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A simple color concentration measurement technique for powders

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ABSTRACT

Looking for a way to measure residence time distributions of an 80 micron fluidized cracking catalyst (FCC) powder, a simple measurement technique was discovered that quantifies tracer color concentration. Using a color spectrophotometer that measures percent reflectance as a function of wavelength, a calibration curve can be constructed for standard mixtures of dyed and un-dyed powder. This calibration curve can then be used to determine the color concentration of an unknown sample by measuring its reflectance. The effects of operating parameters such as dye strength, aperture size, surface roughness, sample volume and depth, and continuous flow were all evaluated. This spectrophotometric technique was found to be a quick and simple way to measure colored mixture concentrations. In addition to being ideal for residence time distribution applications, it has the potential to easily quantify mixing in any unit operation, batch or continuous.

1. Introduction

Powder processing is prevalent in many industries, and is embodied in various unit operations [1–3]. Often, it is desirable to characterize a process via the addition of a tracer. Concentration of the tracer is usually the desired measurement in order to determine process characteristics such as residence time, residence time distribution, axial dispersion, and mixing [4–6]. A tracer can be a portion of the bulk material that is dyed, or it can be an entirely different material. Unless the system is inherently multicomponent, it is ideal to dye a portion of the original material as a tracer. Using the same material reduces potential inconsistencies that may arise due to differing material and flow properties of the bulk powder and tracer. Nonetheless, it is important to ensure that dyeing does not affect particle properties.

The motivation for this work stems from the desire to measure residence time distributions of a powder in a rotary calciner, following a similar protocol that Gao et al. used for millimetric particles [7]. These researchers measured color concentration by visually counting the number of dyed particles in images, which is not practical for the greater numbers of particles that arise with a fine powder. As the particle size decreases, individual particles become more difficult to distinguish from one another, making image analysis exceedingly challenging. In order for colored tracers of various particle sizes to be successfully implemented, there needs to be a robust way to characterize the concentration of tracer. The existing methods of measuring colored tracer concentration include image analysis and color spectrophotometry, which are detailed in the following sections.

1.1. Image analysis

Image analysis for color concentration determination typically involves taking images of the multicolored particulate mixtures with a camera, and performing various post-processing steps on the images to extract the solid fraction. Although the particles are colored, most image acquisition and analysis is in black and white. Through image analysis, some researchers correlated gray scale values to tracer concentration [8,9]. Grasa and Abanades found a logarithmic relationship between the gray scale values in an image to the solid concentration, using white 0.85 mm PVC particles mixed with 0.5 mm coal in a fluidized bed [8]. Realpe and Velazquez correlated image gray scale values with powder concentration of different binary combinations of lactose, chocolate, and cellulose particles 44–90 μm in size in static powder beds [9]. With this method, they produced calibration curve fits by second, third, and fifth order polynomials, depending on the powder combination, all with R^2 values above 0.995. Other authors used image analysis to asses mixing by taking images from a camera placed above a continuous conveyor belt setup containing the outflow of a mixer [10,11]. Muerza et al. studied a binary system of white aspirin and yellow semolina, and analyzed the images via an auto-correlation method [10]. Berthiaux et al. implemented principal component analysis to

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determine a homogeneity ratio from images of white and black semolina particles 100–250 μm in size [11].

Some authors have explored the acquisition and subsequent analysis of images with color, with the analysis performed in RGB (red, green, and blue) color space. Aissa et al. pointed out that gray scale image analysis cannot be implemented for a mixture of more than two colored components due to overlap, resulting in the necessity for RGB image analysis [12]. They studied the mixing of red, yellow, blue, and white linear medium density polyethylene powders 83–550 μm in diameter in a rotating cylinder, analyzing the RGB images via multivariate image analysis of the pixel color intensities. Also implementing RGB analysis, the SolidSizer (JM Canty Inc., Buffalo, NY) measures size, shape, and color characteristics from images it takes of a stream of falling particles [13]. Using this instrument, Langroudi et al. studied white particles with a median size of 453 μm, and dyed some of these particles red to create the tracer. Here, the authors were able to distinguish the red particles as those having a larger R value relative to the sum of all of the color values (i.e., R + G + B, the total brightness). They measured tracer concentration as a function of time, allowing for the visualization of the residence time distribution of an axial flow Couette device [13].

Although the aforementioned researchers were successful in using image analysis, they employed many intensive post-processing and analysis steps. Attempting to replicate these methods is often not straightforward and cannot be accomplished simply by reading the relevant manuscript, resulting in time consuming efforts that may not be fruitful. Besides the image processing procedures, environmental factors such as lighting must be just right in order to capture quality images in the first place. Another point to consider is that, in each case, specific protocols were developed for a given particulate system that may not work for formulations outside the studied particle size range. Thus, there is not a single image analysis procedure that can be successfully applied in any situation.

1.2. Color spectrophotometry

Color spectrophotometers, or colorimeters, are instruments that shine light on a sample and measure reflectance as a function of wavelength, and usually report color information in CIE color space. Most users tend to work with the CIELAB (Cartesian coordinates) or CIELCH (cylindrical coordinates) color information, where L^* characterizes white and black, a^* characterizes red and green, b^* characterizes yellow and blue, C^* characterizes hue intensity, and h characterizes hue angle [14–17]. CIELCH can be obtained from CIELAB via a mathematical transformation [17]. The spectrophotometry/CIE method seems to be employed often in the food and agricultural industries, where color can indicate freshness, ripeness, and in general whether or not a substance is visually pleasing [14,16].

As a direct measurement of substance color, McCaig measured $L^*a^*b^*$ values of 50 different particulate food samples using two different categories of instruments, colorimeters/spectrophotometers and visible near-infrared (VNIR) reflectance instruments [14]. They used a tristimulus colorimeter (CR-310, Minolta Canada Inc., Mississauga, ON) with a granular material attachment that extracts color information from a 50 mm diameter circle of the sample, as well as two hand-held spectrophotometers (CM-525i Minolta Canada Inc., Mississauga, ON), which use a 25 mm diameter circle of the sample. McCaig compared these direct $L^*a^*b^*$ color measurements to those calculated from spectra taken from the VNIR instruments. Three different VNIR NIRSystems 6500 instruments (Foss North America, Eden Prairie, MN) were used, which measure reflectance of a 36 mm diameter circle sample in the 400–2499 nm wavelength range. Reflectance measurements in the 400 to 780 nm wavelength range were used in the calculation of $L^*a^*b^*$ values from the VNIR data. In comparing these $L^*a^*b^*$ values to those directly obtained from the spectrophotometers, McCaig concluded that both types of instruments produce similar $L^*a^*b^*$ values. While colorimeters directly provide $L^*a^*b^*$ values, VNIR requires time consuming manipulations to extract these values, but VNIR has the added advantage of providing chemical information from the NIR spectra.

A few researchers used spectrophotometers to characterize powder mixtures [15–17]. Slettengren et al. used a portable tri-stimulus colorimeter (Chroma-Meter CR-300, Minolta AG, Dietikon, Switzerland) which analyzes an 8 mm diameter circle of the sample [15]. They used the a^* values to calculate the coefficient of variation in order to assess mixing quality of powder–powder and powder–liquid systems. These systems comprised various combinations of palm stearin fat, sunflower oil, and two different types of flour, with particle sizes in the 1–200 μm range.

Shenoy et al. applied the DigiEye (VeriVide Ltd., UK) digital color imaging system to assess mixing in food powders, examining binary mixtures of different combinations of salt (454 μm), black pepper (369 μm), paprika (252 μm), and onion (65 μm) [16]. This instrument has the capability to measure color in $L^*a^*b^*$, as well as reflectance as a function of wavelength in the 400–700 nm range. From the $L^*a^*b^*$ values, the researchers calculated ΔE, which is the color value relative to white space, and characterized mixing quality by the variance in ΔE. However, they found that ΔE could not distinguish color concentrations above a certain color threshold, which was dependent upon the specific binary mixture (e.g., the ΔE for onion–salt increased until 30% onion, above which there was no relationship).

Barling et al. used color tracers to assess mixing of pharmaceutical dry powder inhaler blends using CIELCH color space [17]. They studied blends of 1% sub-micronized iron oxide (red) and various grades of 99% lactose (white) in seven different mixers. This low amount of tracer did not appear to impact particle size distributions of the lactose. In each experiment, the authors sampled 5 g of material at different time points, and measured color with a colorimeter (ColorFlex EZ 45/0, HunterLab Inc., U.S.A.). Four measurements were taken of the sample at different rotations of a 6 cm diameter by 3.5 cm high sample cup and averaged. The authors found hue intensity (C^*) to identify dispersion and hue angle (h) to identify de-agglomeration in powder mixing, and developed mixing curves based on these coordinates. From this information, a given formulation's mixing curve can be constructed, which can be used to assess mixing degree.

Although the above researchers used spectrophotometers to measure color using the CIE system, we could not find any literature reporting the use of the raw spectral data, reflectance as a function of wavelength. Our interest in this spectral data stems from the desire to measure color concentration. With reflectance as a function of wavelength for standard tracer concentrations, a calibration curve can be constructed at a given wavelength. With a calibration curve of tracer concentration versus reflectance, the reflectance of unknown samples can be measured with the spectrophotometer, and their color concentrations determined. Thus, the technical gap that must be addressed is the analysis of spectral data from a color spectrophotometer for powder color concentration determination.

In this paper, we present a straightforward way to measure color concentration of a powder mixture via spectral data from a spectrophotometer, which has not been demonstrated previously. Testing all of the different operational modes of a color spectrophotometer, we present our findings on the use of the fundamental spectral measurements, make recommendations on the configurations that provide optimal calibration curve fits, and apply the technique to an axial mixing experiment.

2. Materials and methods

2.1. Sample preparation

The material used in this work is a fluidized cracking catalyst (FCC) powder supplied by Grace Davison (Columbia, MD). The tracer powder is created by dying the FCC powder with Sharpie ink. First, the top of the