

Preparation of spray-dried lactose and influence of the solution concentration on geometric characteristics of particles

Introduction

Spray drying is a simple and quick method in which the material in the liquid state is converted into a dry solid form by spraying it into a hot drying medium (air, inert gas). The spray-drying product is a powder, granulate or agglomerated powder. This method allows spray drying of biological and thermolabile materials, as well as hydrophilic and hydrophobic materials, low and high molecular weight compounds, including a variety of polymers. Spray-dried lactose is obtained by spray drying an aqueous solution of lactose. Thus, it is possible to obtain a final product with the required compressibility and flow properties which are suitable, for example, for direct compression of tablets.

Task 1. Preparation of spray-dried lactose

The aim of the task is to prepare spray-dried lactose from solutions of various concentrations.

Materials: α -lactose monohydrate, purified water

Equipment and aids: Büchi Mini Spray Dryer B-290 (Fig. 1), scales, beakers, filter paper

Workflow: The procedure is suitable for both concentrations

1. Prepare 250 ml of 10 % and 30 % solution of α -lactose monohydrate
2. Check the spray dryer connection to the compressed air source and turn the spray dryer and compressed air on.
3. Set air flow at approx. 30 mm using the adjustment knob on the rotameter (Fig. 2).
4. Set the parameters for drying using the arrows on the control panel (Fig. 3.)

INLET °C : 170

ASPIRATOR %: 100

PUMP %: 10

NOZZLE CLEANER: 4

Turn on the aspirator and heating (ON/OFF button)

5. Wait until the system has reached steady conditions. Place the beaker containing the solution on the panel (Fig. 1) and insert into it the feeding tube. Turn on the pump (ON/OFF button)
6. After the lactose solution has been sprayed, place a beaker with 100 ml of purified water on the panel and let it spray. This cleans the deposits in the tubing and nozzle
7. Turn off the nozzle cleaner, pump and heating. As soon as the temperature sinks below 70 °C, the aspirator can be turned off.
8. Remove the product collecting container and place the dried powder on filter paper
9. After the unit is cold, clean all the glass parts and the nozzle

Suggestion for discussion: Influence of individual parameters on the spray drying process. Possible problems that may occur during drying.

Evaluation: Spray-dried lactose samples will be further evaluated using an optical microscope



Fig. 1: Büchi Mini Spray Dryer B-290



Fig. 2: Rotameter for the air flow setting



Fig. 3: Control panel

Task 2. Evaluation of geometric characteristics of spray dried lactose particles

The aim of the task is to evaluate of geometric characteristics (shape and size) of prepared spray dried particles using optical microscopy and to monitor the influence of the solution concentration on these parameters.

Materials: spray dried particles of lactose, liquid paraffin

Equipment: optical microscope Olympus BX 51, glass slides and cover glass, small glass bottles, ultrasonic water bath, spatula

Workflow:

1. Sample preparation: put small amount of spray dried powder into the glass vial and add liquid paraffin to approx. $\frac{3}{4}$ of the volume. Close the bottle well and place it into the water bath. Turn it on and let the sample there for 5 minutes. Check continuously the disaggregation of particles. If the particles are not sufficiently disaggregated after 5 minutes, you can increase the time.
2. Put small drop of the prepared suspension on the glass slide and cover by cover glass.
3. Run analySIS auto 5.1 on the computer and tur on the microscope. Set the process parameters: magnification 20x, video camera resolution 1360x1024 px, camera resolution 4140x3096 px
4. Set the evaluated parameters: area, maximal and minimal Ferret diameter, shape factor, sphericity and Max Y
5. Place the sample in the microscope, focus and take the picture. Measure the particle size and transfer the values into a table. To take another shot, turn on the video camera and systematically locate the next position, take the picture and continue to measure the particles.
6. Sort the particles according to the Max Y value into size fraction and plot the cumulative and particle size distribution curve.

Suggestion for discussion: Influence of spray drying process parameters on properties of the powder. Use of spray dried materials.

Evaluation: Based on your results compare the geometric parameters of spray dried lactose and evaluate the influence of solution concentration on these parameters.