Extrusion

Process 16 Twin-Screw Extruder.

Encapsulation of flavours with co-rotating twin-screw extrusion

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Introduction

Flavors, fragrances, and bioactive food compounds like those employed in the nutraceutical and the pharmaceutical domains, are often supplied in powder or granulated form for easier handling and more accurate dosing in final products. Over recent decades, encapsulation technologies have added new functionalities to these forms. Encapsulation can protect against evaporation, oxidation, moisture, and other aggressive environmental agents to extended product shelf life, or it can aid in the controlled release of key compounds under pre-determined conditions. The most common encapsulation technologies used in the flavor industry consist of spray drying, spray coating, and extrusion.

Although spray drying is widely used in the food industry, the technique's high temperatures and fast drying rates do not allow for encapsulation of temperature-sensitive bioactive compounds. Additionally, the viscosity of the feed can pose a limiting factor for the dry matter content of the initial formulation and might increase the solvent use during processing. This in turn increases the cost and energy consumption of the process.

Twin-screw extrusion offers a modular and flexible method for the encapsulation of flavor, fragrance, and aroma compounds. This process has two primary advantages. During twin-screw extrusion the encapsulating matrix is melted due to thermal and mechanical energy input, therefore no solvents are used. This means solvent supply and recovery are not cost-increasing factors. Moreover, twin-screw extruders allow for the reduction of floor space usage. Other advantages of twin-screw extrusion include lower energy consumption, greater control over the transformation of raw materials to obtain the desired material properties, and the ability to apply different temperature zones during processing to avoid compound degradation.

In this application note we aim to showcase the encapsulation of citrus oil in a sugar matrix using a Thermo Scientific™ Process™ 16 Twin-Screw Extruder, including some tips for process development to optimize production of the desired product.

Extrusion process

Importance of formulation

Formulation is key to the design of the extrusion process. Each ingredient or component serves a role and can be classified roughly as one of three categories: matrix-forming; disperse phase; or additives.

Matrix-forming compounds have the crucial role of serving as the base for encapsulation. Typically composed of (semi) amorphous carbohydrate oligomers, starches, and occasionally proteins (which may interact with the disperse phase), matrix-forming substances play a pivotal role in various applications requiring encapsulation. They determine both retention/load of the disperse phase and working temperature. This term encompasses the rheological properties and physicochemical characteristics essential for the desired outcome.

The disperse phase refers to the compound to be encapsulated, whether that be a flavor, aroma, fragrance, or bioactive compound. It can take the form of a solvent or be oil-based, and it can be introduced either as an emulsion or as a single phase, depending on the desired application and processing requirements.

The polarity of the disperse phase plays a significant role in determining the most suitable matrix for encapsulation. 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. Additionally, the heat sensitivity of the disperse phase is a critical factor that dictates its dosing point within the extruder barrel. Since different materials might degrade or be highly volatile, it is crucial to carefully consider the temperature profile along the length of the extrusion process to ensure that the disperse phase is added at the appropriate stage so it will not undergo undesirable degradation.

Additives play a crucial role in encapsulation processes, offering various benefits to the final product. Some additives such as water create a plasticizing effect, decreasing the material's glass transition temperature (T_g) and enhancing flexibility of the polymer matrix. They may also modify rheological properties, altering viscosity to control flow behaviour during extrusion. Moreover, additives can also help avoid oxidation in materials containing unsaturated fatty acids, preserving their nutritional value.

Additives such as emulsifiers increase retention and influence droplet distribution, improving product quality and shelf life. Whether an emulsifier is added to the formulation also determines the morphology of the resulting particle. Figure 1 depicts some possible particle morphologies obtained via extrusion:

1. Coarse dispersion within the matrix
2. Fine dispersion facilitated by emulsifiers and/or other additives
3. Application of film coating to the core material
4. Fine dispersion with an additional external film coating
5. Fine dispersion and simultaneous coating of both the core and the matrix

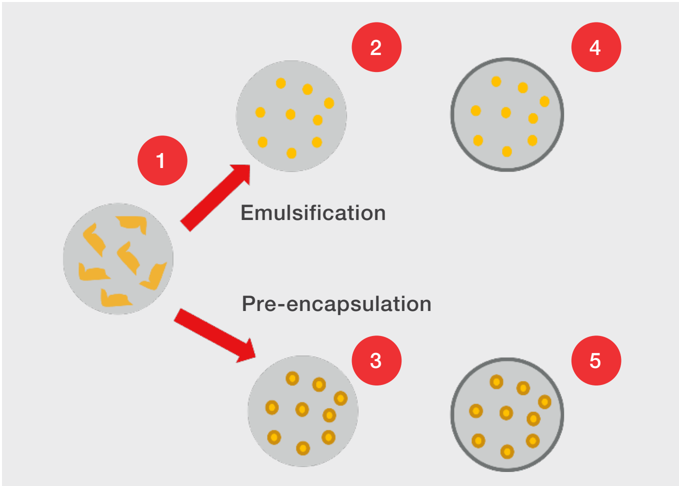

Figure 1: Possible particle morphologies.

The particle morphology will depend on the formulation of the product as well as the processing steps, such as pre-emulsification or the extrusion process, used for product development. Particle morphology will affect the targeted release of the disperse phase and particle loading, among other properties.

Extrusion parameters

For this application note, a mixture of maltodextrin and sucrose were chosen for the matrix, with citrus oil selected as the disperse phase. Water was used as a plasticizer in certain experiments, in concentrations up to 5 wt%. Water was added in the 2nd feeding zone and the citrus oil was added in the 4th feeding zone. As mentioned above, water decreases the T_g and viscosity of the melt, affecting the encapsulation of the disperse phase and morphology of the resulting product. Figure 2 shows an example of the experimental setup.

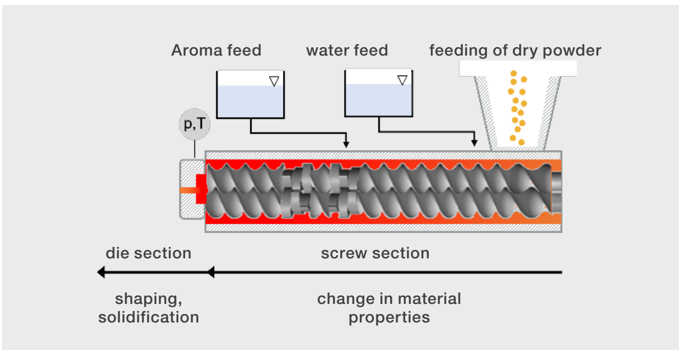

Figure 2: Schematic representation of the extruder setup.

The extrusion speed range from 200 to 600 rpm and temperatures were $T_{barrel2}$= 50 °C, $T_{barrel3}$= 90 °C, $T_{barrel4-6}$= 120 °C, $T_{barrel7}$= 110 °C, $T_{barrel8}$= 90 °C, $T_{die\_adapter-}$= 90 °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 down-stream processing.

Figure 3 shows two samples produced with 5 wt% citrus oil and either with 5 wt% water (right) or without it at all (left), respectively.

Figure 3: 5 wt% citrus oil in a sucrose-maltodextrin matrix with water (right) and without water (left).

Downstream processing

The final product can be cooled down further to ambient temperatures, and then shaped into a specific form and/or diameter during downstream processing. Typical ancillary instruments include pelletizers (either strand or face-cut), chilled rolls to produce flakes, and sheet take-off devices to produce films. Each of these is adjustable to obtain an intermediate or final product with the desired characteristics. In the process described in this note, we used a face-cut pelletizer with two rotating blades and a single orifice of 3 mm diameter.

Conclusions

This application note addressed the process design for hot-melt extrusion using a co-rotating twin-screw extruder for the encapsulation of flavor compounds. **View the full video** on this topic.

References

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