Dry particle coating in a pan coating device

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Introduction

The Coating technology takes part of "microencapsulation" field. The coating is an application of a layer of liquid and/or powder, by different kind of techniques, on material, and which gives to the product characteristics of surface. It is used primarily to form a barrier or film between the host particle and its environment. Its purpose is to produce extended or delayed release, protection from water vapour, light or oxygen, taste masking. In addiction modification of powders' properties is very important for many applications in food products (Naito et al. 1993). Even more and more biologically active substances are used in various industries because of their beneficial functional properties and effects on the environment and human health (Ivanova et al. 2004). The coating process of food materials can be employed to enhance, time or tune the effect of functional ingredients and additives (Arshady, 1993).

In the traditional coating, process consists to spray a coating material solution. In case of organic solvent solution, this one has to be recovered to avoid pollution, leading to important overcost. Coating processes with aqueous dispersions are time and energy consuming (Wheatley et al. 1997) caused by the low concentration of coating polymer and large amounts of water, which have to be evaporated.

Dry particle coating, consists in coating relatively large particle size (core material or host) with fine particles (guest). The adhesion of these particles is made using mechano-chemical treatment or using plasticizer. The using of plasticizer is particularly indicated to coat particles that are relatively soft and very sensitive to heat and can be deformed by severe mechanical forces (Obara et al., 1999, Pearnchob and Bodmeier, 2003a,b). Compared to solvent and water based coating the dry coating method is favourable regarding environmental friendliness, safety and cost.
Present contribution wish to provide preliminary result to apply dry coating in food and feed field. More specifically, the aim of this study is to obtain coated pellets by dry coating process with polysaccharides materials.

Materials and methods

Materials.
Core particles: Microcrystalline Cellulose Spheres (CELLETS®1000-1400µm, IPC Process-Center GmbH & Co. KG, Dresden, Germany) as inert support.
Coating powder: Aluminium starch octenyle succinate (Roquette, Lestrem, France), Starch octenyle succinate (E1451), Tapioca Dextrin (National Starch, Düsseldorf, Germany), Malto-dextrin (Roquette, Lestrem, France).
Plasticizer: Triethyl citrate (TEC, Merck KGaA, Darmstadt, Germany).
Equipments. Pan Coater (prototype designed and manufactured in our laboratory) (Fig.1).

Methods.
Coating process.
The process was conducted in a Pan Coater (Fig. 1). Coating powder was quantitatively charged with feeder system while with the plasticizer was sprayed through the pneumatic concentric nozzle. The coating process was performed in 25 minutes with the parameters describes in Table 1.

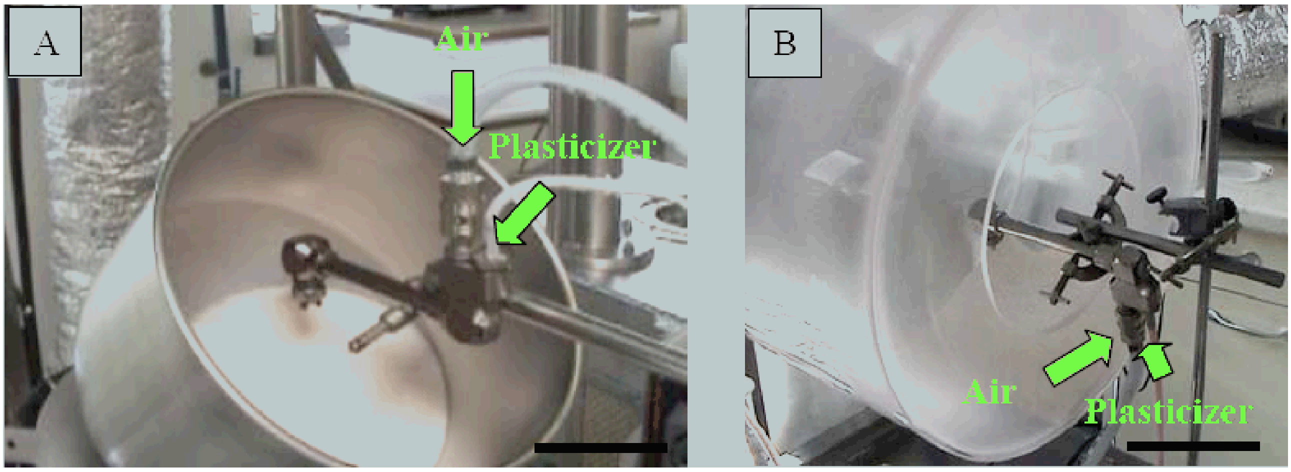

Fig. 1. Images of Pan Coater prototypes: 45° in Steel (A) and horizontal Plexiglas (B).

Tab. 1. Process parameters.

Batch Size	Temperature	Air nozzle flow pressure	Powder feed rate	Plasticizer feed rate
400g	25°C	0.3 Bars	4g/min	2g/min

Particle size : Particle size of coating polymers was measured by laser light diffraction (Mastersizer, Malvern, United Kingdom).

Glass transition : Glass transition temperature of the coating powder was measured by Differential Scanning Calorimetry (DSC Pyris 6, Perkin Elmer, USA)

Coating efficiency. : The coating efficiency is defined as: $\textit{Coating Efficiency}\ (\%) = \frac{M_{CB} - M_B}{M_C}$

where M_{CB} is coated beads mass (g) and M_B is the uncoated beads mass and M_C is sprayed coating material (powder + plasticizer).

Coating Level. The coating level is the weight of the polymer powder and plasticizer (M_C) divided by the weight of uncoated pellets (M_B).

$$\textit{Coating Level}\ (\%) = \frac{M_C}{M_B}$$

Coating film thickness and surface morphologies. Surface and cross-sectional morphologies of coated particles were observed with a stereo-microscope (WILDMC3, Leica, Germany), and with a SEM microscope (JSM-6400M, Jeol, Japan).

Results and discussion

The experiments carried out results with coating efficiency between 85 and 95 % for the majority of polymers, as reported in Table 2.

Tab. 2. Manufacturing parameters used for the production of coated particles.

Polymer	Mean diameter	Coating Efficiency	Observations
Malto-Dextrin	80 µm	< 50 %	Presents of aggregates onto pellet surface
Octenyl succinate starch	50 µm	< 60 %	Presents of aggregates onto pellet surface
Tapioca Dextrin	30 µm	85-95 %	Homogenous shell onto core particles surface
Aluminium octenyl succinate starch	20 µm	85-95 %	Homogenous shell onto core particles surface

Particles powders shape is an important parameter; in fact results have shown that, for example, particles with needle shape (Fig.2B) don't allow to get a layer formation. This is caused because particles don't stick with MCC pellets surface, or they don't remain onto pellets surface to don't allow forming of more particles layers. In fact experiments conducted with particles having shape too much spherical (Fig.2A) cannot obtain a film formation because powder particles during the process are behaved like flowing agent and for this reason the particle motion can't be control in the reactor, that come out impossible to go on the coating process.

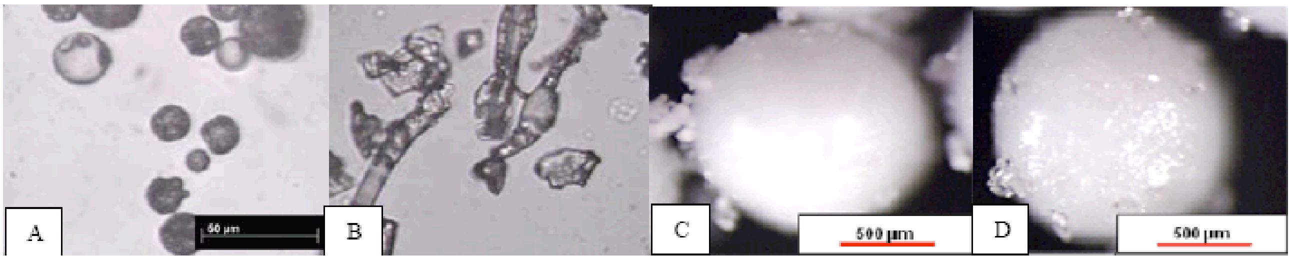

Fig. 2. Optical stereo-photomicrographs of polymer particles (E1451 in A and MaltoDextrin in B) and coated pellets with E1451 (C) and MaltoDextrin (D).

Figures 2C and 2D show particles coated with E1451 and MaltoDextrin. Trials conducted with these polymers have shown presence of polymers aggregates onto MCC pellets surface, and in particular in Fig. 2D, where shiny pellets surface show that plasticizer sprayed coat pellet's surface and only some polymer aggregates remains attached to it. Therefore it results impossible use particles with these properties in order to obtain a coating layer.

With regard to particle size, our results have shown that polymers with particles size more than 50µm give problems during the coating process. Particles bigger than 50µm are not in close contact with pellet surface for time enough to have layer formation. They remain in reactor during the process but they don't take part in coating process remaining as a discarded.

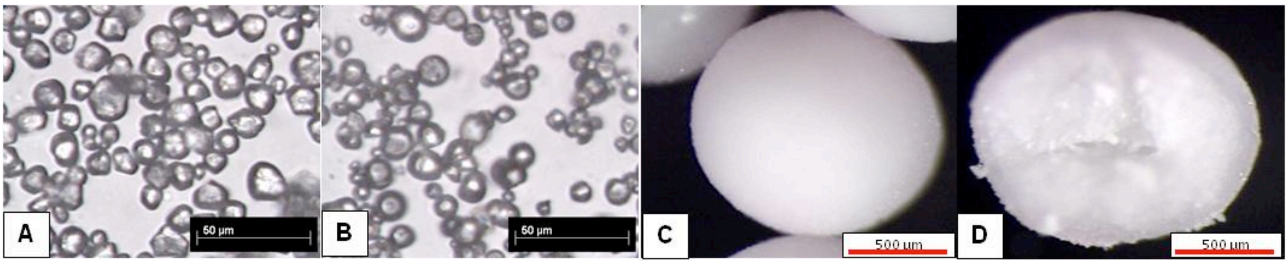

Fig. 3. Optical (A,B) and stereo (C,D) photomicrographs of polymer particles (Tapioca Dextrin in A and Aluminium starch octenyle succinate in B) and coated pellets with Tapioca Dextrin (C) and Aluminium starch octenyle succinate (D).

Instead particles under 50µm show a good ability to form coating layers onto pellets surface (Fig. 3 A and B). This permit to send our choice to commercial powder with size less than 50µm in order to obtain coating process with Pan Coater device.

In Fig.3 C and D optical microscopy images show that coating shell thickness is uniform enough around all pellet's surface. Image obtained by Scanning Electron Microscopy (SEM) instead has shown that polymer particles are compacted uniformly on MCC pellets surface (Fig. 4A and C), but the morphology of film shell show that thickness is not completely homogeneous and there are not much cohesion between polymer's particles (Fig. C).

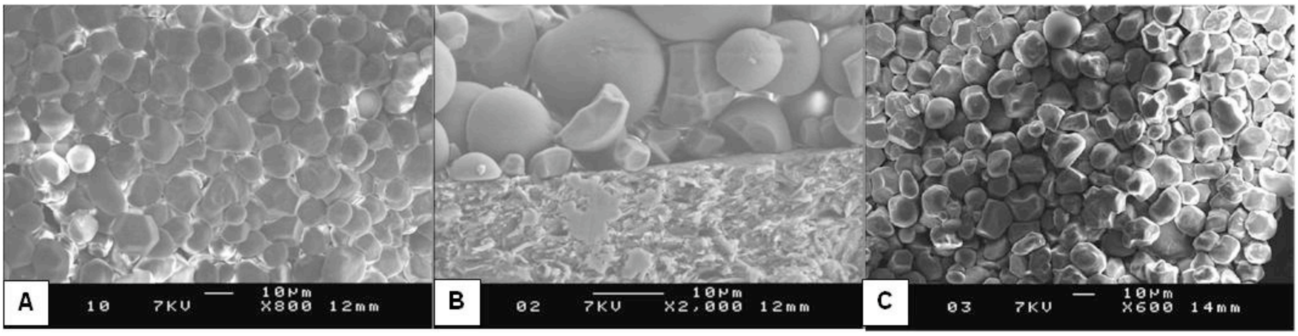

Fig. 4. SEM image analysis of MCC particles coated with Tapioca Dextrin (A,B) and Aluminium octenyl succinate starch (C).

Conclusion

The results have shown the feasibility of a dry coating process in Pan Coater device.
The use of formulation with different kind of polysaccharides has shown a capability to produce coated particles with small losses of coating material at room temperature.
It is possible to obtain a uniform coating with polysaccharides particles size under 50µm and spherical shape.
The coating layer obtained doesn't present coalescence between polymer particles. Further experiments are in progress in order to obtain homogeneous coating layer onto pellets surface.
In conclusion this coating process is a highly efficient process convincing with its short processing time of 20-25min causing low energy consumption compared to standard coating process.
The perspectives are to study physical principles of adhesion; study of coated particles' field employment in food or pharmaceutical applications.

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