Encapsulated and Powdered Foods

Single Encapsulation Process

A

Cluster

Single Capsule

Sucrose Wall Ridges

30-50μm

Waxy Fat Bodies

Double Coat

B

C

Double Coated Particles
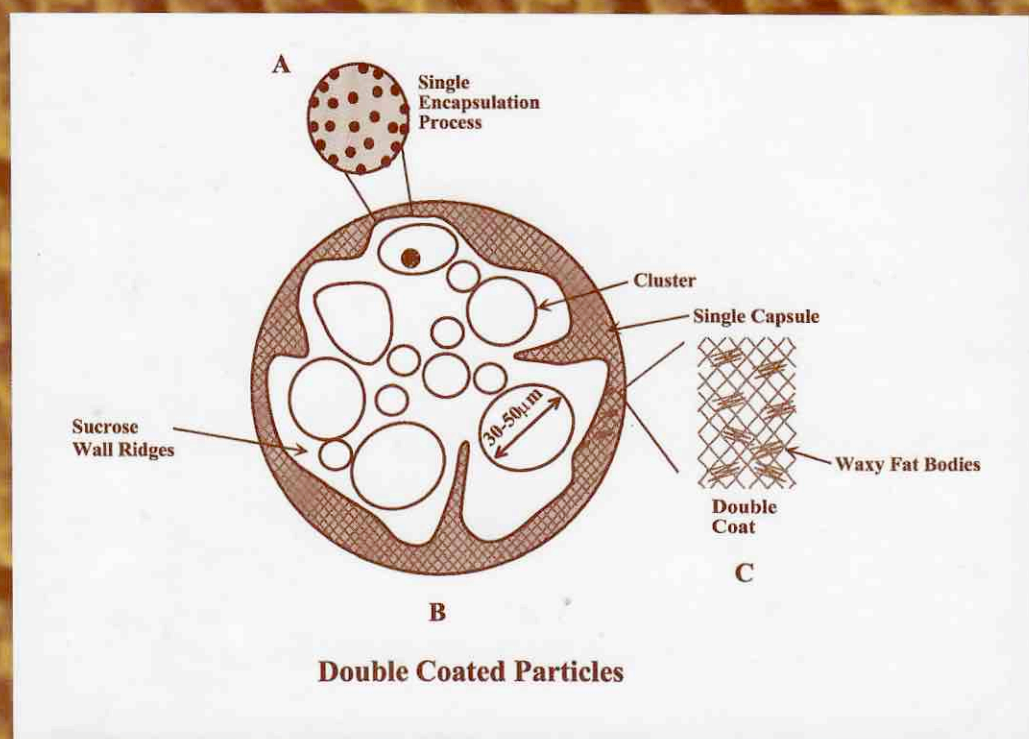
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Dry Coating

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CONTENTS

I. SUMMARY

Dry coating is suitable for coating of core materials that must not be wetted during processing, for example, some pharmaceuticals and most of the neutraceuticals. Different dry coating processes are reviewed, particularly dry impact-blending, coating with plasticizer, and hot-melt coating. A specific case is presented in which spherical granules made of crystalline cellulose (750 to 800 mm) are coated by a fine powder (AQOAT) using triethyl citrate as plasticizer. The examined characteristics are the water barrier properties, their water content, and their quick solubility. The results show that the AQOAT film presents fast dissolution in water (2.5 min) and relative protection. Unfortunately this protection is not sufficient in certain cases, for example, the 2% moisture content of the resulting coated granules is not satisfactory for probiotics requirements, even if this value is smaller than the one obtained with the aqueous coating system. Finally, dry coating using a plasticizer appears quite adequate for coating moisture-sensitive food products but this requires some improvement of the processing conditions.

II. INTRODUCTION

Dry coating in the present case must be considered as all coating processes where the core materials are not wetted (by water) during processing. It is a response to the coating of all products that are sensible to water and hence to all traditional aqueous coating methods such as some pharmaceuticals and most of neutraceuticals (specially probiotics). These types of products are of great interest for different industries. This interest is based on production of new, healthy, and natural tablets and innovative ingredients for improvement of human health. One of the important factors, which have an influence on human health, is the quality and security of nutrition. Today, it must be admitted, not almost unanimously, that the inclusion of functional foods or neutraceuticals in foods can help to fulfill these objectives.

Although there is no specific definition of neutraceuticals, they can be defined as ordinary foods that have components or ingredients incorporated into them to give them a specific medical or physiological benefit, besides a purely nutritional effect. This definition can include supplementation of food (cereals, grains, drinks, and yoghurts) with minerals, vitamins, antioxidants, probiotics, prebiotics, herbs, botanicals, and oils [1]. Probiotics may be defined as life mono or mixed cultures of microorganisms which, when applied to animals or humans, have a beneficial effect on the host by improving the properties of the indigenous flora. The presence of probiotics in the gastrointestinal tract, for example, prevents colonization with photogenic microorganisms [2]. Moreover, they can influence the secretion of intestinal mucosa, corresponding with pathogenic attachment in intestines. The beneficial effects of probiotic bacteria are also related to (1) significant suppression of the tumor growth [3,4]; (2) antiallergy effect [5]; (3) inhibition of *Helicobacter pilori*, which is associated with chronic gastritis, peptic ulcers, and risk of gastric cancer [6]; (4) decreasing the cholesterol level [7]; and (5) improving lactose utilization.

Most of these products, which are in granular form, are very sensitive to water. It is quite understandable that the most important thing, for preserving the positive effects of these functional foods, is the quantity of life (probiotic bacteria) or active (minerals or fats) components in the final ready-to-use products (granules, pellets, or tablets). This quantity depends mainly on the moisture content of the product, which in itself is related to the production technique and also to the storage conditions. Spray and freeze drying and different aqueous coating techniques have been successfully used for this purpose but when the active components are too sensitive to moisture, these methods are not adapted. Indeed, during

the last twenty years, pharmaceutical coating technology has been based on the aqueous systems, which do not cause environmental pollution [8]. During the aqueous film coating process, the humidity in the coating chamber increases significantly and Clementi and Rossi [9] demonstrated that the viability of dry probiotics decreases rapidly, especially with high moisture content. Hence aqueous coating systems are not applicable for encapsulation of dry probiotics. Dry coating is an alternative to these conventional methods, which can allow maximal saving of the viability and activity of the components by minimizing the effects of atmospheric moisture on their shelf life and by slowing down the rate of viability loss at room temperature. The growing demand for functional foods and additives (e.g., the European market was estimated at £1000 million in 2000) is a consequence of two parallel trends in the food and life science industries: consumers now spend less time cooking per day, down to an average of 20 min from 50 years ago and they also demand more and more safe, hygienic, and healthy foods, this last point being amplified by the aging of the population [10]. There is a real need to find new dry coating techniques, which propose better conditions to preserve the sensitive dried powders against water vapor and oxygen and offer excellent potential for commercial application. These techniques are grouped into four main approaches:

1. The use of nonaqueous solvent in traditional coaters.
2. The hot-melt coating.
3. The dry particles coating where the fine particles are fixed on the surface of the core material by higher agitation using mechanochemical treatment with no wetting.
4. Coating with plasticizer where the fine particles are fixed on the core using a plasticizer.

The aim of this chapter is to describe these different approaches and to analyze a case study where a cellulose granule (as a probiotic model) is dry coated by a fine powder (AQOAT) using a plasticizer. The formed coating shell was tested, keeping in mind its permeability, its water content, and its dispersion in water, and compared to a membrane obtained by aqueous coating.

III. DRY COATING

Dry coating is not really a new technology. Indeed, dry coating was first used in the preparation of pharmaceuticals, cosmetics, dental materials, HPLC packing, ceramics, copy toners, cements, inks, pigments, detergents, and so on [11]. Its application in chemical [12], aeronautical, metallurgical [12–17], wood and paper industries consists, most of the time, in coating or recovering woods, metals, or any desired surface by paints, varnishes, different polymers, or precious metals to protect them against corrosion or to reinforce them [18]. But its application to granular material and specifically in the food area is quite recent and is growing for various purposes (improvement of powder flowability, control release, and fast-soluble powders) but it can be said that it is still in its infancy.

In its strictest sense, dry coating refers to techniques where the core materials are strongly surrounded by fine particles simply by collision or by the use of nonaqueous plasticizers. But, as specified in the introduction, dry coating for food ingredients refers to all these techniques that allow coating without wetting (with water) a core material. So defined, the use of nonaqueous solvents and hot melt are considered as dry coating techniques. Figure 1 summarizes the principles of the four dry coating approaches. These approaches are briefly described in the next paragraph; the solvent approach is equivalent
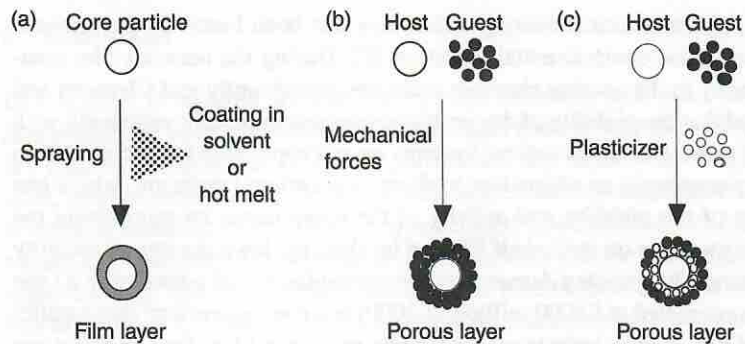
Figure 1 Principles of different dry-coating approaches: (a) solvent and hot-melt, (b) dry particle coating, and (c) use of plasticizer.

to the conventional coating with the difference that the coating material is dissolved in an adequate and suitable nonaqueous solvent. The hot-melt approach requires a coating material that is liquid at relatively higher temperature (hot melt) and solid at handling and processing temperature. The dry particle coating approach requires high-energy throughput to create collision and adhesion between core particles and the fine dry particles while the fourth approach uses a plasticizer to bind them.

IV. DIFFERENT APPROACHES FOR COATING PARTICLES WITHOUT WETTING

A. Use of Nonaqueous Solvents

Vaporizable solvents for coating were first used to enhance the material efficiency of the coating process by increasing the solid content of the sprayed coating material while keeping low viscosity and their application as a dry coating process was limited to nonaqueous solvents. Indeed, the use of the solvents helped to increase the solid content from 4 to 20% and brought some satisfaction to the pharmaceutical industries in the past. The most used solvents are alcohol (methanol, ethanol, n-propanol, and n-butanol), cetones (acetone), ether, and esther (dichloromethane, ethyl acetate, chloroform, etc.).

The process can take place in all types of conventional fluid-bed and pan-coating systems (Figure 2), which are well-described in other chapters. As mentioned above, the coating material is dissolved in a solvent, which is evaporated after spraying on the core. It is quite clear that the main drawbacks of this approach are the emission of solvent into the atmosphere, the solvent residue in the final product, and the risk of explosion. The permissible emission rate for solvents, which is determined by the local authorities, has been further reduced by European regulations. So the application of this approach requires an expensive solvent recovery system (Figure 3) where the solvent can be recycled properly. Nevertheless, in the best cases 5 to 10% of the solvent is not recovered [19].

The use of vacuum installation during this process has been known to reduce the limitations of this technique, specifically the explosion hazard, the environmental strains, and the solvent losses. The system relies on the physical properties of solvent evaporation in vacuum and the process can be optimized to minimize energy consumption. With this improvement the process is sterile and suitable for coating active agents, aromatic substances, easily oxidizable, hydroscopic, and/or heat-sensitive products.
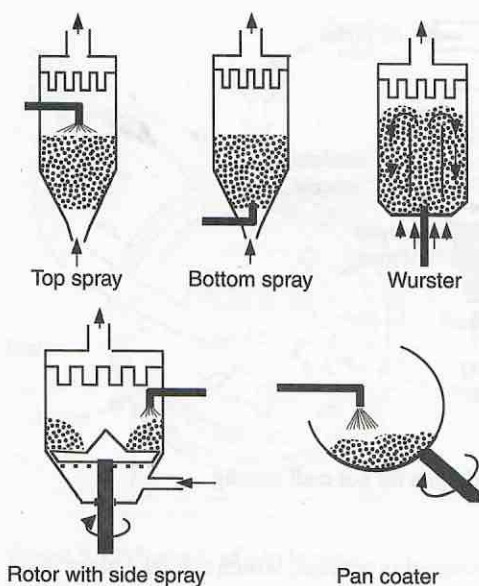
Top spray   Bottom spray   Wurster

Rotor with side spray   Pan coater

Figure 2   Different types of particle coaters.
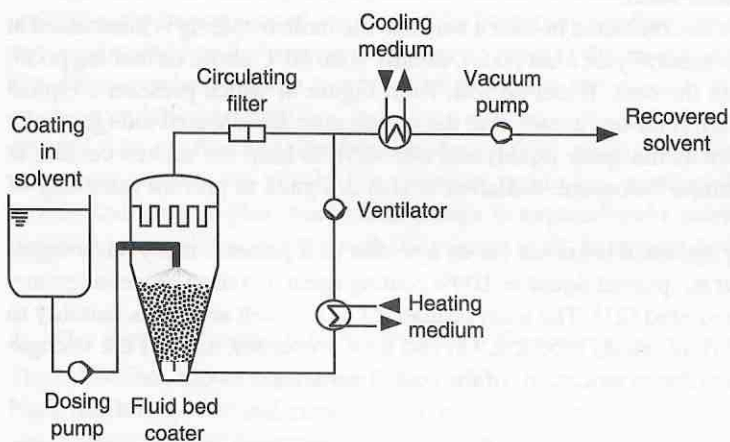

Figure 3   Diagram of a fluid-bed installation with a simplified solvent recovery system.

To summarize, although there have been massive improvements in all aspects of this process, its application to coating in the food area is not easily accepted today in the food industry and even in other industries because the new international regulations are becoming more and more severe about solvent release in the atmosphere and their concentration in food, drugs, and cosmetic products. The explosion hazard and the cost of the installation are also unfavorable factors.

B.   Hot-Melt Coating

This second approach of dry coating was also found indirectly through the research of moisture barrier polymers or membranes. The use of some molten materials such as solid fats, waxes, and some polymers as coating materials provides a shell with very good barrier
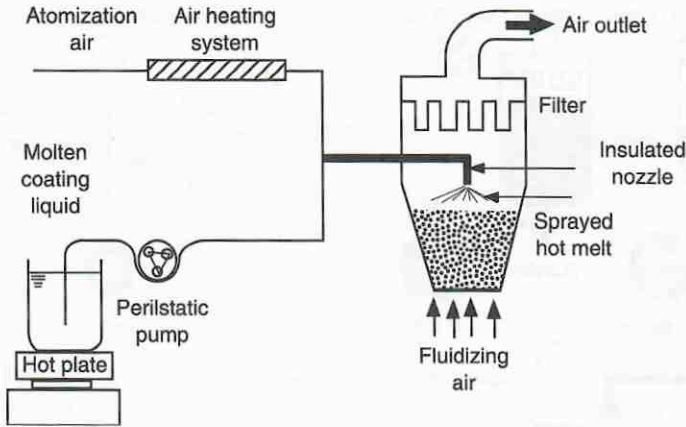
Figure 4 A conventional top spray fluid-bed installation for hot-melt coating.

properties against water vapor and gas [20]. During this process, which can take place in all types of fluid beds (top, bottom, rotor, and Wurster), the coating material, melted by heating (hot melt) is sprayed on the core and solidified directly by cold air. The core material is then never in contact with water.

The process must be conducted in such a way that the molten coating is maintained at a constant temperature (generally on a hot plate), usually 40 to 60°C above its melting point, before any contact with the core. It can be seen from Figure 4, which presents a typical installation for a hot-melt fluid-bed coater, that the atomization air is heated too (generally to the same temperature as the spray liquid) and can serve to keep the molten coating at its application temperature. Adequate insulation is also designed to prevent remelting of coated particles.

This dry coating approach is known for its low cost as it presents many advantages: (1) no solvent used, that is, sprayed liquid = 100% coating agent, (2) short processing time, (3) no drying process required [21]. The main limitations of hot melt are (1) its inability to adapt to heat-sensitive (biological) products, (2) bad flow properties, and (3) the strength and sometimes the odor (due to oxidation) of the coated particles.

C. Dry Particle Coating

As shown by the principles in Figure 1(b), this approach consists of coating a relatively large particle size (core material or host) mechanically with fine particles (guest). This leads to coated particles that can dissolve quickly in water because of their high porosity. The mechanisms of dry particles coating is described as follows [22]: initially the agglomerates of fines coating (guests) are separated into their primary particles before adhering to the host particles. This occurs rather quickly and is followed by the dispersion and rearrangement of fines spread over the surface due to collisions between coated hosts and noncoated hosts. No liquid of any kind (solvents, binders, or water) is required, that is, compared to other approaches mentioned above, dry particle coating is the true dry coating.

This process is very similar to a dry mixing of a binary mixture of powders where the smaller particles slowly adhere onto the larger particles after separation and rearrangement and cannot be easily removed from them because, as the size of the guest particles is quite small, van der Waals interactions are strong enough to keep them firmly attached to the host particles. This type of mixing is referred to as ordered mixing or structured mixing [23].
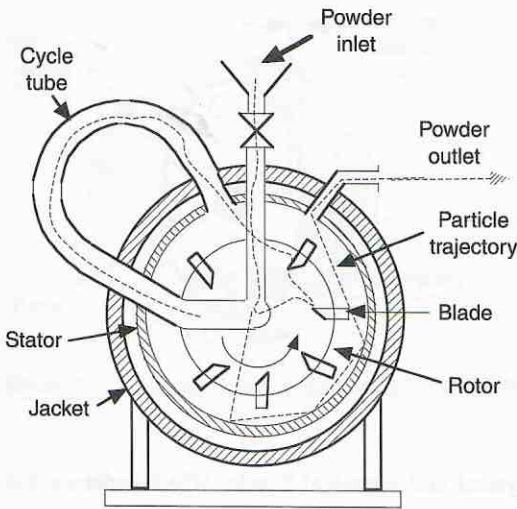
Powder
inlet

Cycle
tube

Powder
outlet

Particle
trajectory

Blade

Stator

Rotor

Jacket

Figure 5   Schematic of a hybridizer.

This similarity between dry coating and dry mixing is clearly described by Pfeffer [24] who underlines the importance of referring to the literature on ordered mixing before conducting any serious study on dry coating since the two types of drying processes are closely related. The difference between the two types of process is the strength of the bonding between the guest and the host particles. Indeed, in ordered mixing, the larger particles are loosely covered with the fine particles while in dry particle coating, the surface covering is more permanent because the physical or chemical bonding is stronger. To achieve this strong mechanical force, higher mechanical energy is required and a number of devices, whose names are generally associated with the process itself, have been designed for this purpose.

1.   Dry Impact Blending (Mechanochemical Treatment or Hybridization)

The hybridizer, shown schematically in Figure 5, was developed by the Japanese company, Nara Machinery Co. and consists of a very high-speed rotating rotor with six blades, a stator, and a powder recirculation circuit. The powder (host and guest particles) placed in the blending chamber is subjected to high impaction and dispersion due to the high rotating speed of the rotor. This agitation breaks down its agglomerates and produces an electric charge by contact and collisions between particles leading to embedding of the fine guest particles onto the surface of the host particles (this phenomenon is called hybridization or mechanochemical treatment).

2.   Mechanofusion

A mechanofusion machine is shown in Figure 6. Its principle is almost the same as a hybridizer with the difference that the outer vessel rotates whereas the inner piece and a scraper are stationary. A measured amount of host and guest particles is placed into the rotating vessel. As the vessel rotates at speeds between 200 and 1600 rpm, the particles, which are forced to pass through the gap between the inner piece and the rotating drum are submitted to intense shearing and compressive forces. These forces generate sufficient heat energy to "fuse" the guest particles onto the surface of the host particles (mechanofusion).
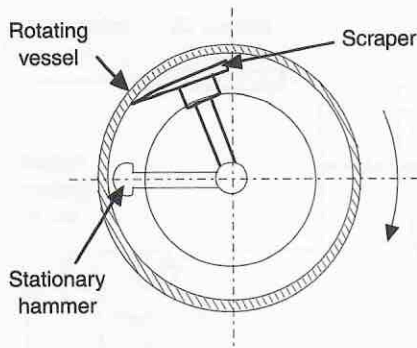
Figure 6 Schematic of the mechanofusion machine.

Mechanofusion produces very strong physical and chemical bonds, which enhance the coating process.

More details about the two above-mentioned devices can be found in [24,25], but it has to be kept in mind that they present short processing time and require such high-energy throughput that the internal temperature of the process can rise up to 60°C, a characteristic that can be useful for mechanofusion or filming of the coating shell by fusion. However, they may not be appropriate for certain applications if there are special constraints such as very low temperature range, material hardness, and cost factors that need to be considered. This is the case for many food and pharmaceutical ingredients, which, being organic and relatively soft, are very sensitive to heat and can quite easily be deformed by severe mechanical forces [24]. For such products, there is real need for a soft dry coating system where the guest particles are attached to the host particles with minimum degradation of particle size, shape, and composition. The literature inventory shows three soft techniques that have been designed for such applications. They are briefly described below from the hardest to the softest.

3. High-Speed Elliptical-Rotor Type Mixer (HEM or Theta Composer)

A schematic cross-section of the HEM is presented in Figure 7. It consists of a slow rotating elliptical vessel (around 30 rpm) and a faster (500 to 3000 rpm) elliptical rotor. The rotor and the vessel rotate coaxially in opposite directions and the powder mixture inside the vessel, consisting of host and guest particles, is subjected to shear and compressive stresses as it is forced into the small clearance between the vessel and the rotor. Successive and repeated feeding of the minimum clearance region with the powder mixture lead to strong immobilization of the guest particles on the surface of the host particles [24,27].

4. Rotating Fluidized Bed Coater

This newly developed coating device is described by Pfeffer [24] as a system operating on the principle of a rotating fluidized bed coater (RFBC). The host and guest powder mixture are placed into the rotating bed and is fluidized by the radial flow of gas through the porous wall of the cylindrical distributor, as seen in Figure 8. Due to high rotating speeds, very high centrifugal and shear forces are developed within the fluidized gas-powder system leading to the breakup of the agglomerates of the guest particles and their adhesion on the host particles.
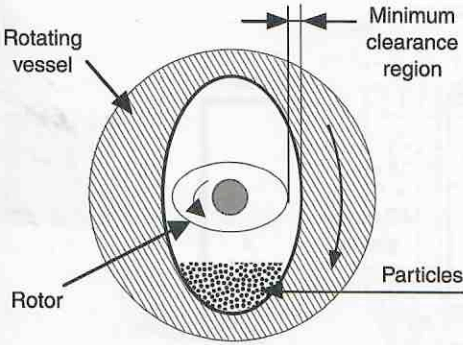
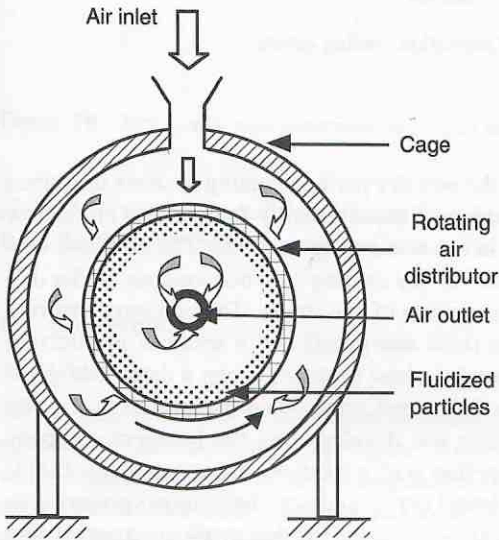**Figure 7** Schematic cross section of a high-speed elliptical-rotor mixer (HEM).

Figure 8 A rotating fluidized bed coater.

5. Magnetically Assisted Impaction Coater

A schematic of the magnetically assisted impaction coater (MAIC) device, proposed by Ramlakhan [26], is shown in Figure 9. A measured amount of magnetic, host and guest particles are placed into a processing vessel (200 ml glass bottle). The magnetic particles are made of barium ferrite and coated with polyurethane to help prevent contamination of the coated particles. An external oscillating magnetic field is created using a series of electromagnets surrounding the processing vessel. When a magnetic field is created, the magnetic particles are excited and move furiously inside the vessel resembling a gas-fluidized bed system, but without the flowing gas. These agitated magnetic particles then impart energy to the host and guest particles, causing collisions and allowing coating to be achieved by means of impaction or peening of the guest particles onto the host particles. Note that this apparatus is very versatile (i.e., can operate both as a batch and continuous system and for various types of materials) and generates no increase in temperature of the material although there is some heat generated on a microscopic level.
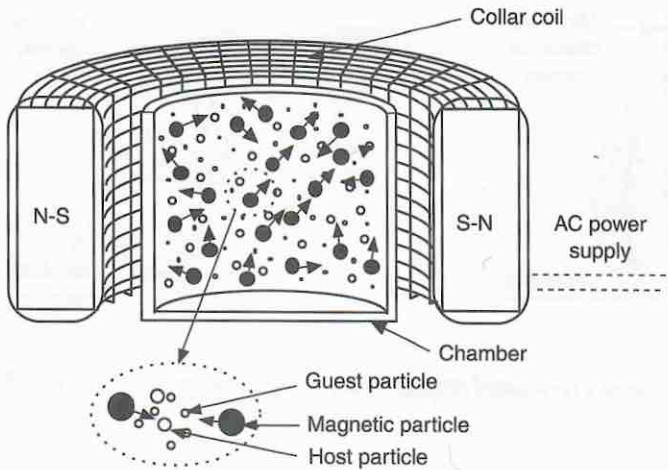
Figure 9   Schematic of a magnetically assisted impaction coating device.

D.   Use of Plasticizer

Despite the improvement brought about by the soft dry particle coating systems described above, their application to certain foods or ingredients, especiallly food, is still critical for various reasons, particularly the investment in a new blending machine. The idea here is to simply replace the required mechanical forces for dry coating by a nonaqueous binder or a plasticizer (Figure 1[c]). The coating process consists of two stages. The first stage involves feeding simultaneously the powder mixture (host and guest) and a spray of plasticizing mixture. At this stage, the layer formed around the host particles is just a deposit of guest particles. This deposit is turned into film in the second stage by curing, which is done by heating for a short time. This novel technique was developed by the Japanese company Shin-Etsu for enteric coating of tablets and pellets with a mixture of guest particles (Shin-Etsu AQOAT and talk) using a mixture of triethyl citrate and acetylated mono glyceride as plasticizer [11–28]. Note that the technique can be extended to other applications providing the formulation of suitable guest particles and plasticizer for a given purpose. The technique was quite welcomed in the food and pharmaceutical industries because of obvious advantages: (1) no deformation of the resulting coated particles and (2) no investment in one of the new blending machines mentioned above as the process can take place in any conventional coating system (Figure 2) providing a powder feeding unit as specified in Figure 10.

V.   COATING OF CRYSTALLINE CELLULOSE GRANULES USING AQOAT — A CASE STUDY

Our research was concentrated on the characterization of coated crystalline cellulose granules using the dry-coating technique developed by Shin-Etsu. As mentioned above, a large and growing number of companies are trying to adapt this technology to various applications (powders or granular materials), so knowledge of the parameters for its optimum application is needed. The objective of this study, which was done partly in collaboration with Glatt Pharmatech-Binzen, was to compare the water content in the beads during coating with aqueous and dry-coating systems with particular reference to coating water-sensitive products. The experiments were carried on successfully with a modified pan coater
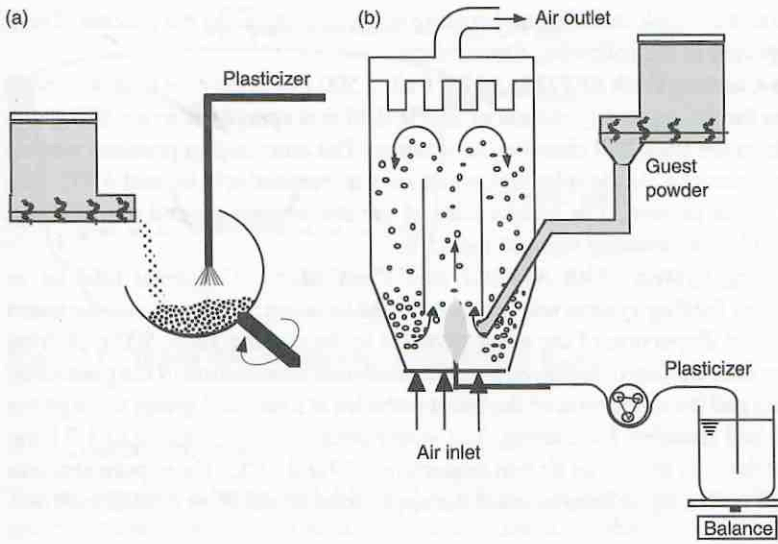
Figure 10   Dry feeder units associated to (a) pan dry coater and (b) Wurster dry coater.

(Figure 10[a]) and a modified Wurster fluid-bed coater (Figure 10[b]) but only the results from the fluid bed will be presented here.

A. Experiment

1. Materials

White spherical beads of crystalline cellulose (IPS – Cellts), with mean particle diameter equal to 835 μm and density 828 kg/m^3, were used as core materials (host particles). Their water content (6%) was too high compared to the recommended 0.5% for probiotics. So, in order to assimilate these core materials to probiotics, the latter were dehydrated in a drying oven for 48 h at 100°C and cooled after using a desiccator before coating.

A 3% solution of SEPIFILM LP 010 (SEPIC, Paris — France) was used as coating solution for aqueous coating. SEPIFILM consists of hydroxypropyl methylcellulose, microcrystalline cellulose, and stearatic acid.

The materials for dry film coating were a combination of coating polymer powders AQOAT (SYNTAPHARM — Mülhein — Germany), consisting of hydroxypropyl methylcellulose acetate succinate and talk (guest particles), and a liquid plasticizer (triethyl citrate — Sigma and acetylated monoglyceride) in the following proportion: 7 ml of plasticizer for 30 g of AQOAT. The particle size of the guest particles was not more than 10 μm.

Adequate dyes were added to the SEPIFILM solution and the plasticizer solution to color the formed coating shell. This precaution was necessary for the dissolving test.

A classical lab-scale Wurster (a bottom-spray coater), UNIGLATT (Glatt-Binzen) was used for aqueous coating. A powder-feeding device was adapted to the system for dry coating as specified in Figure 10(b).

2. Procedure

Different operating parameters of the fluid-bed coater (flow rate of the plasticizer through the nozzle, air pressure for atomization, and powder feed rate) for aqueous and dry coating

were optimized to reduce agglomeration and coating material lost during the process. These parameters are reported in the following descriptions.

a. Aqueous-Coating With SEPIFILM LP 010.   500 g of dried core materials were first fluidized, then the 3% aqueous solution of SEPIFILM was sprayed at a rate of 5 g/min through the nozzle in the fluid-bed chamber for coating. The atomization pressure was set to 1.5 bars. The temperature of the inlet and outlet air was respectively 60 and 42°C. The experiment was run for 60 min. The temperature of the atmosphere around the fluid bed was 20°C and the relative humidity was around 45%.

b. Dry-Coating System With AQOAT and Plasticizer.   The metal tube ($d = 1.5$ cm) of the powder feeding system was first positioned between the nozzle and the insert to allow homogeneous dispersion of the guest particles in the coating zone. 500 g of dried core materials were then fluidized, followed by a simultaneous atomization of the plasticizer at a rate of 1 ml/min and the dispersion of the guest particles at a rate of 2 g/min through the nozzle in the fluid-bed chamber for coating. The atomization pressure was set to 1.5 bars. The temperature of the inlet and outlet air was respectively 40 and 27°C. The experiment was run for 30 min with no curing to keep in mind the application to sensitive neutraceuticals.

3. Analysis

a. Physical Properties of the Granules.   The water content of different granules was determined by the classical gravity method. Samples (approximately 10 g of the encapsulated beads) were maintained at 100°C for 48 h in the oven and were weighed until constant weight.

The diameter was measured using a graduated microscope. The mean diameter was calculated from the diameter measurements of 50 beads. The thickness of the membrane was calculated as the difference between the initial and the final diameter of the granules.

b. Permeability of the Granules.   Different methods exist for measuring the permeability of water vapor through a membrane. The principle of these methods is to determine the quantity of water that diffuses through the film or the membrane separating the humid space from the dry space. This principle was not applied here because large films of coating materials could not be elaborated. So it was decided to measure the hydration of granules during storage in a relatively dry medium.

This hydration was measured using a desiccator. It consists of a small box filled with $Mg(NO_3)_2$ and covered hermetically by a stopper (Figure 11). The granules (10 g approximately) were put in a cup, weighed, and placed in the center of the box. They were allowed to stand for 48 and 168 h in the closed box at room temperature (~25°C). The final weight of the granules was measured for the calculation of the water uptake. Note that at 25°C, the $Mg(NO_3)_2$ provided a humidity of about 50% RH in the box.

c. Dispersion in Water.   For this experiment, 1 g of colored granules was poured into 50 ml of water and the solution was mixed using a magnetic stirrer (800 rpm). The experiment was run and stopped when the colored coating shell disappeared or when the white core beads appeared. This lapse of time was recorded as dispersion time.

VI.   RESULTS AND DISCUSSION

The results are presented here as a comparison to the aqueous coating and the dry-coating systems. This approach may appear inadequate with regard to some parameters but it finds its justification in the fact that SEPIFILM and AQOAT are closed in composition and were both designed to improve the stability of moisture-sensitive ingredients.
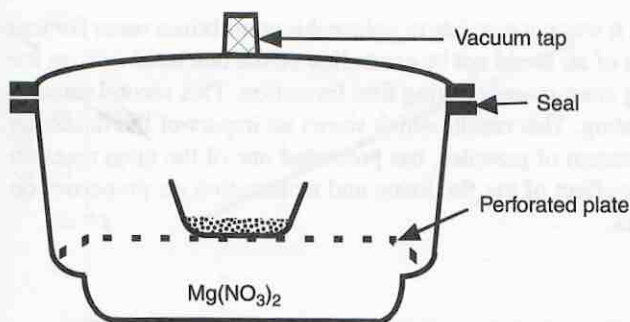
Figure 11   Testing device of granules permeability or hydration.

A.   Coating Processes

During aqueous coating with SEPIFILM LP 010, a dried coating solution was collected on the insert cylinder wall as a thin "paper" sheet, so that the coating material was lost at the end of the experiments. Despite different optimization strategies, this "paper" sheet could not be avoided during coating. At least its quantity could be reduced drastically to approximately 3% of the total coating material when the coating liquid was batch fed, that is, the coating liquid was alternatively sprayed in the system for 5 min and stopped for 5 min allowing some drying. The final mean diameter of the coated granule was 856 μm, that is, a thickness of about 10.5 μm.

It was observed during preliminary experiments of this dry coating with AQOAT that, over a coating flow rate of 2 g/min and a plasticizer flow rate of 1 ml/min, there was agglomeration of granules and a great loss of the guest particles, which were dispersed in the fluid-bed chamber before finally getting stuck on the air filter of the fluid bed. This underlines the difficulty in performing such an experiment that is rather described as simple by Shin-Etsu [28] and the necessity to establish optimum operating conditions for each case. The final mean diameter of the coated granule was 870 μm, that is, a thickness of about 17.5 μm and a coating shell, which consisted of a double layer of guest particles, assuming an ordered adhesion of the guest particles.

Dry coating was a quick process. Its processing time was half of the aqueous coating time and led to a shell thickness that was about two times larger than the SEPIFILM membrane. But its efficiency was much less than that of conventional aqueous coating (96%). Indeed, despite the use of optimized parameters mentioned above, the efficiency of dry coating was 75% — the efficiency was calculated as actual weight gain of coated samples divided by the theoretical weight gain. Note that, it is reported [11] that the efficiency can be increased to more than 95%.

B.   Hydration During Coating Processes

As mentioned above, the core materials were introduced in the fluid-bed chamber at water content optimistically equal to zero. During coating with SEPIFILM, samples of granules were collected for every 100 ml of coating solution and tested to determine their water content. It appears that the granules were always at 4% water content whatever the added quantity of the coating material, while the water content of the granules was 2% during dry coating with AQOAT. This means that granules absorb water during coating and that, with the above process parameters, the air conditions in the fluid-bed chamber were in such a way that the equilibrium water content of particles in the chamber was 4 and 2% respectively

during aqueous and dry coating. It was not possible to reduce this equilibrium water content because the initial water content of air could not be controlled on the one hand and, on the other, the water was evaporating continuously during film formation. This second cause is especially valid for aqueous coating. This result, which shows an impact of the fluidizing air properties on the moisture content of granules, has promoted one of the main research topics of our research work: the effect of the fluidizing and atomization air properties on the quality of the coated particles.

C. Permeability of the Shell (Hydration of Granules During Storage)

The hidden objectives of these experiments was to increase the shelf life of moisture-sensitive product probiotics by reducing the transfer of water vapor in the capsule or the permeability of the coating shell. The permeability of the shell was simply evaluated through the hydration of the granules as mentioned in the sections on materials and procedures. So, the measurement of the final moisture contents after storage showed that the core beads with and without coating membrane absorb the same ratio of water (~6%) after 48 h. But, after 168 h, this ratio increases to about 8% for beads without membrane and 6% for SEPIFILM- and AQOAT-coated granules. Now, considering the fact that the capsule consists mainly of the core material, it can be said that the SEPIFILM and the AQOAT can protect the core against water vapor, but not sufficiently because the difference of water content between beads with and without the membrane was about 2%. This protection may be improved by an increase of plasticizer concentration as demonstrated by Park and Chinnan [29], who found that the water vapor barrier properties of cellulose films increase as the concentration of plasticizer (Myvacet 7-00 TM) increases, while investigating the gas and water vapor barrier properties of edible films.

Finally, the protection by both SEPIFILM and AQOAT are similar and are not sufficient. So, the only way to retain the advantage brought by about dry coating (low hydration during the coating process) is to provide adequate storage. The relatively high water content of coated particles and their hydration during the dry-coating process and storage underline future challenges to be overcome in order to apply this technology to water-sensitive products.

D. Dispersion of the Coating Shell in Water

The results are given here as disintegrating time of the coating membrane in water. The final SEPIFILM-coated granules could be dissolved in water in 90 sec (Figure 12), while the AQOAT granules totally disintegrated after 5 min, probably due to the difference in coating thickness (10.5 and 17 μm respectively). Indeed, the time necessary for dissolving the membrane depends on the thickness of the SEPIFILM shell (Figure 12).

Knowing that the thickness is a function of the mass of coating material, it can be said that there is a linear relationship between the thickness of the film and the disintegration time of the coating shell. But the thickness alone cannot explain this difference in disintegration time, and other factors such as the composition of the shell and the difference in moisture content level in the granule after coating (4 and 2% respectively) can be pointed out. Also note that the disintegration time of the AQOAT-coated film may have been longer if the coating was followed by a curing step, which normally reduces the permeability of the shell.
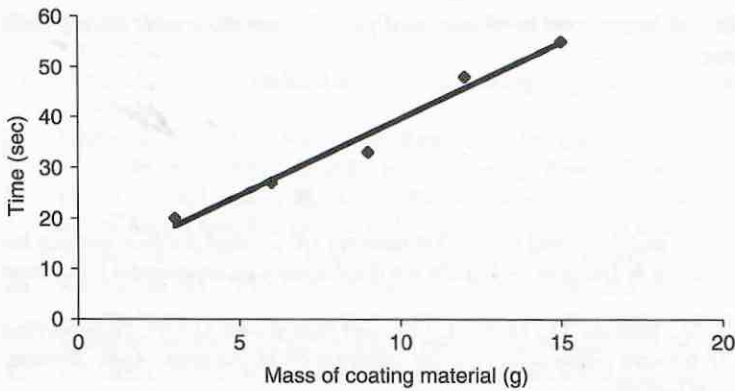
Figure 12   Disintegrating time of granules shell in water versus the thickness of SEPIFILM.

VII.   CONCLUSION AND PERSPECTIVES

Dry-coating processes, where the fine guest particles are spread over the surface of the host particles (core) due to collisions and curing (mechanofusion), without any water, solvent, or binders, are probably the best for coating moisture-sensitive ingredients. A number of works have been reviewed and they reveal a real infatuation for this type of coating processes during the last ten years, owing to the interesting potential that they present for various industrial applications. Unfortunately, their implementation requires an investment in a specific blending machine and higher energy throughput that renders them ineffective for coating soft products such as food ingredients. Some major innovations have been made to develop softer and cheaper dry-coating systems, but the use of plasticizer is the technique that looks promising, from all points of view, for coating food ingredients. This technique has shown low moisture levels compared with aqueous coating systems. However, a very small level of water content was detected in the granules (2%), and this is not acceptable for application to some sensitive products such as probiotics. That is why further experiments must concentrate in designing an air-dry system in order to improve the processing conditions (e.g., drying of the fluidizing and atomization air), leading to very low moisture content (less than 0.5%) of the final granules.

With reference to the properties of the granules, it appears that there is a need for a powerful method that will allow measuring without doubt the percentage of water that can cross a given film on a coating particle. The absence of this method can explain some contradictory results such as the AQOAT shell, which could not be dissolved easily in water and could not protect the core against water vapor. The emphasis must also be on the formulation aspect to find the suitable combination and composition of plasticizers and guest particles that can fulfill the two opposite objectives (fast solubility in water and barrier for water vapor). Hence, beyond all the functional properties of the granules the testing of which is required to check the success of the coating process, the strength of the coated granules is another parameter that must be taken into account when dealing with dry coating. Outstanding research works have been published on this topic [30,31], analyzing the strength of agglomerates and presenting different testing methods; but the selected method should be adequate to report the binding strength of the guest particles to the host particles' surface, for example, an adapted abrasion test was actually developed in our laboratory for this purpose, which was designed in such a way that the mechanisms

of constraint during the test correspond to what coated particles can encounter during their handling and processing.

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