Powder Technology 278 (2015) 130–137

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Mixing quality of powder-liquid mixtures studied by near infrared spectroscopy and colorimetry

Katarina Slettengren *, Peggy Heunemann, Olivia Knuchel, Erich J. Windhab

Laboratory of Food Process Engineering, Institute of Food, Nutrition and Health, ETH Zurich, Switzerland

ARTICLE INFO

Article history:
Received 23 October 2014
Received in revised form 10 February 2015
Accepted 14 March 2015
Available online 23 March 2015

Keywords:
Powder-liquid
Powder mixing
Mixing quality
Near infrared spectroscopy
Colorimetry

ABSTRACT

In many industries, mixing powder with a low amount of liquid is a frequently used process step, *e.g.* in order to generate agglomerate/granule product structures or for sintering and extrusion of wet powders or pastes. There are still great difficulties in achieving a homogeneous mixture due to local agglomeration and lump formation. A new mixing methodology has therefore been developed, in which the liquid is transformed into a solid powder by spray chilling, generating so-called powder-liquids. These powder-liquids are then mixed with other dry powders.

An even liquid distribution, as well as the related even distribution of functional components in the final product is of high relevance. We thus aimed to evaluate the mixing quality of such powder/powder-liquid mixtures studied by near infrared (NIR) spectroscopy and colorimetry. For this purpose we quantified the homogeneity of fat based powder-liquids mixed with flour. NIRS is a well-recognized quantification method that successfully has been used to analyze powder mixtures before, while colorimetry is a method less used for such an application. The results demonstrated that both methods are applicable for quantifying the mixing behavior of such powder mixtures. We showed that cheap, easy and fast colorimetry is as reliable as NIRS measurements and hence can be a first choice validation method in industry. Comparing the powder-liquid mixing with spraying the liquid directly into the powders, the powder-liquids mixed faster and resulted in better homogeneity. Our results support the use of powder-liquids to process new highly homogeneous products required in functional foods or pharmaceutical products.

1. Introduction

Mixing powder with a small fraction of liquid is an industrially applied process step in many industries, such as the food, chemical, pharmaceutical, and ceramics industries [1]. The main task is to add and uniformly distribute the liquid within the powder during the shortest possible mixing time. This is not a trivial task. Inhomogeneity due to liquid enriched lump formation can hardly be avoided in particular for hygroscopic powder systems. There are different ways to add liquid to a powder, such as atomizing the liquid and mix the spray drops with the powder particles under turbulent mixing conditions. De-agglomeration is necessary, either by mechanical treatment using delumpers [2], or by adding more liquid. High-energy input by mechanical treatments may cause losses in functionality for sensitive materials. While, adding more liquid to the mixture in turn needs an additional time- and energy-consuming drying step, in case a low liquid content has to be reached in the final product [3]. A quite recent approach for homogeneous admixtures was given by Windhab [4]. This method allows to add low fractions of liquid to powders, by transforming the liquid into a solid powder by spray chilling prior to mixing, forming so-called powder-liquids to be mixed with the other powder.

Powder characteristics, such as particle size, shape and density, influence the flow properties of powder and powder-liquids and thus their mixture-homogeneity. Hence, in order to characterize the quality of a mixture, a suitable measure has to be fixed. Sommer [5] introduced a definition of mixing quality by how much a measured property X_i, such as concentration, deviates from the ideal value P. For a finite number of samples k, this is indicated by the empirical variance s^2 (Eqs. (1), (2)).

$$s^2(X) = \frac{1}{k}\sum_{i=1}^{k}(X_i - P)^2 \tag{1}$$

$$s^2(X) = \frac{1}{k}\sum_{i=1}^{k}(X_i - \overline{X})^2 \tag{2}$$

* Corresponding author at: Schmelzbergstrasse 9, 8092 Zurich, Switzerland. Tel.: +41 44 632 85 36.
E-mail address: katarina.slettengren@hest.ethz.ch (K. Slettengren).

http://dx.doi.org/10.1016/j.powtec.2015.03.020
0032-5910/

According to Eq. (2) the mean value $\overline{X} = \frac{1}{k}\sum_{i=1}^{k} X_i$ is used, if the nominal value P is unknown [5,6,7]. Furthermore, the variation coefficient CoV can be calculated as the ratio of the empirical standard deviation s and the ideal value P, or the mean value $\overline{X}$, giving a relative deviation of the mixing efficiency (Eq. (3)) [8,9]. The variation coefficient is thus a good measure for comparing mixtures of different concentrations.

$$CoV = \frac{s}{\overline{X}} \tag{3}$$

With a defined measure of mixing quality, there are various techniques that can be used to analyze powder mixtures in respect to their mixing homogeneity. Near Infrared Spectroscopy (NIRS) is a fast, non-invasive method used widely, such as in food, feed, pharmaceutical and chemical products. It has previously been successfully demonstrated for determining the mixing quality of a sugar, cocoa powder component mixture used in cold-extrusion of chocolate [3], for determining qualitative [10,11] and quantitative [12] models predicting blend homogeneity of pharmaceutical powder blends; as well as determining on-line monitoring of blend processes [13].

In case of colored components, reflection measurements can also be applied [2]. In literature, there are several reports on using image analysis to assess the mixing quality of binary or multicomponent mixtures [14–19]. However, not much is reported on using simple colorimetry. With colorimetry the reflection or transmission of light from an object is measured in order to give a numerical description of its color. For manufacturers producing colored products, color measurement is already an essential tool in quality control [20]. In this work the application of colorimetry is further extended to the quality control of binary powder mixtures and compared to NIRS measurements. The aim is to evaluate the analysis methods as well as the mixing performance of the previously mentioned powder-liquid mixing process.

2. Material and methods

2.1. Materials

Powder-liquids were prepared from palm stearin fat (Florin AG, Switzerland) in a spray-chilling process. Palm stearin fat was used as a material for the powder-liquids due to its high melting temperature (40–60 °C) and thus being a stable solid at ambient temperature. Powder/powder-mixing trials of powder-liquid with wheat flour (type 400, SwissMill, Switzerland) were studied by near-infrared spectroscopy (NIRS) and colorimetry respectively for a detailed evaluation of the two methods. (i) Powder/powder and (ii) liquid/powder-mixing trials of powder-liquid with biscuit flour (type 10, SwissMill, Switzerland) or analogously sunflower oil (Florin AG, Switzerland) sprayed into biscuit flour were analyzed by colorimetry for a detailed evaluation of the two different mixing-procedures (i/ii). The colorimetric measurements required a coloring of fat and oil (palm stearin fat, sunflower oil) with a lipophilic magenta dye CY1[1] [21], 0.3 wt.‰ respectively in fat or oil prior to admixture. As all stated mixing experiments were carried out at ambient temperature conditions, sunflower oil with a melting temperature of about −17 °C was used as sprayed liquid, analogous to the palm stearin fat powder-liquids. The differences in fatty acid composition of palm stearin fat and sunflower oil; and their influence on the material (fat/oil) physical properties are given in Table 1.

[1] 1-Ethyl-2-[3-(1-ethyl-1,3-dihydro-3,3-dimethyl-2H-indol-2-ylidene)-1-propenyl]-3,3-dimethyl-3H-indolium perchlorate.

Table 1
Material properties of the palm stearin fat, sunflower oil, wheat flour type 400, and biscuit flour type 10, used for the mixing trials. Data was taken from the product sheets of each material coming from either Florin AG or SwissMill.

	Palm stearin fat	Sunflower oil
Palmatic acid C16:0 (%)	78–85	5.5–7.0
Stearic acid C18:0 (%)	3–6	3.0–5.0
Oleic acid C18:1 (%)	9–13	22.0–32.0
Linoleic acid C18:2 (%)	1–3	56.0–66.0
Solid fat content, 20 °C (%)	85–90	–
Melting point (°C)	40–60	−(15–20)
Viscosity (mPas)	107	49
	Wheat 400	**Biscuit 10**
Fat (g)	1.1	1.4
Carbohydrates (g)	71.0	72.5
Protein (wt.%)	11.5–12.5	10.0–12.0
Moisture content (wt.%)	13.5–15.0	12.5–14.0

2.2. Sample preparation

2.2.1. Coloring of fat and oil

As palm stearin fat is a solid at room temperature it first was melted at 80 °C. The sunflower oil is a liquid at room temperature. Both melted palm stearin fat (80 °C) and sunflower oil (22 °C) were colored with the lipophilic magenta dye CY1 (absorption spectra is stated in Appendix A). The dye was dissolved in 99.94 wt.% ethanol before adding 5 wt.% of the dye solution to the melted fat and oil, giving a total amount of 0.3 wt.‰ dye in either fat or oil.

2.2.2. Powder-liquid production

The melted and colored palm stearin fat was spray-chilled into a cooling tower with a height of 3.7 m and a diameter of 1 m, built at the Laboratory of Food Process Engineering, ETH Zurich. The temperature in the cooling tower was regulated by spraying in and evaporating liquid nitrogen. For the studies presented two batches of powder liquids were produced, one for the method evaluation (Batch 1) and the other for the mixing procedure evaluation (Batch 2). The melted palm stearin fat was sprayed with an air-assist atomizing nozzle with external mixing characteristics (fluid cap PF2850DF-SS: 0.7 mm, air cap PA120-SS: 3 mm, Spraying Systems Co., USA). The volumetric flow rate of the melted fat was set either to 2.3 or 2.7 mL/s. The atomizing pressure was adjusted to 4.6 or 4.1 bar. A detailed overview of the process parameters for the two studies can be found in Table 2.

2.2.3. Method evaluation (powder/powder-mixing)

The mixtures were prepared in a DFML 20 powder-speedmixer (Bühler AG, Uzwil, Switzerland) with a filling volume of 20 L. 5, 10, 15, and 20 wt.% of sieved powder-liquid (for decompaction reasons) was given into the wheat flour. Each mixture was produced only once for the two powder-liquid fractions (PL $x_{50,3}$). The powder mixtures were analyzed by NIRS and colorimetry for different mixing times (0–1800 s), respectively. At every selected mixing time interval, 4 samples of 50 g each were systematically taken (Fig. 1a) with a thief probe, measured five times (Fig. 1b) and thus resulting in $k = 20$

Table 2
Process parameters in spray chilling of powder-liquid from palm stearin fat.

Batch	Experiments	Feed rate (mL/s)	Atomizing pressure (bar)
1	Method-evaluation	2.3	4.1/4.6
2	Mixing-evaluation	2.7	4.1