SPRAY (MICRO) ENCAPSULATION OF OXIDATION AND LIGHT-SENSITIVE VOLATILE SUBSTANCES IN MATRIX FORM – CASE STUDY ON ESSENTIAL OILS

Glatt Ingenieurtechnik GmbH, Weimar, Germany

Dr.-Ing. Michael Jacob, Director Process Engineering / Process Technology Food, Feed & Fine Chemicals (michael.jacob@glatt.com)

Background and target

Essential oils are volatile substances that are susceptible to oxidation and light. They change or can even lose their properties when coming into direct contact with the environment. Up to now, these phytochemicals have found numerous uses and applications.
For protection and easier handling, as well as for better dosing and targeted release properties, these substances are increasingly offered and used by various industries in an encapsulated and often free-flowing form.

In general product and process optimization becomes increased importance in all kind of industry. For instance in food, chemical and agricultural industry granular products must be developed with focus on quality and costs of formulation.

The poster reports a case study involving the spray granulation of a solution using continuous fluidized bed and spouted processing.
Based on various experiments in laboratory and pilot scale a statistical process model was derived based on artificial neural networks. A parameter study and sensitivity analysis was performed to determine the main process conditions and formulation variables. The multi-dimensional model of the process supported the optimization of product properties (e.g. particle size, particle sphericity) and related process conditions (e.g. product temperature, spray rate).

Equipment

Experimental work of case study was carried out using a laboratory scale unit from Glatt. The ProCell-Labsystem is designed modular and is very suitable for any kind of process development and evaluation.

Figure 1: ProCell Labsystem (Glatt Ingenieurtechnik GmbH)

Various processing inserts are available for batch and continuous processing, fluidized and spouted bed operation and any kind of liquid spray.

ProCell system with ProCell 5 and ProCell 10 inserts
continuous spouted bed: spray granulation, encapsulation
batch spouted bed: agglomeration, coating

Vario system with Vario 3 and Vario 7 inserts
continuous fluid bed: spray granulation, encapsulation-
batch fluid bed: agglomeration, coating, Wurster coating

GF system with GF 5 insert
continuous fluid bed:
spray granulation, encapsulation, agglomeration, coating, drying

AGT system with AGT 2 insert
continuous fluid bed:
spray granulation, encapsulation

Rotor system with Rotor 7 and CPS 7 inserts
batch fluid bed:
powder layering, spheronizing, direct pelletizing

Figure 2: Summary of processing options

(Micro) Encapsulation by spray granulation

The basic principle of encapsulation using fluidized and spouted bed principles involves spraying a liquid onto fluidized particles to wet their surface. By simultaneously drying and hardening the liquid film, the particles grow, layer by layer.

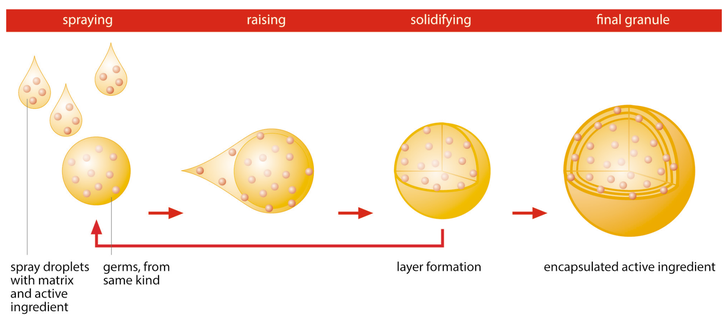

Figure 3: Principle of encapsulation

With this process, nearly spherical particles can be produced in a size range of 200 microns to a few millimeters. Microcapsules produced have a closed surface and compact structure, resulting in a stable, long-term inclusion of the active substance.

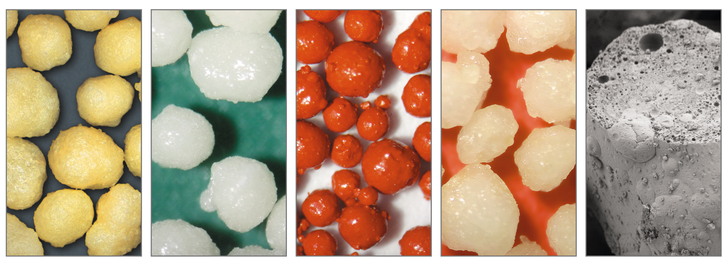

Figure 4: Product samples

Depending on final application and material properties of substances to be encapsulated two basic concepts are available for formulation.

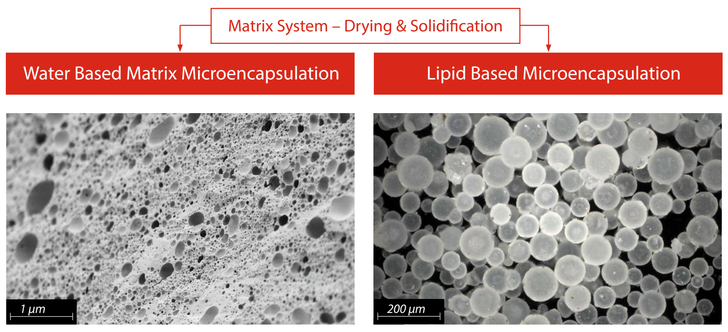

Figure 5: Formulation concepts

Water based microencapsulation of water insoluble substances utilizes emulsification into a liquid matrix system as pre-processing step. Emulsion has to be stable and droplet size distribution in proper range to ensure high recovery rates and product stability.

Using a matrix containing oil-in-water emulsion as a raw material, the continuous spray granulation in fluidized or spouted beds yields solid particles of defined size in which the oil has been very finely distributed in an optimal way.

Lipid based microencapsulation uses lipid solubility of substances to be encapsulated. In that case lipid carriers are transformed to liquid state by melting first and active substances are then mixed and dissolved. Thermal stability of ingredients have to be considered in product development.

Case-study of water based microencapsulation

Data analysis was used to study influence parameters of product composition (formulation) and processing (process conditions).

An artificial neural network (aNN) was build-up to be used as a statistical process model. In the case study 12 input parameters (e. g. water content of solution, spray rate) and 3 output parameters (e. g. yield, mean particle size) were used in the process model. Process variables (e. g. residence time, temperature, spray rate) and formulation variables (e. g. water content, type and concentration of additives, type and concentration of actives) were analyzed.
Parameter study and sensitivity analysis resulted in knowledge about sensitivity of individual process parameters and allowed complex multi-parameter analysis. Formulation and process conditions could be optimized.

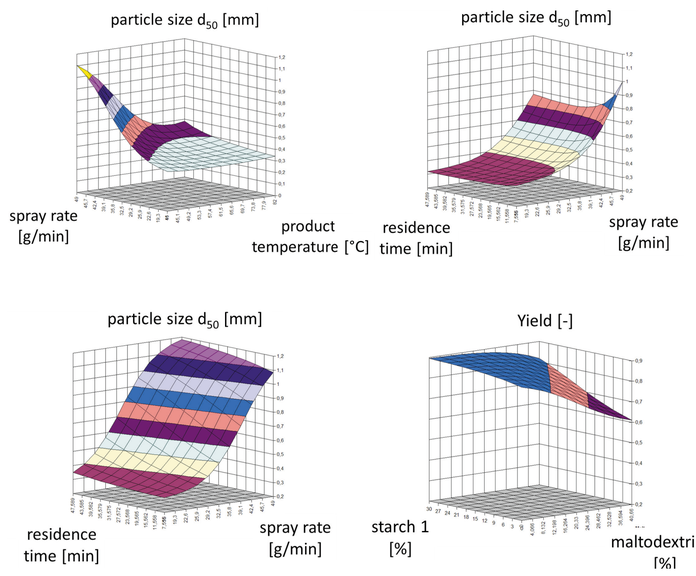

Figure 6: Results of sensitivity studies (variation of process conditions and formulation)

The case study showed the capability of the use of process models for product and process optimization. In the case of granulation very valuable information of different formulations could be pointed out.
It could be detected that process units which can be operated at low residence time are to prefer when sensitive substances have to be incorporated into a granular structure. Here the Glatt-ProCell apparatus shows advantages compared to standard fluidized beds.

Case-study of lipid based microencapsulation

Lipid based encapsulation offers an alternative approach to stabilize sensitive ingredients. Especially principle of atomization (pressure nozzle vs. binary nozzles) and composition of spray liquid influence particle morphology and product stability.

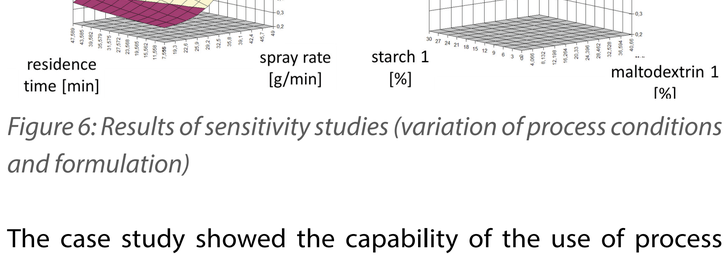

Figure 7: Shift of melting/solidification point and crystal structure of granules

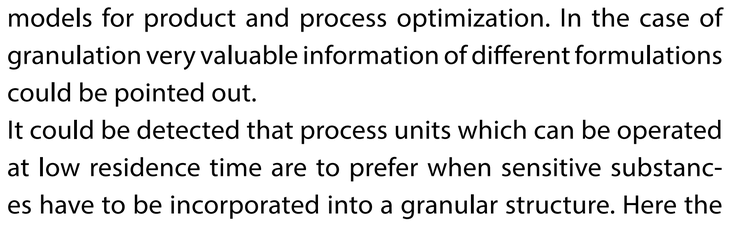

Figure 8: Particle morphology (amorphous vs. crystalline)