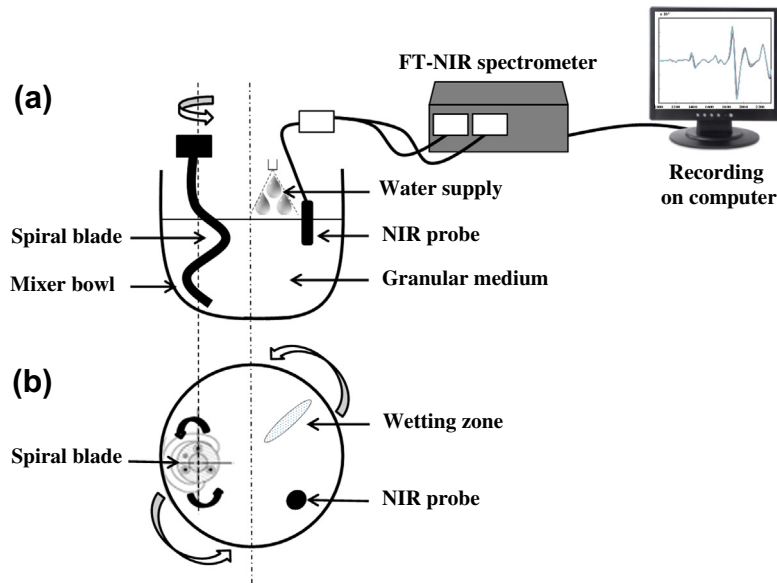


Fig. 1. Schematic representation of the experimental set-up used for the in-line monitoring of the wet agglomeration of durum wheat semolina by NIR spectroscopy.

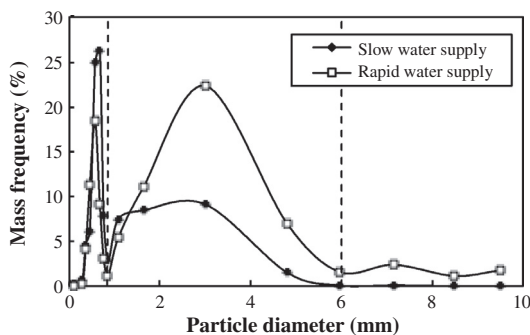


Fig. 2. Diameter distribution for the agglomerates produced by two different conditions of water supply (i.e. low or high water flow rates) during wet agglomeration of durum wheat semolina, at 40% final mean water content, for 2 min of mixing after the end of water supply. The dotted lines represent the diameter transition between identified size fractions.

allow to successfully deconvolute physical and chemical information present in the NIR spectra, even if it still remains a difficult and challenging task (Nieuwmeyer et al., 2007).

Near infrared (NIR) spectroscopy is a useful and cost effective method well adapted for wheat products, to analyze raw materials, ingredients, and processes (Aït-Kaddour and Cuq, 2011). The applications of NIR spectroscopy mainly concern three domains. (i) Rapid prediction of composition parameters and properties of raw materials using consolidated NIR databases. (ii) Screening method in plant-breeding for the selection of cross-breeds with the desired

applications (Alcalà et al., 2009; Findlay et al., 2004; Frake et al., 1997; Nieuwmeyer et al., 2007; Rantanen and Yliruusi, 1998; Rantanen et al., 2000, 2005). Frake et al. (1997) demonstrated that NIR spectroscopy could be useful to follow in-line the increase in water content and particularly, in size of particles during a granulation stage in fluidized bed, by plotting the absorbance at 2282 nm versus process time. Alcalà et al. (2009) developed a quantitative calibration model based on partial least squares regression methodology to predict relevant parameters (such as water content, particle size distribution, or bulk density) of a fluidized bed wet granulation process of pharmaceutical powders. Only few studies have still been conducted to in-line monitor the agglomeration process for food powders applications. Aït-Kaddour and Cuq (2009) demonstrated the high potential of NIR spectroscopy to follow in-line the wet agglomeration process of wheat-based food powders. This study reveals the possibility of NIR spectroscopy to describe physical and physico-chemical changes occurring during the mixing stage of the process (after rapid water addition), through agglomeration or breakage mechanisms. It can be noted that most studies analyzing NIR spectra use chemometric methods, such as principal component analysis (PCA) or partial least squares regression (PLS-R). Multivariate data analyses of the NIR spectrum

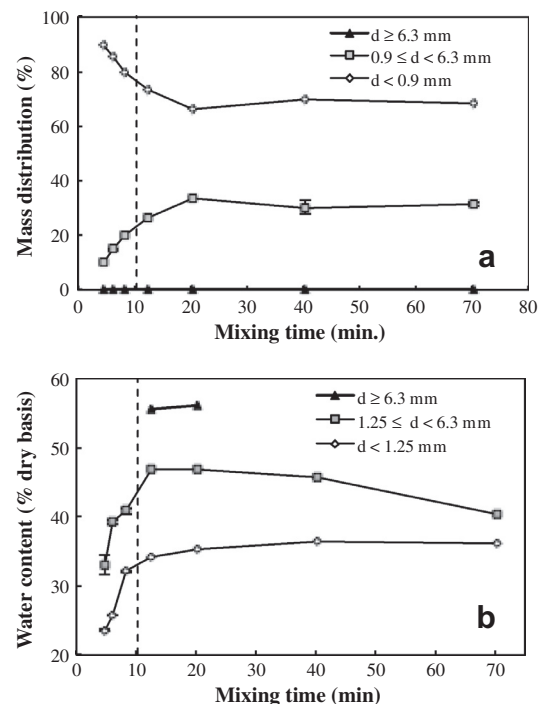


Fig. 3. Changes in the mass proportion (a) and water content (b) of the three size populations (fine, medium, and coarse) during wet agglomeration of durum wheat semolina under low water flow rate condition, at 40% final water content. The vertical dotted lines represent the end of the water supply period.

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