

# Use of high melting point solid vegetable oils in encapsulation processes

#### Introduction to encapsulation

Encapsulation relates to technologies that enable the incorporation of a compound into individualized small particles. Depending on the process applied, these particles can have different sizes, shapes and specific structures. Many kinds of active compounds can be encapsulated with many different encapsulation materials. High melting point solid vegetable oils are one of them.

The main objectives for encapsulating an active principle can be classified in 3 major topics [1]:

- 1. Protection / stabilization: encapsulation enables to reduce interactions between the active compound and the outside environment. High melting point vegetable oils are particularly efficient in protecting active ingredients against oxygen and water, preventing them from oxidation.
- 2. Functionalization / structuration: encapsulation can be very effective in taste, odor and color masking but also allows to give specific surface properties and rheological characteristics to powders, avoiding dust formation and promoting an easier handling. It is as well a mean to convert a liquid into a solid form.
- 3. Controlled release: depending on the active ingredient to protect and the encapsulation material, it is possible to fine tune the release profile kinetics upon the action of a selected trigger (temperature, pH, water, pressure...). [2]

#### Encapsulation processes using high melting point solid vegetable oils

The general principle of encapsulation is to incorporate an active ingredient within micro particles.

Depending on the technology used, the structure of the particles can be [3]:

- A core / shell structure when the encapsulation material forms a coating around the active ingredient core (a),
- A matrix structure when the active ingredient is dispersed in the encapsulation material (b),
- A combination of both with a matrix core structure coated with a shell (c).



Once the active ingredient is incorporated in the particle, the structure is stabilized.

The encapsulation technique is selected based on desired end size, release mechanism, active ingredient and encapsulation material [2].

Among all the different existing encapsulation techniques, high melting point solid vegetable oils are particularly suitable for physical encapsulation processes like prilling and hot melt coating [4].

# - Prilling -----

Prilling technology, also called spray cooling, spray chilling or spray congealing depending on the melting point of the lipid used, can be applied to produce solid particles within the size range from 50  $\mu$ m to 500  $\mu$ m.

The active agent might be soluble in the molten lipid or present as a suspension of dry particles in the molten lipid phase [5]. The droplets of molten lipid mixed with the active agent are atomized into a chilled chamber resulting in the solidification of the lipids through crystallization and finally recovery of fine solid particles.

Particles are then maintained at low temperature in a system similar to a fluidized bed spray granulation on which molten lipid droplets may adhere to already hard lipid particles before solidification.

This technique is usually used for encapsulation of vitamins, minerals, ferrous sulfate or acidulants.

# - Coating -----

Coating technology using high melting point lipids also called hot melt coating consist is spraying the molten lipid on solid particles under agitation.

The particles are suspended in a temperature and humidity controlled chamber and the coating material is atomized. Molten lipid fine droplets deposit to the surface of the particles until they form a continuous film. The contact with the air of lower temperature leads to solidification of the produced droplets and the formation of the shell.



This technology is well adapted when protection from the environment is needed and can be used for example to isolate iron from ascorbic acid in multivitamins tablets or to encapsulate many functional ingredients like acids (citric acid, lactic acid) or sodium bicarbonate in baked goods [4], [6].

One of the crucial advantages of these techniques is to avoid the use of solvents and all the resulting constrains of their use. Other advantages include the possibility of large scale production, high flexibility in controlling and prolonging the release profile and slower degradation rate of the bioactive [7].

They also allow high active loads compared to other encapsulation techniques [8].

June 2018



## Critical characteristics for high melting point lipids used in encapsulation processes

### — Thermal properties ——

To give good encapsulation performances, high melting point lipids used for encapsulation should have the following thermal properties [9]:

- Physico-chemical stability at temperatures up to 150°C
- Melting point no higher than 85°C since the product is maintained 40 to 60°C above its melting point during the coating process
- A narrow melting range to prevent sticking sometimes seen with low melting point encapsulating products
- Stable fusion / crystallization profile

Differential scanning calorimetry is a widely used tool for the characterization of melting and crystallization behavior of high melting point lipids. DSC thermograms provide very useful information to fine tune process parameters and adjust formulations [10].

#### - Polymorphism -----



One of the major properties of fats is their polymorphism. Fats are constituted of more than 90% of triglycerides which exist in 3 basic crystalline structures that are  $\alpha$  (alpha),  $\beta$ ' (beta prime) and  $\beta$  (beta) in order of increasing stability. Each polymorphic form has a different melting point. The  $\beta$  form is the most stable and has the highest melting point,  $\beta$ ' is intermediate in stability and has a lower melting point and  $\alpha$  is the least stable with the lowest melting point.

The temperature at which the crystallization of the lipid takes place dictates the polymorphs and the structure of the polycrystalline matrix. This impacts particle attributes such as mechanical properties and bioactive release profile [10].

If not controlled, it can cause polymorphic inhomogeneity from batch to batch or polymorphic transitions during storage.

Several approaches have been described to control lipid polymorphism [11]:

- Tempering during process by keeping the temperature between the melting point of the unstable  $\alpha$  and the stable  $\beta$ -form
- Tempering after processing ("maturing")
- Addition of crystallization seeds with stable β-form
- Formulation with additives to promote polymorphic stabilization at lower temperatures



#### Some application examples

#### — Food ——

Lipid-encapsulated functional ingredients are widely used in bakery applications to improve handling and machinability of doughs and also to improve texture, volume, flavor and freshness of the final baked products.

Functional bakery ingredients that can be encapsulated include enzymes, antioxidants, raising agents, flavorings and acids.

For example, ascorbic acid, a weak acid commonly used to add strength to frozen dough, is readily destroyed by mild alkali solutions such as baking soda. Encapsulation protects it until baking time, and when released, it improves baking properties and baked product quality [12].

## — Feed ——

The trend in the use of phytobiotics in animal feed has been increased during last two decades [13].

Indeed, bacterial resistance and antibiotic residues in animal products led to raising the concern in using antibiotics as growth promoters and finally resulted in the study of phytogenic compounds as a possible alternative to antibiotics.

Phytogenic compounds are defined as plant-derived natural bioactive compounds with positive effects on animal growth and health, and are often applied to essential oils, botanicals and herbal extracts [14].

They are very sensitive substances, susceptible to high temperatures and oxidation.

Encapsulation of these bioactive compounds ensures a high level protection and a continuous release in the digestive tract of animals.

Another major use of encapsulation in feed industry is for ruminant nutrition. Fat encapsulation is used to protect nutrients like amino acids (lysine, methionine) from degradation. Thus, they are able to bypass the rumen and to be released in the gastrointestinal tract where they are absorbed more efficiently [15].

#### - Pharma -----

Spray chilling encapsulation technique was used for the first time in the 1960s by Merck and Co Inc. to achieve taste masking for iron particles and to protect water soluble vitamins.

Since that, the importance of lipid-based formulations has increased, due to their outstanding benefits such as providing modified release profiles or taste masking while using solvent-free processing techniques.

Active pharmaceutical ingredients are often administered via the oral route.

Hot melt coating is especially powerful in masking the unfavorable taste of active pharmaceutical ingredients [16].



#### ADM-SIO product range

#### — Product range ——

SIO is a European leader in the production of high melting point flaked fats. Easy to store, handle and dose, they can be either fully hydrogenated or fractionated oils.

<b>Commercial Name</b>	Origin	Melting point (°C)		
GV 60 (MB or SG)*	Palm	60-63		
VGB 4	Soybean (non-GMO)	68-72		
VGB 5 ST	Sunflower	69-74		
VGB 6	Rapeseed (low erucic)	68-74		
VGB 22	Rapeseed (high erucic)	61-66		
Olive WAX	Olive	60-70		
<b>VGB 760 (MB or SG)*</b>	Palm	59-64		

\* MB (Mass Balance) or SG (Segregated), ADM is member of RSPO (Roundtable for Sustainable Palm Oil)

# — Technical information ——

	GV 60	VGB 5 ST	VGB 6	VGB 4	VGB 22	VGB 760
Average fatty acid profile (%)						
C 16:0	43	7	5	12	< 1	83
C 18:0	54	90	91	86	40	5
C 18:1	< 1	< 1	< 1	< 1	< 1	8
C 20:0	< 1	< 1	2	< 1	10	< 0.1
С 22-0	< 1	< 1	< 1	< 1	44	< 0.1

#### **Conclusions**

Encapsulation techniques with high melting point vegetable oils can be used for a wide range of applications when to ensure protection from oxygen and water, taste masking or controlled release.

These techniques are economic, fast, reproducible and safe as they do not need to use any solvent.

The selection of the right lipid for the application requires knowledge of their physico-chemical properties, thermal behavior and their associated effect on the bioactive release.



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