Particle coating using dry powder technology

L. Bilancetti[1], M. Huchet[1], C. Loisel[1], D. Poncelet[1*],
ENITIAA, Rue de la Géraudiére, F-44322 Nantes Cedex 03, France
(luca.bilancetti@enitiaa-nantes.fr)

Introduction

Modification of powders' properties, such as flowability, wettability, flavour, colour, etc., 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).
The process consists of two steps: sprayed coating solution, through a nozzle, on the particles; evaporate the solvent, in order to obtain a coating layer onto particles surface.
During the traditional coating process based on organic solvent, the solvent needs to be recovered due to environmental pollution. 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 need to be evaporated.
Dry coating is not really a new technology. It had been applied in chemical, aeronautical, metallic, wood and paper industries mostly for coating or recovering woods, metals or any desired surface by paints, varnishes, different polymers or precious metal to protect them against corrosion or to reinforce them. Its application to food area is quite recent and is still in its infancy.
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.
Compared to solvent and water based coating the dry coating method is favourable regarding environmental friendliness, safety and cost.
It might be a very suitable coating method in order to coat foods and drugs which are sensitive to organic solvents or water.

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: Hydroxypropyl methylcellulose acetate succinate (AQOAT®, Shin-Etsu Chemical Co., Niigata, Japan), Eudragit E PO (Degussa AG, Düsseldorf, Germany), Shellac (SSB®55 Pharma, de-waxed fine powder, Syntapharm Ges. f. Pharmachemie GmbH, Mülheim an der Ruhr, Germany), modified Polysaccharides modified (Matrix 1, Matrix 2, Matrix3, confidential)

Plasticizer: Triethyl citrate (TEC, Merck KGaA, Darmstadt, Germany).

Equipments.
Wurster bottom spray coater (Glatt, Binzen, Germany). Rotary disk coater (prototype designed and manufactured in our laboratory). Pan Coater (prototype designed and manufactured in our laboratory).

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

Coating efficiency.
The coating efficiency was calculated by this relation:

$$E_C = \frac{\text{Mass of coated particles} - \text{Mass of uncoated particles}}{\text{Mass of sprayed coating material}} \%$$

Coating film thickness and surface morphologies.
Surface and cross-sectional morphologies of coated particles were observed with a stereo-microscope (WILD MC3, Leica, Germany), and particles size were determined by a computer based image analysis software (VISILOG, Noesis, France).

Results and discussion

In the present study, dry coating process is carried out applying three different equipments, Wurster, Rotary Disk, and a Pan Coater (fig.1).

Process with Wurster:
Experiments conducted with Wurster equipment have shown results below the 5% of coating efficiency. During the coating process, a large amount of coating powder gets lost onto the filter placed on the top of reaction chamber. This problem has had to the large difference of size between core particles and guest particles that influence the segregation of powder's particles.

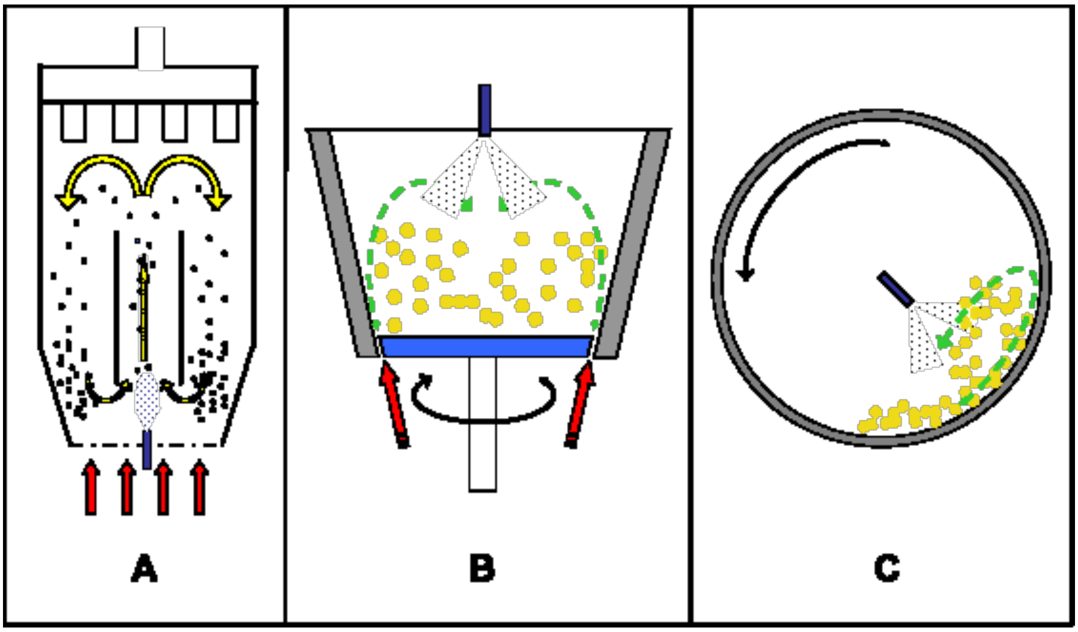

Fig. 1. Schematic representation of Wurster spray coater (A), Rotary disk coater (B), Pan Coater (C).

Process with Rotary Disk:
In experiments conducted with Rotary disk equipment, we have observed problems of abrasion between the particles and the surfaces of reactor chamber. The results have been around 0% of coating efficiency. However we are working to improve design and efficiency of this equipment.

Process with Pan Coater:
Experiments conducted with Pan Coater equipment have shown results around 85% of coating efficiency (Fig.2).

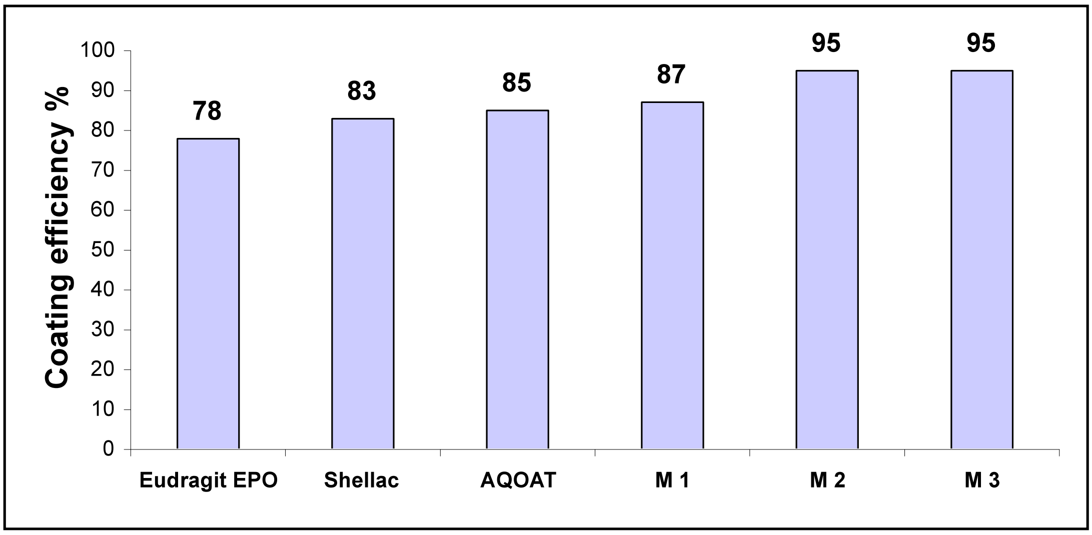

Fig. 2. Coating Efficiency of particles coated with Pan Coater.

In Table 1 are reported the manufacturing parameters used for the production of coated particles.
Selection of polymer.
All the polymers used have shown a high value of coating efficiency (Fig. 2). The polysaccharides Matrix 2 and Matrix 3 have given the best results. These are food compounds, therefore excellent for food applications.

Polymer	Temperature	Process Time	Mean diameter	Coating Efficiency	Observations
E PO	20°C	14 min	40 µm	78 %	Continuous layer onto core particles surface
Shellac	20°C	15 min	90 µm	83 %	Guest particles aggregates onto core particles surface
AQOAT	20°C	23 min	25 µm	85 %	Continuous layer onto core particles surface
Matrix 1	20°C	21 min	50 µm	87 %	Guest particles aggregates onto core particles surface
Matrix 2	20°C	21 min	15 µm	95 %	Continuous layer onto core particles surface
Matrix 3	20°C	25 min	30 µm	95 %	Continuous layer onto core particles surface

Tab.1 Manufacturing parameters used for the production of coated particles.

As reported in Fig. 3, the particles coated with Shellac and Matrix 1 (large particles size) shown an unhomogeneous coating layer, due to presents of some aggregates (pictures B and D). The particles stretch to aggregate together before the adhesion onto the surface of core particles.
As reported in Fig. 3, particles coated with Eudragit E PO (picture A), AQOAT (picture A), Matrix 2 (picture E) and Matrix 3 (picture F) we can observe a homogeneous coating layer.

The micrographs of cross-section of particles coated with Matrix 3 (A) and Matrix 2 (B) (Fig. 4) shown the film thickness of the coated pellets, and we can observe a continuous film without many unevenness.

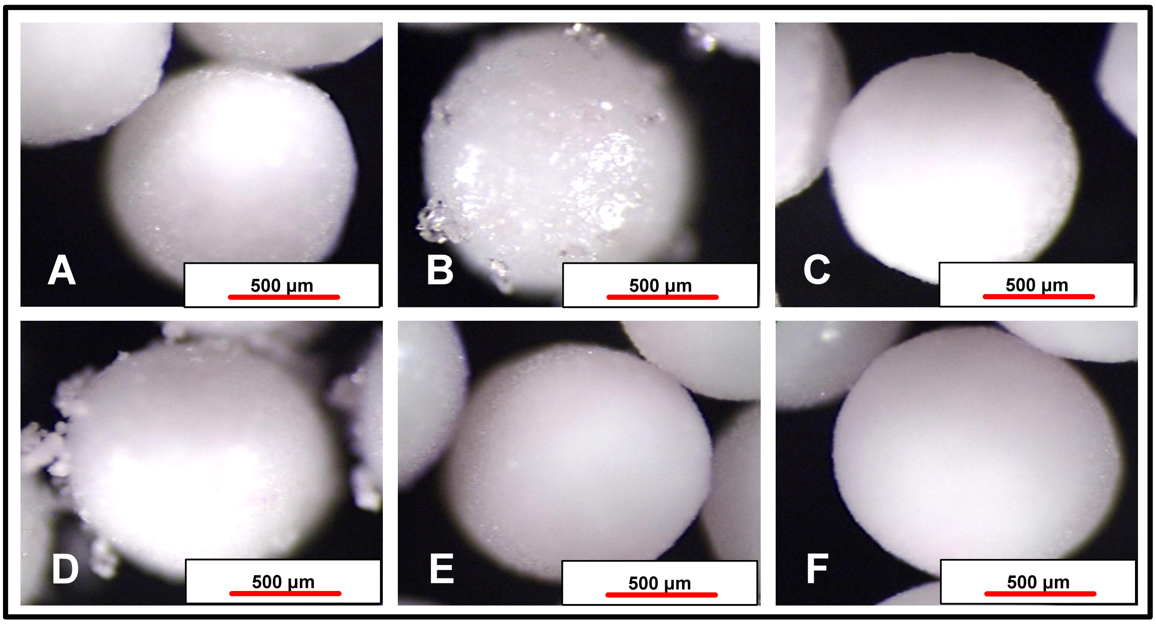

Fig. 3. Optical stereo-photomicrographs of particles coated with Eudragit E PO (A), Shellac (B), AQOAT (C), Matrix 1 (D), Matrix 2 (E) and Matrix 3 (F).

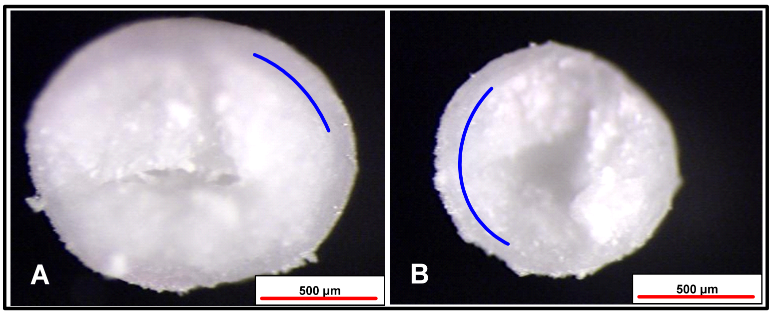

Fig. 4. Stereo-micrographs cross-section of coated particles with Matrix 3(A) and Matrix 2(B).

Conclusion

The dry coating process opens many possibilities for developing innovative formulation, for food and pharmaceutical applications.

The results have shown that the best equipment for dry coating process is Pan Coater. The use of formulation with polysaccharides has shown a capability to produce coated particles with a high coating efficiency. The perspectives are: study of coated particles morphology; study of coating layer mechanism formation; study of coated particles' application field.

References

M. Naito (1993). ISIJ International 33(9)915–924.

Ivanova et al. (2005) *Encapsulation of water sensitive products: effectiveness and assessment of fluid bed dry coating.* J of Food Engineering (71)223–230.

Arshady, R. (1993) *Microcapsules for food.* J. Micro-encapsulation (10)413-435.

Wheatley (1997) *Latex emulsion for controlled drug delivery*. Int. J. Pharm. (154)167–178.