Training Papers
Spray Drying

Contents

English, Version B (19 pages)	**Order Code**
Spray drying	**97758**

1 What is spray drying

1. Introduction

Spray drying is a very widely applied, technical method used to dry aqueous or organic solutions, emulsions etc., in industrial chemistry and food industry. Dry milk powder, detergents and dyes are just a few spray dried products currently available. Spray drying can be used to preserve food or simply as a quick drying method. It also provides the advantage of weight and volume reduction. It is the transformation of feed from a fluid state into a dried particulate form by spraying the feed into a hot drying medium. Intensive research and development during the last two decades has resulted in spray drying becoming a highly competitive means of drying a wide variety of products. The range of product applications continues to expand, so that today spray drying has connections with many things we use daily.

2. Spray drying principle

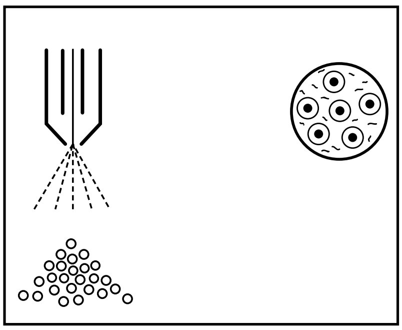
Spray drying involves evaporation of moisture from an atomised feed by mixing the spray and the drying medium. The drying medium is typically air. The drying proceeds until the desired moisture content is reached in the sprayed particles and the product is then separated from the air. The mixture being sprayed can be a solvent, emulsion, suspension or dispersion.

2.1 Dispersion of the feed solution in small droplets

The complete process of spray drying basically consists of a sequence of four processes:

The dispersion can be achieved with a pressure nozzle, a two fluid nozzle, a rotary disk atomiser or an ultrasonic nozzle. So different kinds of energy can be used to disperse the liquid body into fine particles. The selection upon the atomiser type depends upon the nature and amount of feed and the desired characteristics of the dried product. The higher the energy for the dispersion, the smaller are the generated droplets.

Nozzle and product



Example:

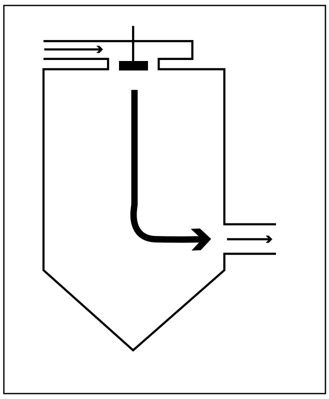
100 ml of a solution are sprayed, resulting in approx. 8×10^8 = 800,000,000 drops (25 microns) representing approx. 12 m^2 of surface area. This clearly demonstrates that the solvent (mainly water) is vaporized extremely quickly.

2.2 Mixing of spray and drying medium (air) with heat and mass transfer

The manner in which spray contacts the drying air is an important factor in spray dryer design, as this has great bearing on dried product properties by influencing droplet behaviour during drying.

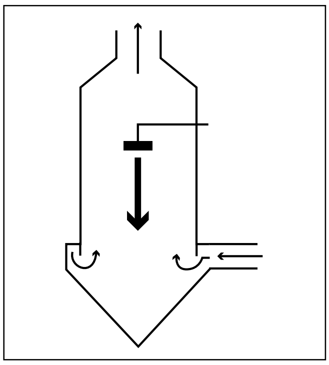
This mixing is an important aspect and defines the method of spray drying:

Co-Current flow



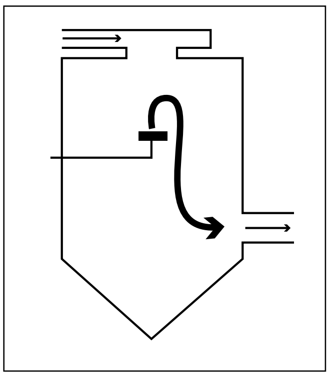
The material is sprayed in the same direction as the flow of hot air through the apparatus. The droplets come into contact with the hot drying air when they are the most moist. The product is treated with care due to the sudden vaporization.

Counter-Current flow



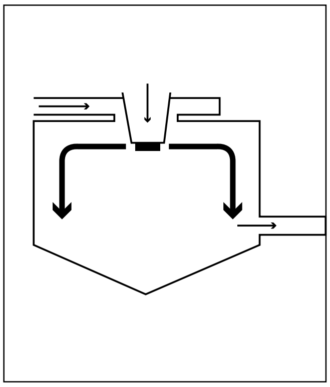
The material is sprayed in the opposite direction of the flow of hot air. The hot air flows upwards and the product falls through increasingly hot air into the collection tray. The residual moisture is eliminated, and the product becomes very hot. This method is suitable only for thermally stabile products.

Combined



The advantages of both spraying methods are combined. The product is sprayed upwards and only remains in the hot zone for a short time to eliminate the residual moisture. Gravity then pulls the product into the cooler zone. Due to the fact that the product is only in the hot zone for a short time, the product is treated with care.

Disk atomizer (rotary wheel)



The material to be sprayed flows onto a rapidly rotating atomizing disk and is converted to a fine mist. The drying air flows in the same direction. The product is treated with care, just as in the co-current flow method.

2.3 Open-cycle and closed cycle system

Air is mostly used as drying medium. The air stream is heated electrically or in a burner and after the process exhausted to atmosphere. This is a open-cycle system. If the heating medium is recycled and reused, typically an inert gas such as nitrogen, this is a closed-cycle system. These layout is typically chosen, when flammable solvents, toxic products or oxygen sensitive products are processed.

The most common type of spray dryer is the open-cycle, co-current spray dryer. In such a design, the atomised feed and the drying air is simultaneously injected into a spray drying chamber from the same direction.

2.4 Drying of spray (removal of moisture)

As soon as droplets of the spray come into contact with the drying air, evaporation takes place from the saturated vapour film which is quickly established at the droplet surface. Due to the high specific surface area and the existing temperature and moisture gradients, an intense heat and mass transfer results in an efficient drying. The evaporation leads to a cooling of the droplet and thus to a small thermal load. Drying chamber design and air flow rate provide a droplet residence time in the chamber, so that the desired droplet moisture removal is completed and product removed from the dryer before product temperatures can rise to the outlet drying air temperature. Hence, there is little likelihood of heat damage to the product.

2.4.1 Separation of product and air

In principal, two system are used to separate the product from the drying medium:

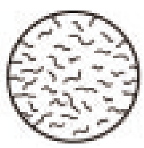
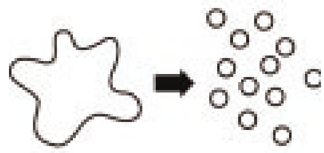
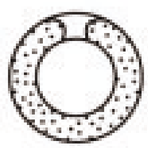
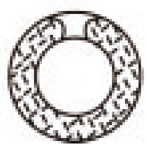
1 Primary separation of the drying product takes place at the base of the drying chamber
2 Total recovery of the dried product in the separation equipment

Most common separation equipment is the cyclone. Based on inertial forces, the particles are separated to the cyclone wall as a down-going strain and removed. Other systems are electrostatic precipitators , textile (bag) filters or wet collectors like scrubbers.

3. General applications

The list of materials which are successfully spray dried is enormous, so only general principles should be listed hereby:

Possible applications

Application	Goal / use	Practicalapplication
Spray drying 	Drying of inorganic and organic products	corn starch pigments dried milk
Micronization 	Reduction of a product's par- ticle size	salt dyes
Micro encapsulation 	A liquid product is embed- ded in a solid matrix	perfumes strawberry aroma peach oil
Englobing 	A solid product is embed- ded in another solid or a mixture of solids	carotenoids in gelatins

4 Mini Spray Dryer

4.1 Spray Drying with the BÜCHI Mini Spray Dryer B-290

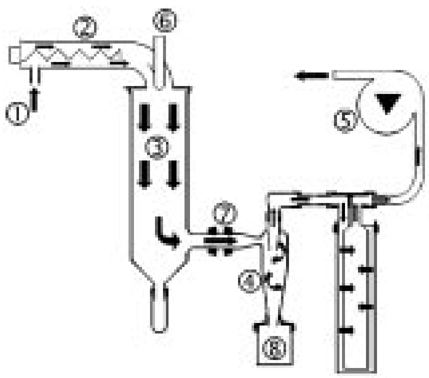
The Mini Spray Dryer B-290 is a laboratory scale system to perform spray drying processes down to 50 ml batch volume and up to 1 litre solution per hour. Due to the glassware, the complete drying process from the two-fluid nozzle down to the collection vessel is visible. Even a lot of fundamental investigations of the spray drying process has been undertaken, it still remains a step with some uncertainties and difficulties to model. One reason is the big influence of material properties and drying behaviour of the product and another is the complex fluid dynamics in a spray dryer.

Thus, small scale feasibility studies and trials are an often used approach to win some experience with a certain product to spray dry. Even the direct scale-up from a lab-bench unit to a big system cannot be easily made, it helps to understand and quantify the drying behaviour. For small batch sizes e.g. in pharmaceutical applications, a small spray dryer is particularly interesting to win small product volumes within a short time. Thermolabile components such as enzymes or antibiotics remain fully active.

4.2 Design of the Instrument

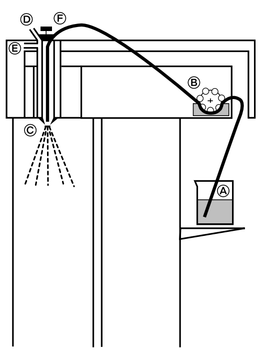
The Mini Spray Dryer B-290 functions according to the same principle as the co-current flow atomizer, i.e. the sprayed product and drying air flow are in the same direction.

4.2.1 Diagram of the dry air flow



1 Air intake
2 Heater
3 Flow stabilizer intake into the drying chamber
4 Cyclone, the product is separated from the air flow here
5 Aspirator
6 Temperature sensor, air inlet
7 Temperature sensor, air outlet
8 Container for collecting finished product

4.2.2 Diagram of the product flow and spray nozzle



A Solution, emulsion or dispersion of the product
B Peristaltic feed pump
C Two fluid nozzle (spray mist, spray cone)
D Compressed air or inert gas supply connection
E Cooling water connection
F Nozzle cleaning device, consisting of needle pneumatically pushed trough nozzle

4.3 Instrument settings

Spray Drying is a method where the result strongly depends upon the material properties. Thus, the instrument settings, namely inlet temperature, feed rate, spray air flow and aspirator flow are in a combined system influencing the product parameters:

- Temperature load
- Final humidity
- Particle size
- Yield

The optimisation of these parameters are usually made in a ”Trial & Error” process. Some initial conditions can be found in the application database for equal or similar products.

parameter / dependence	aspirator rate ↑	air humidity ↑	inlet temperature ↑	spray air flow ↑	feed rate ↑	solvent instead of water	concentration ↑
outlet temperature	↑↑ less heat losses based on total inlet of energy	↑ more energy stored in humidity	↑↑↑ direct proportion	↓ more cool air to be heated up	↓↓ more solvent to be evaporated	↑↑↑ less heat of energy of solvent	↑↑ less water to be evaporated
particle size	-	-	-	↓↓↓ more energy for fluid dispersion	(↑) more fluid to disperse	(↓) less surface tension	↑↑↑ more remaining product
final humidity of product	↑↑lower partial pressure of evaporated water	↑↑ higher partial pressure of drying air	↓↓ lower relative humidity in air	-	↑↑ more water leads to higher particel pressure	↓↓↓ no water in feed leads to very dry product	↓ less water evaporated, lower partial pressure
yield	↑↑ better separation rate in cyclone	(↓) more humidity can lead to sticking product	(↑) eventually dryer product prevent sticking	-	(↓↑) depends on application	↑↑ no hygroscopic behaviour leads to easier dying	↑ bigger particles lead to higher separation

4.3.1 Interaction of the individual parameters

- Larger temperature differences between the inlet and outlet temperatures result in a larger amount of residual moisture.
- A high aspirator speed means a shorter residence time in the device and results in a larger amount of residual moisture.
- A high aspirator speed results in a higher degree of separation in the cyclone.
- Higher spray flow rates tend to result in smaller particles.
- Higher spray concentrations result in larger particles.
- Higher pump speed, result in a lower outlet temperature.

4.3.2 Inlet temperature / outlet temperature

Inlet temperature is understood as being the temperature of the heated drying air. The drying air is sucked or blowed in over a heater by the aspirator. The heated air temperature is measured prior to flowing into the drying chamber. When spray drying a solution, emulsion or dispersion the solvent is removed by vaporization.

The temperature of the air flow does not have to be higher than the boiling point of water to evaporate the individual drops during the short residence time. The gradient between wet surface and not saturated gas leads to an evaporation at low temperatures. The final product is separated and has no further thermal load.

The temperature of the air with the solid particles before entering the cyclone is designated as the outlet temperature. This temperature is the resultig temperature of the heat and mass balance in the drying cylinder and thus cannot be regulated. Due to the intese heat and mass transfer and the loss of humidity, the particles can be regarded to have the same temperature as the gas. Thus, as a rule of thumb is: outlet temperature = max. product temperature.

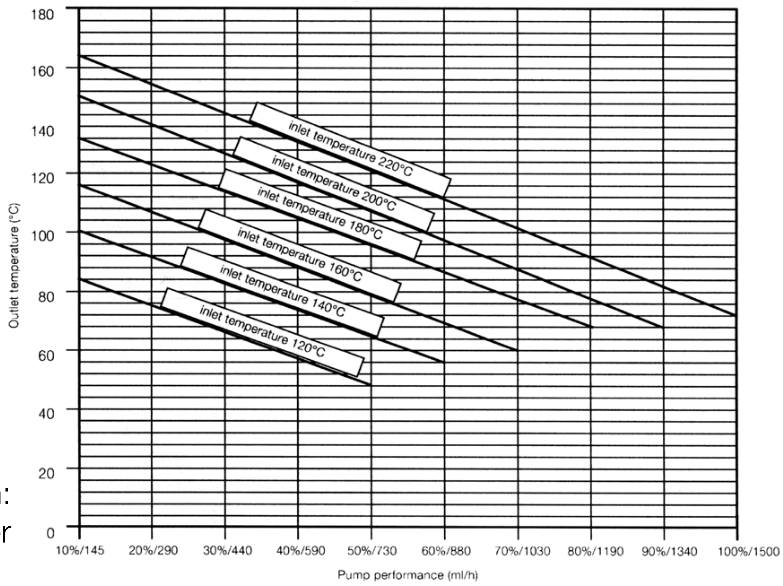
The outlet temperature is the result of the combination of the following parameters:

- Inlet temperature
- Aspirator flow rate (quantity of air)
- Peristaltic pump setting
- Concentration of the material being sprayed

The optimal choice for the temperature difference between the inlet and the outlet temperature is one of the most important points to consider when spray drying. Of course, other product specific factors, such as the melting point or decay temperature, must be taken into consideration. In spite of this, there is still some room for adjustment. The throughput of the device as well as the residual moisture content can be influenced within this temperature difference range.

The following table shows the interaction between the inlet and outlet temperatures, depending on the pump throughput. The following guidelines can be derived from the data:

Outlet temperature depending on the pump performance for various inlet temperatures.



Spray medium:
distilled water

For a final product with a very small amount of residual moisture, the inlet temperature must be as high as possible and the temperature difference must be as small as possible.

Increasing the temperature difference while holding the inlet temperature constant increases the residual moisture content in the final product as well as the spray flow rate of the device.

4.3.3 Aspirator

The drying air is sucked or blown through the device by the aspirator motor creating under pressure conditions. By regulating the aspirator speed, the amount of heated drying air can be increased or decreased. If the system is running in the sucking mode, a slight underpressure will take effect in the spray dryer. Because the amount of energy available for vaporization changes when the amount of drying air is increased or decreased, the aspirator speed setting has a significant effect on the drying performance of the device.

The optimum setting must be determined experimentally using the following guidelines:

High aspirator speed → higher degree of separation in the cyclone

Lower aspirator speed → lower residual moisture content

4.3.4 Pump performance

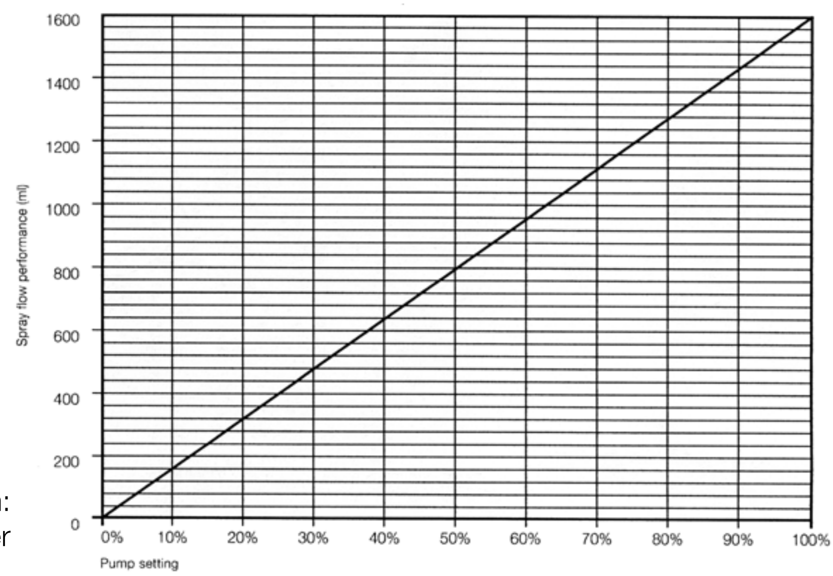
The peristaltic pump feeds the spray solution to the nozzle. The pump's speed affects the temperature difference between the inlet temperature and the outlet temperature. The pump rate directly corresponds to the inlet mass. The higher the throughput of solution, the more energy is needed to evaporate the droplet to particles.

Thus, the outlet temperature decreases. The limitation of the pump is when the particules are not dry enough resulting in sticky product or wet walls in the cylinder. The pump throughput is also dependent upon various factors such as the viscosity of the spray solution and tubing diameter.

The following guidelines can be derived from the facts described above as they relate to the pump rate:

B-290 Spray flow performance

Spray medium: distilled water



Tube used: Silicon tube, inner diameter 2.0 mm

> Increasing the pump rate lowers the outlet temperature and thus increases the temperature difference between the inlet temperature and the outlet temperature.

> Reducing the pump rate while holding the inlet temperature and aspirator flow rate constant increases the dry content of the final product.

4.3.5 Spray flow and concentration of solution

The spray flow rate is the amount of compressed air needed to disperse the solution, emulsion or suspension. A gas other than compressed air can be used.

The spray flow rate can be set to between 300 and 800 l/h on the device. A rotameter indicates the spray flow throughput. The table below gives a correlation of the flow meter and the gas throughput.

The particle size of the final product can be influenced by the spray flow rate setting.

Height (mm)	Normlitre/hour	Pressure drop	Volume flow (real)
5	84		
10	138		
15	192		
20	246	0.15	282.9
25	301	0.18	355.18
30	357	0.23	439.11
35	414	0.3	538.2
40	473	0.41	666.93
45	536	0.55	830.8
50	601	0.75	1051.75
55	670	1.05	1373.5
60	742	1.35	1743.7
65	819	1.8	2293.2

A guideline is:

> The higher the spray flow rate, the smaller the size of the particles in the final product.

The spray concentration influences the particle size.

> The higher the concentration of the spray solution, the larger and more porous the dried particles.

5 Applications

5.1 Spray drying

Spray drying is suited for most real or colloidal solutions, for emulsions and dispersions as long as the dried product behaves like a solid.

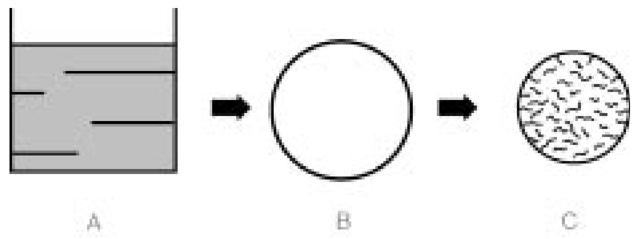


Diagram of spray drying inorganic or organic products

An aqueous solution of the product (A) is dispersed into fine droplets (B) using a two fluid nozzle. The solvent evaporates immediately surrounding the product in a vapor cloud that protects the product from thermal load. As soon as the critical concentration is exceeded, nucleation starts forming a solid shell. After the solvent is dryed away from the surface, the interface moves into the core (second step of drying).The final product (C) is a fine, amorphous or crystallized material. Spraying a highly concentrated solution results in a more porous final product.

Examples

Product	Inlet °C	Outlet °C	Spray concentration %
Foodstuffs			
Low-fat milk	174	102	50
Yeast	95	55	60
Aroma/Cosmetics			
Beer concentrate	150	110	30-40
Olive leaf extract	150	90	36
Medical/pharmaceutical			
Blood plasma	180	100	5
Peptides	110	70	2
Chemical products			
Dispersion dyes	150	95	20

5.2 Micronization or structural change

The micronization or structural change is the change of morphology, e.g. if a fine powder is needed. This has a positive effect on the solubility or measurability of the final product. The main advantage of micronization is that a very regular particle size is achieved.

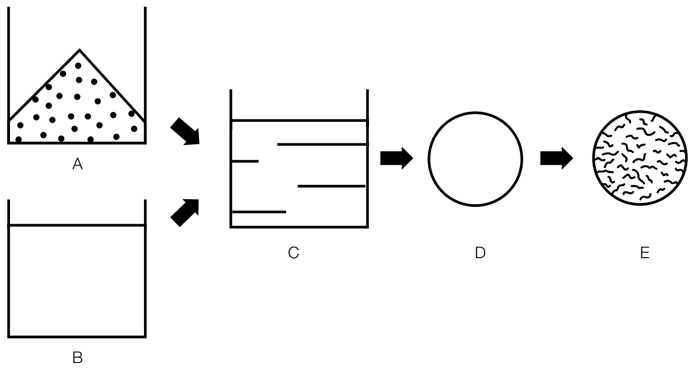


Diagram of the micronization or structural change process

The crystalline product (A) is dissolved in a solvent (B) and this solution (C) is dispersed into small droplets (D). The result is a final product (E) just like the final product described in the section on spray drying.

Examples

Product	**Inlet °C**	**Outlet °C**	**Spray concentration %**
Foodstuffs			
Lactose	160	105	30
Corn starch	130	70	40
Aroma/Cosmetics			
Metalsoap	165	122	60
Detergent	200	110	40
Medical/pharmaceutical			
Mixed products of fructose-amino acid compounds	180	80	37
Chemical products			
Calcium carbonate	220	100	10
Sodium citrate	160	90	20

5.3 Micro encpsulation

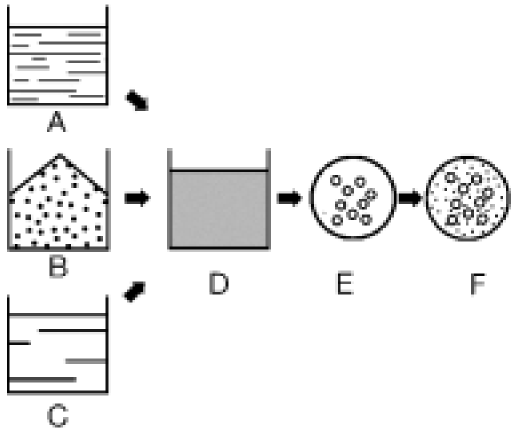


Diagram of the micro encapsulation process

An emulsion (D) is created from the liquid product to be treated (A), a carrier substance (B) such as maltodextrin and a filmogen solution (C) such as gum arabic in water. This emulsion is then sprayed into small droplets (E). The solvent evaporates leading to a solid matrix around the dispersed second phase (F). The result is that the small droplets of the product (A) are stored in the carrier substance (B) and embedded in the filmogen (C).

Examples

Product	**Inlet °C**	**Outlet °C**	**Spray concentration %**
Foodstuffs			
Soyabean oil in maltodextrin/gelatins	150	90	30
Aroma/Cosmetics			
Aroma, strawberry in maltodextrin/gelatins arabic 1,5 : 1,5 : 3	150	90	35
Medical/pharmaceutical			
Guajazulene in maltodextrin/gum arabic 1 : 2 : 1	120	70	ca. 30

5.4 Englobing

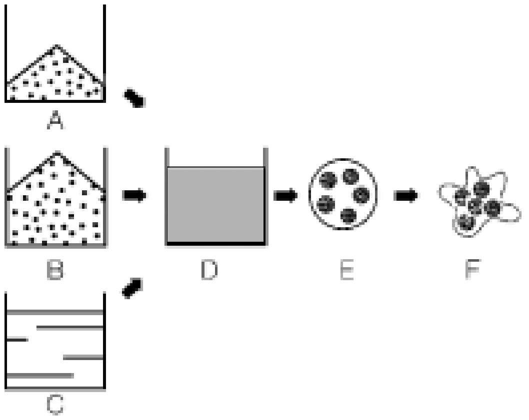


Diagram of the englobing process

The englobing process is analogous to the micro encapsulation process, whereby a solid material is used instead of a liquid product. A solution or dispersion (D) is created from the product to be treated (A), a matrix (B) and water eventually with additional filmogen (C). This solution is then sprayed into small droplets (E).The matrix and / or filmogen lead to an agglomeration or coating of the suspended particles (F).

Examples

Product	Inlet °C	Outlet °C	Spray concentration %
Foodstuffs			
Inverted sugar (date pulp) in Lactose 1:1	100	80	20
Medical/pharmaceutical			
Streptococci in low-fat milk powder/ glucose/gelatin 1:1:1:3	90	70	40

5.5 Overview of selected applications

The application depends on the kind of product used, such as viscosity, density, additives etc. Therefore, the given parameters can not previsely be overtaken

Foodstuffs

Spray drying

Product	Inlet °C	Outlet °C	Spray concentration %
Baby food	160	95	40
Beer	180	108	
Casein	150	90	6
Yeast	95	55	60
Krill	180	80	10
Lactose	160	105	30
Low-fat milk	174	102	50
Corn starch	130	70	40
Milk	110	70	15
Whey	180	80	6 /45
Soyabean extract (suspension)	130	75	80
Tofu	110	60	17

Englobing/micro encapsulation

Product	Inlet °C	Outlet °C	Spray concentration %
Fruit concentrate, raspberry, in maltodextrin,2:8	150	90	30
Fruit concentrate, orange, in maltodextrin,2:8	150	90	40
Inverted sugar (date pulp) in lactose 1:1	100	80	20
Black currant juice in maltodextrin	170	100	47
Soybean oil in maltodextrin/gelatin	150	90	30
Sugar/fat mixture in maltodextrin/ gum arabic 25:15:50:10	160	90	22

Aromas, cosmetics, cleaners and detergents

Spray drying

Product	Inlet °C	Outlet °C	Spray concentration %
Valerian extract	150	100	25
Beer concentrate	150	110	30 - 40
Chicory extract	130	75	38
Pine bark extract	120	85	4
Chestnut extract	200	130	20
Metal soap	165	122	60
Microfoam beads	160	114	3
Sodium citrate	160	90	20
Sodium orthophosphate	180	110	40
Olive leaf extract	150	90	36
Liquorice extract	100	75	36
Detergent	200	110	40
Fabric softener	125	75	20
Xanthane mixture	130	70	-
Zeolite	180	120	10

Englobing/micro encapsulation

Product	Inlet °C	Outlet °C	Spray concentration %
Aroma, strawberry in maltodextrin/ gum arabic 1,5 : 1,5 : 3	150	90	35
Aroma, orange in maltodextrin/ gum arabic 1,5 : 1,5 : 3	150	90	17
Cardamom oil in maltodextrin/ gum arabic 1 : 20 : 39	170	100	20
Date juice in maltodextrin/ gum arabic 25 : 25 : 1	120	90	30
Caraway oil in maidex/ gum arabic	140	100	50
Perfume oil in maltodextrin/ gum arabic 1 : 2 : 1	130	70	
Peach oil in maltodextrin	150	100	20
Bubble bath in sodium chloride1 : 2	160	100	15
Cinnamon oil in maltodextrin/ gum arabic 1 : 3 : 6	170	100	20
Lemon oil in maltodextrin/ gum arabic	130	90	20

Medical/Pharmaceutical

Spray drying

Product	Inlet °C	Outlet °C	Spray concentration %
Albumin	110	60	5
Lyophilized anti-progresterone serum	80	60	1
Blood plasma	180	100	5
Dextran	154	120	20
Enzymes / coenzymes	80	55	12
Fructose-amino acid compounds	180	80	37
Galactomannan	200	115	5
Gelatin capsule dispersions	105	80	20
Glucose / amino acid compounds 1:1	130	80	10
Mannitol with enzymes	100	55	15
Combination vaccines	190	140	-
Organ extracts with tetra-Na-diphosphate 1 : 0,43	150	88	11
Peptides	110	70	2
Vitamin A + E / gelatin-emulsion	100	55	-
Cell suspension (bacteria cultures)	90	60	ca. 50

Englobing/micro encapsulation

Product	Inlet °C	Outlet °C	Spray concentration %
Carotinoid in gelatin 40 : 60	170	100	25
Guajazulene in maltodextrin/ gum arabic 1 : 2 : 1	120	70	ca. 30
Streptococci in low-fat milk powder / glucose/gelatin 1 : 1 : 1 : 3	90	70	40

Chemical products

Spray drying

Product	**Inlet °C**	**Outlet °C**	**Spray concentration %**
Acrylamide	125	69	50
Albigen	180	90	10
Ammonium chloride	180	75	20
Ammonium nitrate	180	100	20
Lead oxide	150	90	—
Calciumhydrogen citrate	200	110	50
Calcium carbonate	220	100	10
Calcium phosphate	190	100	—
Dicalcium phosphate	170	90	20
Disodium phospate	200	140	50
Dispersion dyes	150	95	20
Iron oxide	170	125	—
Pigments	130	110	36
Glass powder	120	90	20
Latex rubber	120	70	20
Indigo-sodium sulfate compound	150	90	30
Potassium hydrogen citrate	200	110	50
China clay	180	130	33
Various ceramics	150	120	46
Synthetic glues	100	70	20
Latex	160	90	31
Lignin	130	55	7/4
Magnesium phosphate	120	90	15
Melamine resin	120	80	—
Metal oxide	210	135	—
Sodium citrate	160	90	20
Sodium orthophosphate	180	110	40
Sodium sulfite	180	90	20
Cermaic oxide	100	80	26
Phenolic resin	135	105	50
Polyacrylamide	204	111	3
PVC-latex	160	90	31
Clay suspension	200	100	1,2
Peat extract	120	80	1,5
Vinyl acetate polymer	90	50	25
Zeolite	180	120	10
Tin oxide	230	170	—
Zirconium oxide	180	100	—