Importance of formulation development for encapsulation through extrusion

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Introduction

Many flavor compounds are highly volatile and sensitive to environmental conditions such as oxygen, heat, moisture, and light. During food processing and storage, these compounds can easily evaporate or degrade, leading to reduced flavor intensity and lower product quality.

Encapsulation helps protect sensitive flavors and improves their stability during processing, transportation, and storage. In addition, encapsulation can enable controlled release of aromas, mask undesirable tastes, and improve flavor retention during thermal processing such as baking or cooking.

Hot melt extrusion (HME) is a continuous, scalable, and solvent-free technology increasingly used to encapsulate flavors, aromas, nutraceuticals, and other sensitive bioactive compounds. In this process, carrier materials are heated and mixed inside an extruder, where rotating screws provide transport and homogenization. Flavor compounds are incorporated into the molten matrix, which is then forced through a die, cooled, and solidified, entrapping the active ingredients. The resulting extrudate can be milled or pelletized into powders or granules for further use.

Finding the right formulation is not trivial. Various food-grade carrier materials can be used in HME for flavor encapsulation, including carbohydrates, proteins, lipids, and biopolymers. Common examples include starches, maltodextrins, whey proteins, and lipid-based carriers. One criterion to choose the matrix is the polarity of the disperse phase. Different matrices interact differently with polar and non-polar substances, influencing the efficiency and effectiveness of encapsulation. Matching the polarity of the disperse phase with the matrix is essential for achieving optimal encapsulation results.

The carrier material or matrix can also influence the processing parameter due to the material's glass transition temperature. The glass transition temperature (Tg) of the matrix determines the temperature range where the material changes from a hard, glassy state to a softer, rubbery state. This has major implications for the stability, processing, and encapsulation performance of food and pharmaceutical formulations, as it will determine the temperature profile used during extrusion.

How formulation development helps processing parameter decisions

Higher-molecular-weight polysaccharides generally have higher Tg values due to reduced chain mobility, while blending with lower-molecular-weight carbohydrates can lower the overall Tg. In compatible mixtures, a single Tg is observed, whereas incompatible or phase-separated systems may exhibit multiple Tg values. In encapsulation and hot-melt extrusion processes, materials are typically processed above their Tg to enable flow and mixing, then cooled below Tg to form a glassy matrix that enhances the retention and stability of volatile compounds. For this reason, polysaccharide mixtures are commonly used for encapsulation.

Another key formulation parameter is the addition of emulsifiers to the blend. Whether an emulsifier is added to the formulation also determines the morphology of the resulting particle. Emulsifiers improve the compatibility between hydrophobic flavor compounds and hydrophilic carrier matrices. They enhance flavor dispersion, reduce phase separation, improve processability, increase encapsulation efficiency, and minimize flavor losses during extrusion.

Common food-grade emulsifiers include lecithin, mono- and diglycerides, sucrose esters, polyglycerol esters, and sodium stearoyl lactylate. Emulsifiers can also act as plasticizers, lowering the glass transition temperature (Tg), which facilitates processing but may affect storage stability if used excessively. Therefore, emulsifier type and concentration are carefully optimized alongside formulation and processing conditions to achieve high flavor retention, good processability, and desired release characteristics.

In this application note, we aim to showcase the encapsulation of canola oil using a Thermo Scientific™ Process™ 16 Twin-Screw Extruder. We show how a mixture of maltodextrin and dextrose behave as carrier materials in comparison to the pure polysaccharides, and how the addition of emulsifiers can help in the encapsulation process.

Rheological characterization with closed cavity rheometer for choosing the right matrix

In order to find the right processing conditions for the encapsulation trials, the melt temperature of the mixture was determined and compared to the melt temperature of maltodextrin and dextrose alone. For this purpose, the viscosity of maltodextrin, dextrose and a mixture of both at 70:30 maltodextrin:dextrose was measured over the temperature using a closed cavity rheometer (RPA Ultra, Bareiss).

The temperature dependence of complex viscosity (η*) of all three carbohydrate systems is found in Figure 1. In all cases, viscosity decreased with increasing temperature, reflecting progressive softening and the transition from a rigid, glassy structure to a more fluid melt. However, the magnitude and onset of this viscosity reduction varied significantly among the samples.

Pure dextrose maintained the highest viscosity across most of the temperature range. Its viscosity remained relatively stable up to approximately 80 °C, followed by a gradual decrease between 80 and 140 °C. A more pronounced drop occurred only near 145–150 °C, suggesting a broad softening region and a gradual transition to a flowable state.

Pure maltodextrin displayed intermediate behavior. Its viscosity remained stable until approximately 95–100 °C before decreasing significantly between 100 and 120 °C. Beyond this range, viscosity continued to decline steadily, remaining lower than that of dextrose but considerably higher than that of the blend.

On the other hand, the maltodextrin–dextrose blend exhibited the earliest and most pronounced decrease in viscosity. Starting from an initial viscosity comparable to the other materials (approximately 10^6–10^7 Pa·s), the blend showed a sharp decline between 80 and 100 °C, losing nearly two orders of magnitude in viscosity.

After a short plateau between 100 and 125 °C, a second substantial decrease occurred above 130 °C, resulting in viscosity values below 10^3 Pa·s at temperatures approaching 150 °C. This behavior indicates reduced thermal stability and enhanced flowability compared with the individual polysaccharides. The addition of dextrose to maltodextrin appears to exert a strong plasticizing effect, lowering viscosity and shifting softening to lower temperatures. These findings have important implications for extrusion processing, where the blend would provide the easiest flow. For this reason, the extrusion trials were conducted with this mixture.

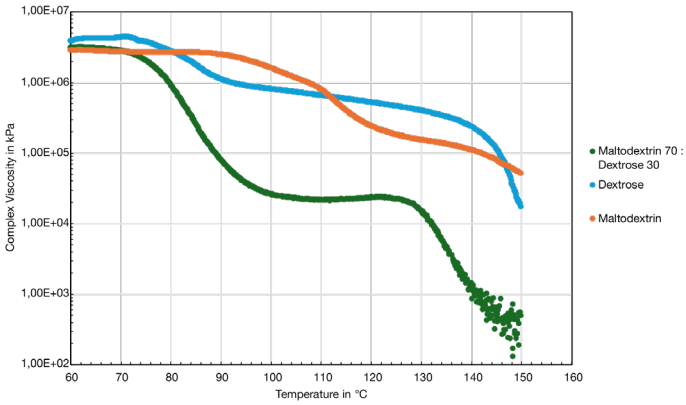

Figure 1. Complex viscosity of three formulations over the temperature.

Extrusion trials and characterization

For this application note, a mixture of maltodextrin and dextrose were chosen for the matrix. After selecting the main polysaccharide for encapsulation, canola oil was selected as the disperse phase as placebo for any oil-based aroma or flavor. In order to assess the encapsulation efficiency of the formulation, one trial was conducted with the addition of soy lecithin (1 wt%) as an emulsifying agent and one without it. Otherwise, both trials were conducted under the same conditions.

Water was added in the second feeding zone, and the canola oil was added in the fourth feeding zone. The extrusion speed ranges from 200 to 600 rpm and temperatures were Tbarrel2 – 5 = 115 °C, Tbarrel6 – 8 = 80 °C, Tdie_adapter = 80 °C. The screw configuration possessed various mixing and kneading zones to ensure proper polymer melting, adequate mixing, and dispersion of the oil into the polymer matrix. Moreover, the screws also retained the melt in the last zones of the extruders to guarantee sufficient cooling, necessary for subsequent downstream processing. The samples were produced with a die plate with 6 holes, 1 mm and pelletized using a face-cut pelletizer.

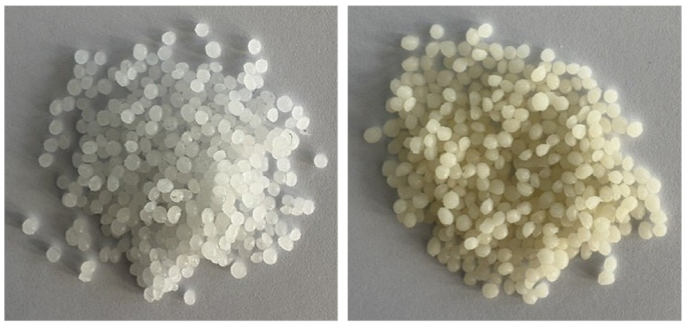

Figure 2. Two samples produced with 10 wt% canola oil and either with 1 wt% lecithin (right) or without it at all (left).

Influence of emulsifying agent on resulting oil droplet distribution

The oil droplet size in disperse system is one of the most important factors affecting encapsulation efficiency (EE) because it influences the total interfacial area, product stability, and the ability of the wall material to completely surround the oil droplets during drying. In general, smaller oil droplets lead to higher encapsulation efficiency, provided that sufficient emulsifier and wall material are available. Smaller droplets are more uniformly dispersed within the continuous phase, allowing the encapsulating matrix (in this case, dextrose-maltodextrin mixture) to form a continuous protective barrier around each droplet. As a result, less oil remains exposed on the particle surface after extrusion, reducing surface oil content and improving encapsulation efficiency.

Conversely, larger droplets are more difficult to completely encapsulate. During processing, they tend to migrate toward the particle surface, increasing the amount of unencapsulated (surface) oil. This lowers encapsulation efficiency and makes the powder or granules more susceptible to oxidation, stickiness, and reduced storage stability.

In order to assess the droplet size distribution of both formulations, 10 g of pellets with and without the addition of lecithin were dissolved in water for 3 min. The resulting emulsions were measured using laser diffraction. Figure 3 shows the volumetric cumulative droplet size distribution (Q3) of the released oil droplets encapsulated in the same formulation (70% maltodextrin and 30% dextrose), but with or without the addition of lecithin as emulsifying agent. The x-axis shows the droplet size (µm) on a logarithmic scale, while the y-axis represents the cumulative volume percentage of droplets smaller than a given size.

The two curves are clearly separated, indicating significant differences in droplet size distribution. The sample with added lecithin (filled squares) is shifted toward smaller particle sizes, demonstrating that this sample contains a larger proportion of fine droplets. In contrast, the sample without the addition of lecithin (open squares) is shifted to the right, indicating a coarser distribution with larger droplets.

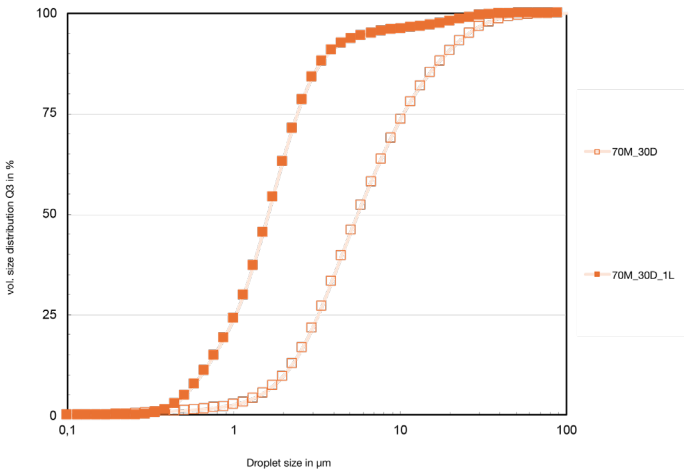

Figure 3. Cumulative droplet size distribution (Q3) of oil droplets with and without lecithin.

The median droplet diameter (d50), corresponding to 50% cumulative volume, is substantially lower for the lecithin sample. The median droplet diameter d50 appears to be approximately 1.5–2 µm, whereas the d50 of the sample without lecithin is closer to 6–8 µm. Similarly, the droplet sizes corresponding to 10% and 90% cumulative volume (d10 and d90) are also lower for the lecithin sample.

Both distributions exhibit relatively steep slopes, suggesting moderately narrow size distributions. However, the sample appears with added lecithin slightly narrower, indicating a more homogeneous droplet population. The sample without lecithin spans a broader size range and contains a higher fraction of larger droplets, extending beyond 30 µm.

Overall, the results indicate that the processing condition with the addition of emulsifying agents was more effective at droplet disruption, producing a finer and more uniform dispersion. Consequently, the sample with lecithin would be expected to exhibit greater stability and a larger interfacial surface area than one without any additives.

To get a closer look into the droplet size distribution and to corroborate the results obtained from the laser diffraction measurement, both formulations were observed using a Thermo Scientific™ Phenom™ XL G3 Desktop SEM. The micrographs are found in Figure 4.

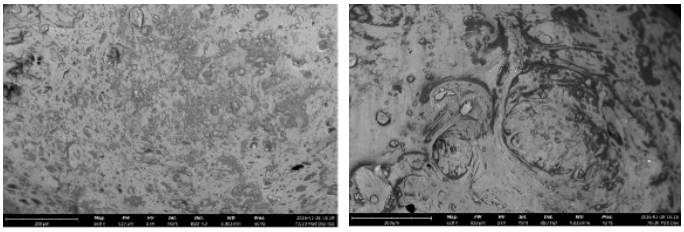

Figure 4. Micrographs of maltodextrin-dextrose capsules, with 10 wt% canola oil and with 1 wt% lecithin (left) or without lecithin (right).

Both micrographs were acquired using similar SEM conditions (BSE detector, 5 kV, ~500× magnification, 200 µm scale bar), allowing a direct comparison of the microstructure.

The most striking difference between the two micrographs is the size and distribution of the dispersed phase. The lecithin sample contains predominantly fine, uniformly distributed structures, whereas the lower micrograph contains much larger inclusions and a broader size distribution. This indicates that the addition of lecithin led to a more efficient dispersion and homogenization, resulting in a more homogeneous microstructure. In contrast, the image of the sample without lecithin suggests that larger droplets or particles persisted during processing or that coalescence occurred, leading to a less uniform matrix. Such large domains can negatively influence mechanical properties, oxidative stability (for encapsulated oils), and product consistency. The addition of emulsifying agents, such as lecithin, results in a better-dispersed and more stable microstructure, whereas refraining from adding emulsifying agents leads to greater heterogeneity and larger dispersed domains.

Conclusions

This application note demonstrates that successful encapsulation by hot melt extrusion depends strongly on both formulation design and process optimization. Rheological characterization using a closed cavity rheometer proved to be an effective tool for selecting suitable carrier materials and processing temperatures. The combination of 70 wt% maltodextrin and 30 wt% dextrose exhibited a pronounced reduction in melt viscosity compared with the individual carbohydrates, enabling extrusion at lower processing temperatures while maintaining adequate flow properties.

The addition of 1 wt% soy lecithin significantly improved the encapsulation of canola oil. Laser diffraction measurements showed a marked reduction in oil droplet size and a narrower droplet size distribution, indicating more efficient emulsification and dispersion within the carbohydrate matrix. Smaller droplets are expected to enhance encapsulation efficiency by reducing the amount of surface oil and improving oxidative stability.

SEM analysis further confirmed these findings. Samples containing lecithin exhibited a finer and more homogeneous microstructure with uniformly distributed oil domains, whereas formulations without emulsifier contained larger inclusions and evidence of droplet coalescence. The improved morphology observed in the lecithin-containing formulation is expected to contribute to enhanced product stability, more consistent release behavior, and improved overall product quality.

This application note highlights the importance of combining formulation development with analytical characterization to optimize encapsulation processes. The Thermo Scientific Process 16 Twin-Screw Extruder, together with rheological measurements, laser diffraction, and Thermo Scientific Phenom XL G3 Desktop SEM imaging, provides a comprehensive workflow for developing robust encapsulation formulations. This integrated approach enables efficient optimization of carrier composition, emulsifier content, and processing conditions, ultimately leading to improved encapsulation performance and facilitating the development of stable food, flavor, and nutraceutical products.