Experimental Study of Coating in a Bottom Sprayed Fluidized Bed

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ABSTRACT: *Several investigations have been devoted towards understanding the coating process in fluidized beds. Most of these studies focused on spraying liquid droplets over the bed. However, due to the complexity of these systems, more investigations are still required to quantify the effect of operating conditions on the coating criteria. The present work aims at fundamentally understanding and precisely determining the effect of different operating conditions on coating quality in a jetting fluidized bed where the coating agent is sprayed as liquid droplets into the bed. A 3-D fluidized bed with a spraying nozzle situated in the middle of a perforated distributor plate was used for the experimental study of the effect of the operating condition on the coating quality. Results from the experimental part showed that at maximum jet gas flow rate which the jet didn't turn into a spout and turbulent stage of fluidization the coating quality improves, but there is a limitation for the binder flow rate(NAR=4.0E-5) which is recognized in this study and could be use for any fluidized bed coater in the same condition. Based on the experimental results, an empirical function was derived to predict the coating efficiency in different operating conditions and this function also could be used in the mathematical model.*

KEY WORDS: *Coating, Fluidized bed, Bottom spray, Jet.*

INTRODUCTION

The fluidized bed coating of particulate solids is a technique widely used in diverse branches of chemical engineering. This multiphase unit operation can be defined as a process where a desired amount of a coating agent is deposited on the surface of solid particles. The particles are fluidized by a stream of heated gas and the coating agent contained in a solution or a suspension is introduced within the bed of fluidized particles. The latent heat of evaporation of the solvent is brought by the hot fluidizing gas. Typically, the coatings mass content could attain up to 20 to 30%, corresponding to coating layers varying from a few microns to several millimetres after the solidification of the coating agent. It is well established that for Geldart's A and C powders the growth predominantly occurs by agglomeration whereas for larger particles layering mechanism prevails [1].

In the coating process, the most important criteria to be controlled are the quality of the deposited layer as well as the coating homogeneity and efficiency. The latter is defined as the ratio of actually deposited quantity and the

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total amount of coating agent introduced into the system during the same period. These criteria are governed by a multitude of simultaneous and complex phenomena which are dependent on both product- and process-related parameters. Among these phenomena some research has been done to study the effect of different parameters in a fluidized bed coater:

- Coating-substrate interactions which are mainly conditioned by physico-chemical properties of materials such as surface tensions, contact angle, viscosity that influence the wetting parameters [2].
- Liquid distribution among solid particles. This is a function of atomiser type (mono- or bi-fluid, external or internal mixing nozzle), geometrical specifications of the coater (top- or bottom-spray, conical-based or cylindrical, etc.), the liquid flow rate as well as hydrodynamic conditions which govern solids circulation (i.e. fluidizing gas velocity)
- Thermal conditions in the bed imposed by the bed temperature, fluidizing gas temperature [3].
- More accurately, as the coating efficiency is concerned deviations from unity can be attributed to drying of sprayed droplets before colliding a particle, attrition phenomena leading to detachment of deposited coating agent, carried away droplets by fluidizing gas as well as droplets rebound phenomena. As the homogeneity of coating agent distribution is mainly dependent on hydrodynamics conditions.

Another major factor to be supervised and controlled is the operation stability which can be disturbed by bed quenching phenomena. If the size of agglomerates formed becomes too large to be suspended by the fluidizing air, dry quenching occurs. On the other hand, if the liquid flow rate exceeds the evaporation capacity of the apparatus or if the liquid is unevenly distributed leading to creation of locally over-wetted areas the wet quenching takes place.

Furthermore, according to several experimental and theoretical works [5] a bottom sprayed fluidized bed coater can be considered as a juxtaposition of two communicating regions: Spraying zone where particles wetting takes place and drying zone where solvent evaporation and drying occurs. Particles circulate between these two zones are coated through a number of wetting-evaporation cycles. The relative magnitudes of these zones as well as the particle exchange rate between them play a significant role either on the stability of the operation or on the coating criteria.

Due to the existence of two different zones inside the bed the phenomena which occur during the coating is very complex and there are not many studies in such a system. Most of the studies mentioned above are focused on the top sprayed fluidized bed and a few that study the bottom sprayed bed didn't focus on the effect of the operating conditions on the whole process. To have a good control of this operation it is necessary to understand different steps of the process. The present work focuses on the study of a bottom-spray fluidized-bed coater in the absence of any liquid (solvent) evaporation. The main objective is to point out the effect of operating conditions on the coating efficiency and the coating agent distribution on the solid particles and recognize the controlling parameter during the process. However, one should keep in mind that the coating efficiency under dry conditions is generally lower than that obtained in this work because here the only source of coating loss is the elutriation of not adhered droplets by fluidizing gas stream.

MATERIALS AND METHODS

The experimental set-up used in this study is presented in Fig. 1. It consists of a bottom spray fluidized-bed coater. The fluidizing chamber is a cylindrical column with 0.16 m ID and 0.85 m height made from acrylic glass. The gas distributor is a brass perforated plate containing 2325 holes with 1.5 mm diameter, disposed following a triangular path. The column is also equipped with a bi-fluid spray nozzle (Spraying system Co.). The nozzle is situated in the centre of the distributor producing an upward continuous spray. According to the literature [5] this type of system leads to a more uniform wetting process. The atomizing air was supplied from an independent external air source and was fed through the wind box. The coating liquid, an aqueous solution of CMC (carboxy methyl cellulose) 1% w/w, was fed from a pressurized reservoir by means of compressed air. The liquid flow rate, as well as atomizing and fluidizing air flow rates were measured by means of rotameters.

Mono-dispersed Alumina beads in the sieve range of 2 - 2.36 mm which is in group D in terms of Geldart classification, were used in this work as coating support.

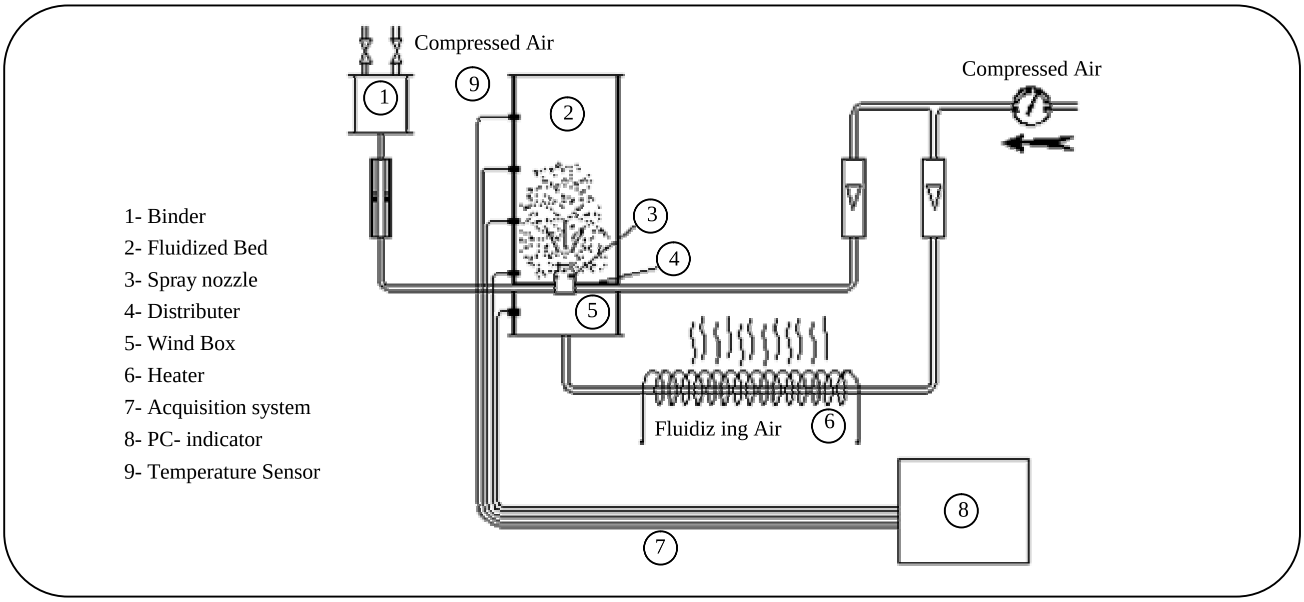

Fig. 1: Experimental set-up.

Some of physical characteristics of alumina particles are summarized in table 1. Alumina porous particles were chosen for several reasons. Firstly, the high porosity of particles permits to reach high liquid contents (about 24%) before the deposition at external surface takes place. On the other hand, the good wettability of this support minimizes the rebound phenomena and consequent loss in liquid.

Initially the bed was charged with 0.832 kg of alumina beads and fluidized at desired velocity. The coating liquid was then sprayed within the bed at a known rate. The atomization was interrupted when a given amount of liquid (0.004 Kg at turbulent stage of fluidization) was introduced in the system. In order to determine the coating mass distribution of particles, samples were taken randomly from different regions of the bed during the process and as soon as the spraying was stopped. An average of 100 coated beads was taken at each sampling. These samples were dried at 60 °C for 8 hr and weighted carefully using a microbalance (0.0001 g precision). The difference between the initial weight of solids and their weight after the layering process helped to determine the coated layer thickness and size distribution in the bed.

RESULTS AND DISCUSSION

A series of experiments was carried out at different operating conditions. Table 2 summarizes the operating parameters together with their range of variation. The central experimental point for each parameter is highlighted.

Effect of fluidizing gas velocity

In a bottom-spray fluidized-bed coater solid particles move from the bed bulk (mixing region) through the jet boundaries and are entrained laterally into the jet region where the wetting process takes place. This is clearly shown in Fig. 2 which illustrates the functioning of a 2D bottom-spray fluidized-bed coater at operating conditions near to those maintained in this study and filled with the same particles.

The solid entrainment rate plays an important role in the efficiency of the wetting during the coating process. Fluidizing gas velocity has a direct effect on the solids entrainment rate into the jet zone [6]. In this study the effect of this parameter on the coating process was investigated by running experiments at different fluidizing gas velocities providing different fluidization regimes and solid entrainment rates. The minimum fluidizing velocity was determined by measurement of the pressure drop versus gas velocity and based on this study other regimes of fluidization were recognised and examined.

Fig. 3 shows the liquid mass content distribution of the coated particles during the layering process at four different fluidizing gas velocities. In addition, Fig. 4

Table 1: Physical properties of solids.

Solid material used	Alumina Beads
Mean size (mm)	2.00
Particle bulk density (kg/m^3))	2790
Particles porosity	24%
Shape factor	1
Initial mass of particles (gr)	0.0204

Table 2: different experimental condition.

Fluidizing gas flow rate (m^3/s)	0.0045, 0.0055, 0.0072, 0.0083, 0.0092
Atomizing gas flow rate (m^3/s)	0.0008, 0.001, 0.0012, 0.0015
Liquid binder flow rate×10^{-5} (m^3/s)	0.04, 0.06, 0.08, 0.1, 0.15,0.2,0.25,0.3
Liquid binder (CMC 1%) density (kg/m^3)	1000
Bed height (cm)	6.5
Bed temperature (°C)	25 °C

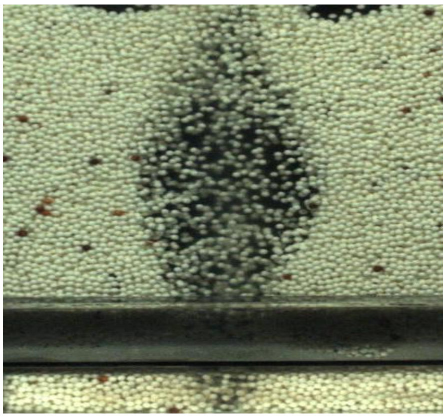

Fig.2: Image of jet which has been captured by high speed video camera at gas jet flow rate 0.0015 m^3/s The red arrows shows the solid circulating pattern

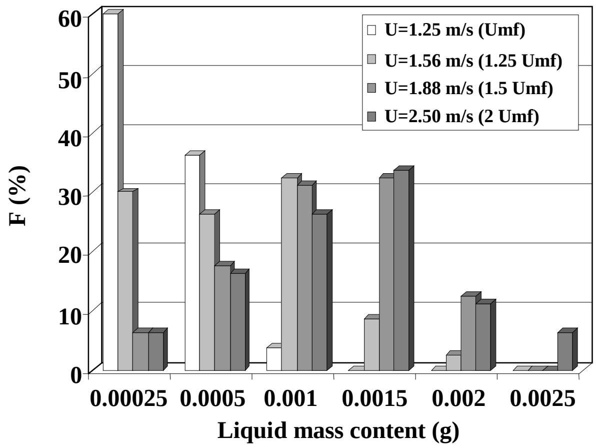

Fig. 3: Liquid mass content distribution of coated particles as a function of the fluidizing gas velocity U (liquid binder flow rate 2×10^{-6} m^3/s, gas jet flow rate 0.0015 m^3/s).

provides the variation of the coating efficiency as a function of fluidizing gas velocity. As it can be seen from these figures, spraying within an incipient fluidized bed leads to a very low coating efficiency and a liquid mass content distribution situated at its lower limits. This results from a poor circulating of the solid particles which is lowest at minimum fluidization condition [7]. Under these conditions the jet could form a cavity within which a very low particle circulation and wetting occurs. In addition, at small fluidization velocities the bed expansion is so low that the jet length exceeds the bed height. The jet completely traverses the bed and the fraction of the liquid which attains the freeboard section is lost. Finally, at these conditions because of the high heterogeneity of the coating the sampling procedure becomes less reproducible and hence more critical. As the fluidizing gas velocity increases the bed height increases with respect to the jet length leading so to a higher coating efficiency. In addition, the solid circulation is improved by increasing the fluidization gas velocity. It can be seen from Fig. 4 that the liquid mass content distribution of coated particles moves from lower limits to upper values when the fluidizing gas velocity is increased.

Effect of atomising gas flow rate

Atomising gas flow rate is an important parameter regarding the coating efficiency [7]. This parameter affects directly the volume of the jet zone as well as the solid concentration in this zone [6]. In addition, increasing the atomising air flow rate leads to a decrease of the droplets mean diameter and a subsequent increase of droplets number. All these phenomena affect the particle-droplet collision probability.

Fig. 5 and Fig. 6 illustrate the effect of this parameter on the coating efficiency. It can be seen that operating

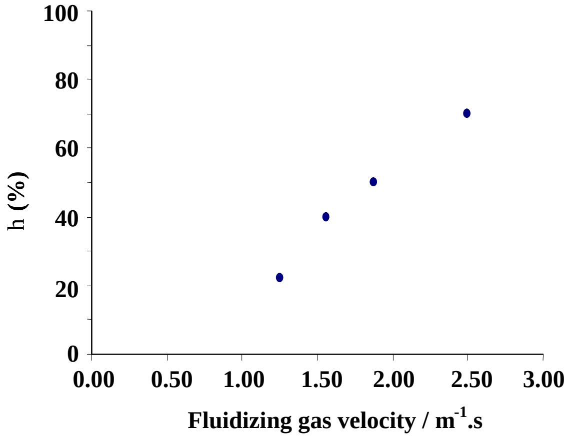

Fig. 4: Influence of the fluidizing gas velocity on the coating efficiency (liquid binder flow rate 2.0×10^{-6} m^3/s, gas jet flow rate 0.0015 m^3/s).

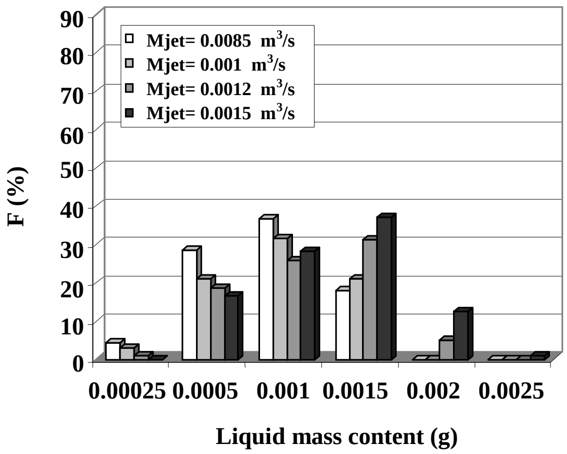

Fig. 5: Size distribution in different gas jet flow rate, liquid binder flow rate 2.0×10^{-6} m^3/s, fluidizing gas flow rate 0.0092 m^3/s.

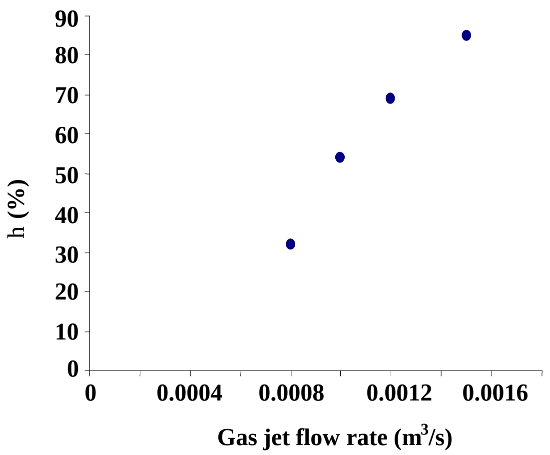

Fig. 6: Coating efficiency in different gas jet flow rate, liquid binder flow rate 2.0×10^{-6} m^3/s, fluidizing gas flow rate 0.0092 m^3/s.

at high atomizing gas velocities results in a translation of mass content distribution from the lower limits to higher limits i.e. from poor to rich extremity. This effect is sensibly reflected in the variation of the coating efficiency with the jet gas flow rate presented in Fig. 6. In addition, the higher the jet gas velocity the narrower the liquid mass content distribution. Although a high jet gas flow rate results in a more dilute jet region, the overall increase of the operating efficiency indicates that other positive aspects (i.e. higher circulating flow rate and droplets number) have a more dominant role. In addition, operating at high jet gas flow rates prevents particle agglomeration due to lower concentration and higher velocity of solid particles in the jet zone.

However, several workers [8] stated that high gas velocities lead to high attrition rates in particular for brittle particles. Although this study did not focus in attrition aspects it must be kept in mind that for more brittle particles the optimum jet gas flow rate is one that allows ensuring the best compromise between the coating efficiency and the attrition rate.

Effect of liquid binder flow rate

The effect of spraying liquid flow rate was investigated using different values of this parameter (6.0E-4 to 3.0E-3) at the best atomizing air flow rate which recognized based on the experiments (0.0015 m^3/s) corresponding to NAR ratios of , 1.0E-6, 1.6E-5, 2.5E-5, 4.0E-5 and 5.0E-5 respectively. Fig. 7 shows that small liquid flow rates (less than 2.5E-5 corresponding to $NAR=M_{liq}/M_{jet}=2.5e\text{-}5$) cause a long period process with no bed quenching. However, due to small amount of the liquid droplets sprayed into the jet region and their small size, even at room temperature a large part of the liquid evaporates before deposition on solids surface occurs. It causes a very low coating efficiency. Figs. 7 and 8 show that increasing the liquid flow rate (e.g. decreasing NAR) up to a critical value (NAR=4.0E-5) increases the coating efficiency to produce a uniform coating. Beyond this value increasing the liquid flow rate has a hostile effect as it may lead to creation of locally over-wetted regions which cause a poor fluidization and may cause forming large agglomerates which lead to bed quenching.

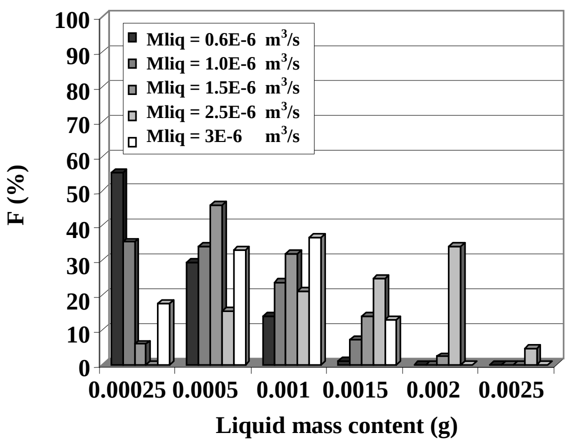

Fig. 7: Size distribution in different liquid binder flow rate, fluidizing gas flow rate 0.0092 m^3/s, gas jet flow rate 0.0015 m^3/s.

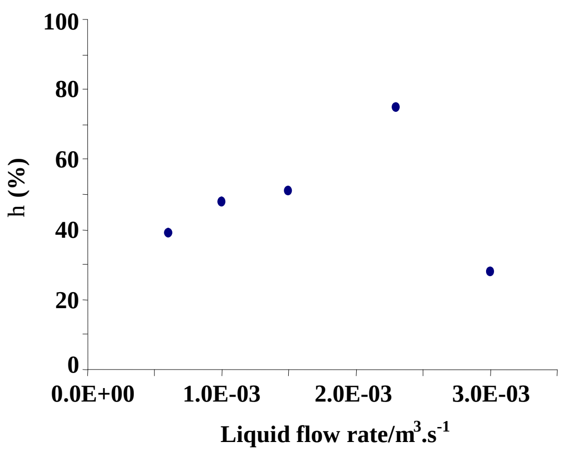

Fig. 8: Coating efficiency in different liquid binder flow rate, fluidizing gas flow rate 0.0092 m^3/s, gas jet flow rate 0.0015 m^3/s.

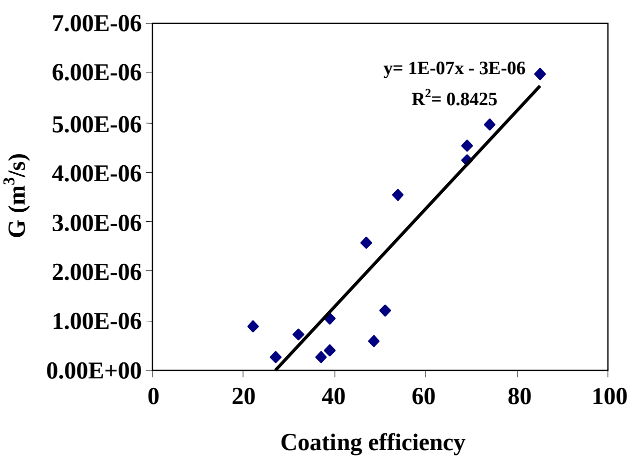

Fig. 9: Variation of growth rate as a function of efficiency.

Coating efficiency and growth rate, G

The particle growth rate, G, is taken to be the rate of increase in particles liquid content which results from liquid deposition into the internal surface of particles due to the fact that there are two zones in the bed and there are no liquid droplet coming out of the spraying zones [9]; it could be concluded that growth only happened in the spraying region. Assuming that particles have all the same characteristic and m, is the rate of mass increase due to the liquid deposition:

$$\frac{dM}{dt} = N_j \frac{dm}{dt} = N_j G \tag{1}$$

Where m is the mass of liquid gained by each particle and N_j is the number of particles in the spraying zones which is calculated based on the following equation:

$$N_j = \frac{V_j}{V_T} \varepsilon_j N_T \tag{2}$$

On the other hand, the variation of particles mass can be related to the deposited fraction of total liquid introduced to the bed. If it is assumed that the coating efficiency is a random parameter which is independent of the particles characteristic, the fraction of deposited liquid is proportional to the fraction of particles which belong to the spraying area and to the coating efficiency for that area:

$$\frac{dM_j}{dt} = \frac{N_j}{N_T} \frac{dM}{dt} = \frac{N_j . \eta . w_{liq}}{N_T} \tag{3}$$

Where η is the coating efficiency and w_{liq} the liquid mass flow rate. Combining equations (1) and (3) lead to an expression for the growth rate, G:

$$G = \frac{\eta . w_{liq}}{N_T} \tag{4}$$

This equation states that the coating efficiency changes linearly with the growth rate and this also can be seen from the experimental results Fig. 9.

Note that according to the experimental observations the coating efficiency is a strong function of hydrodynamic behaviour of the bed and operating conditions. In order to find the relation between these parameters, the modified back propagation artificial neural network method [10] was used to find a non linear function which could fit the experimental data. For operating conditions used in this study, the coating efficiency as a function of fluidizing gas flow rate, jet gas flow rate, liquid binder flow rate and duration of the process (time of bed quenching) could be determined. This model could predict the amount of coating efficiency in any operating condition. Using the following model is a great help in predicting the effect of operating condition on the process by mathematical modelling.

$$y_1 = 0.27\dot{M}_{jet} - 3.91\dot{M}_{liq} - 4.88t_f \quad (5)$$

$$y_2 = -4.47\dot{M}_g - 2.11\dot{M}_{jet} + 0.51\dot{M}_{liq} - 0.1t_f + 0.61$$

$$y_3 = 0.011\dot{M}_g - 5.53\dot{M}_{jet} + 0.02\dot{M}_{liq} + 4.72t_f + 0.14$$

$$\eta = \frac{1}{1 + \exp(4.31y_1 + 2.41y_2 + 4.41y_3 - 0.13)}$$

CONCLUSIONS

The bottom sprayed fluidized bed coater has the advantage of a self controlling system and so it is of great deal of interest these days. This study attempt to understand the phenomena which has happened in the bed during the process of the effect of the operating condition and hydrodynamic behaviour of a bottom-spray fluidized-bed coater on the coating mass content and efficiency was studied. An optimum operating condition for achieving a higher deposited mass on the particles was found. Based on experimental results a mathematical expression for the coating efficiency was derived using modified back propagation artificial neural network to predict the relation between the efficiency and operating conditions in such systems. Besides using the derived equation for predicting the efficiency of the process, this expression will be of great use in making the mathematical models independent from the experimental data and determine the real growth rate in any bottom sprayed fluidized bed coater.

It has been also determined that for achieving a uniform binder deposition and significant liquid contents, the bed should work on the fast fluidization stage and a high jet gas flow rate before it turns to a spout and a high concentration of binder liquid droplet inside the jet region. However, a critical value was recognized for droplet concentration in this zone. These conditions are very useful for any fluidized bed coater for achieving a high efficiency coating process.

Notations

G	Coating rate (m^3/s)
M	Particle mass in group i (kg)
m	Liquid mass content of each particles in group i(kg)
N_j	Class i particle number (-)
N_T	Total particles number in the bed (-)
S	Surface of the jet region (m^2)
t	Time(s)
w_{liq}	Liquid mass flow rate (kg/s)
f	percentage of the coated particle in the bed

Greek letters

ε	voidage (-)
η	Coating efficiency

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