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Using response surface methodology to optimize the operating parameters in a top-spray fluidized bed coating system

Seyed Hadi Seyedin[a], Mehdi Ardjmand[b], Ali Akbar Safekordi[a], Shahram Raygan[c], Ehsan Zhalehrajabi[d,*], Nejat Rahmanian[e]

[a] *Department of Chemical Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran*
[b] *Department of Chemical Engineering, South Tehran brAnch, Islamic Azad University, Tehran, Iran*
[c] *School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran*
[d] *Department of Chemical Engineering, Universiti Teknologi PETRONAS, Bandar Seri Iskandar, Perak, Malaysia*
[e] *Chemical Engineering, Faculty of Engineering and Informatics, University of Bradford, UK*

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ABSTRACT

The fluidized bed coating system is a conventional process of particles coating in various industries. In this work, an experimental investigation was conducted using Response Surface Methodology (RSM) to optimize the coating mass of particles in a top-spray fluidized bed coating. The design of experiments (DOEs) is a useful tool for controlling and optimization of products in industry. Thus, DOE was conducted using MINITAB software, version 16. This process used a sodium silicate solution for coating the sodium percarbonate particles. The effect of the fluidization air flow rate, atomization air flow rate and liquid flow rate on the coating mass in the top-spray fluidized bed coating was investigated. The experimental results indicated that the coating mass of particles is directly proportional to the liquid flow rate of the coating solution and inversely proportional to the air flow rate. It was demonstrated that the flow rate of the coating solution had the greatest influence on the coating efficiency.

1. Introduction

Fluidized bed technology is a common process step in the chemical, agricultural, pharmaceutical and food industries. The coating of solid particles, among others, is used to achieve a variety of functions, including the controlled release of drugs, protection of the core from external conditions, taste or odor masking, easier powder handling, and greater stability [1–3]. In addition, the fluid bed coating technique has been used in drug delivery systems [4]. Fluidized beds are divided into top-spray, tangential spray, bottom spray and the Wurster system (this is used more in the pharmaceutical industry). The coating of high-quality particles is often done using the top-spray system with a circulating fluid (see Fig. 1). In this system, a cylinder of appropriate size is placed in the center of the bed. The system forces the particles to circulate in the bed, which facilitates an even distribution of the wetting and drying time between particles [5,6]. In the top-spray fluidized bed coating, the coating solution is sprayed onto the surface of the fluidizing particles by a nozzle which is placed above the bed. The coating solution that is adhesive on the particle surface is dried due to the heat and vapor removal supplied by the fluidizing gas. The advantage of this coating technique is that the coating and drying take place simultaneously. Moreover, it can be used to coat particles in the size range of 0.1 to several millimeters [7]. The product quality and the process efficiency are largely determined by the spray and bed characteristics, and the particle motion characteristics in terms of the residence and circulation times [8,9]. For spraying liquid droplets in a fluid bed, generally, pneumatic two-fluid nozzles are used. In these nozzles, the liquid jet is changed to droplets by the air flow. Using pneumatic nozzles helps to control the size and distribution of the droplets, particularly when the liquid flow rate is low. The droplets sprayed by the pneumatic two-fluid nozzles are smaller than those using single fluid nozzles. The important aspects in controlling the fluidized bed coating process include controlling the product temperature and the growth of the coating film. Applying a fluidized bed coating to heat sensitive products, such as enzymes, proteins or microorganisms, can protect them from environmental damage [1,10–12].

The tools and techniques used in the DOE have proven successful in meeting the challenge of continuous improvement in many manufacturing organizations over the last two decades. However, research has shown that the application of this powerful technique in many

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* Corresponding author.
E-mail addresses: ehsan_g02908@utp.edu.my, e.zhaleh@gmail.com (E. Zhalehrajabi).

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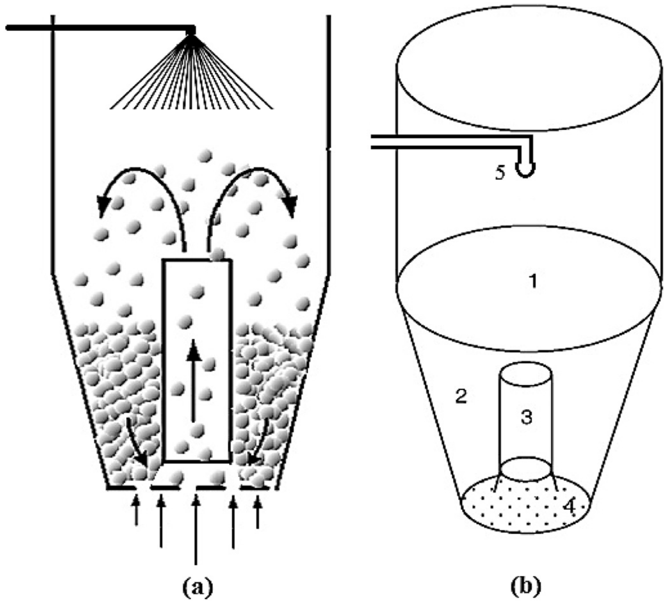

Fig. 1. a) Schematic image of a top-spray system with circulating fluid. b) Different parts of the bed: 1. Expansion chamber (deceleration region, fountain), 2. Down bed region, 3. Draft tube (cylinder), 4. Air distributor plate, 5. Nozzle.

companies is limited due to the lack of statistical knowledge required for its effective implementation [13]. Within the DOE approach, initially a target value or set of target values has to be defined for specific parameters, which is referred to as the response. To become acquainted with a process, in general, DOE uses a screening design to analyze the effect of several influencing variables on the response and to separate the main influencing parameters from those that only have a minor impact. A further step within the DOE approach is the response surface methodology, which investigates the local and global optima of the process and identifies the relevant interactions between the influencing variables [14]. DOE is one of the key elements of the quality by design principles that have been used to study fluidized bed coating process. The use of DOE allows for testing a large number of factors, as well as their interactions simultaneously. The response surface methodology (RSM) is one of the popular methods in DOE, which involves the use of different types of experimental design to generate polynomial mathematical relationships and to map the response over the experimental domain to select the optimal process parameters [15–17]. An efficient way of planning and optimizing such experiments involves the principles of DOE. The key property of DOE is that while several factors are varied simultaneously, each factor may be evaluated independently. The simplest DOE are often factorial experiments, where all factors are varied simultaneously at a limited number of factor levels. More complex DOEs involve response surface designs. Central Composite Designs (CCD) are possibly the most popular type of response surface designs [18]. If the factors act additively, the DOE design does the job with much more precision than one-factor-at-a-time methods, and if the factors do not act additively, the DOE, unlike the one-factor-at-a-time design, can detect and estimate the interactions that measure this non-additivity. Hence, an advantage of DOE is that it allows for the maximum amount of information to be extracted using the minimum number of experiments. DOE is also useful as it allows for straightforward handling of experimental errors and allows for data extrapolation [19]. The top-spray fluid bed system is used for particles coating. In this process, coating materials are sprayed onto the fluidized particles, and the liquid reacts with fluid particles or covers their surface in the bed [20]. The uniform distribution of the coat in the fluid bed depends on the type of coating liquid, size and type of particles being coated, fluidization air flow, and nozzle spray type [21,22]. In the fluidized bed coating, agglomeration is an undesirable phenomenon, and a number of studies have focused on reducing it [23,24]. Agglomeration is mostly used for pelletizing and granulation in the pharmaceutical industry [25]. In recent years, some work in the field of mathematical modelling and the simulation of hydrodynamics, heat and mass transfer in fluidized beds, and the droplet deposition behavior and dynamic particle populations in the granulation process have been conducted [8,26–29]. Some studies have also addressed the optimization of various process parameters for the formulation and scaling parameters in fluid beds [30,31]. Recently, statistical optimization was employed in the various processes because it quickly screens a number of multiple parameters and their interactions, and reflects the function of an individual factor or component [32].In the present study, sodium percarbonate particles are coated by sodium silicate in a top-spray fluidized bed system. The protective silicate layer protects laundry detergent powder particles from humidity and corrosion and conserves the available oxygen in the powder, where oxygen has a bleaching role in the powder. The aim of this study is to recommend an established statistical methodology to optimize the process conditions, such as the fluidization air flow rate, liquid flow rate, and the atomization air flow rate to enhance the coating mass using the RSM experimental design.

1.1. Materials and method

Sodium percarbonate powder (SPC) was supplied from the Solvay company (OXYPER®, general grade, and available oxygen is minimum 30%). SPC particles were fractionated by sieving. The mean diameter of the particles (grade dependent) was in the range of 450 to 500 μm. For each coating experiment, 100 g of SPC was used. Sodium silicate (SS) solution was used as the coating solution with 30% volumetric concentration and diluted in water (300 ml liquid sodium silicate was dissolved in 700 ml water). The chemical composition of the applied materials is summarized in Table 1.

2. Equipment

A Plexiglas fluidized bed chamber was used for the coating process. For spraying of the coating solution, a pneumatic nozzle was placed in the top of the bed. After the spraying process, the coated particles were dried in the bed. The process variables were the atomization air flow rate due to controlling the spray droplet size, the fluidization air flow rate, and the liquid flow rate of the coating solution.

2.1. Description of the coating process

The top-spray fluid bed coating system used in the present study is shown in Fig. 2. As shown in Fig. 2, the shape of the bed is cylindrical, the bed height is 450 mm, and the bed diameter is 170 mm. There is a small cylinder (draft tube with 70 mm width and 100 mm height) in the center of the bed, which provides a circular flow of particles for a better coating process. Also, there are three sensors for controlling the temperature (S1, S2, S3) and one for humidity (S4). The air and liquid flow rates are measured by means of the rotameters.

Table 1
General properties of materials.

Name-formula	Appearance	Density (kg/m^3)	MW (kg/mol)
Sodium percarbonate $Na_2CO_3{\cdot}1.5H_2O_2$	White solid	Water free 900	0.157
Sodium silicate Na_2SiO_3	White to greenish opaque crystals	2610	0.122

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