thermo scientific

# Guide to skilled food extrusion

## Application compendium

Thermo Fisher
SCIENTIFIC

# Extrusion in the food industry

Welcome to the skilled guide on food extrusion! Here you'll find a compendium of useful articles and application notes providing expertise on enhancing food properties via extrusion.

Foods can be structurally complex, incorporating emulsions and mixing solids, liquids and gels into a single product. The information in this compendium can help you better understand what to measure in your food development and processing workflows, and which tools are best to measure it.

Food extrusion is an established and highly versatile technique for producing food, feed, nutritional additives and flavors. Extruders with varying dies are used to design and shape foods with high starch content such as pasta, cereals or snacks, analogue meat products, and pet food, and to determine their final texture. In addition, extrusion enables a cost-effective, continuous means of production with precise control for maintaining high product quality.

Feel free to **contact us** if you have any questions or requests about food rheology.

# Table of contents

# Food design by extrusion

In today's food production and processing, extrusion has established itself as a technology that is routinely used to create high-quality, tasty, attractive in price and, above all, safe products for humans and pets alike.

Starting materials for food extrusion processes are usually starch- or protein-based materials. But secondary sources, challenging to handle with other processes, also make a good ingredient for a high-value extruded food product Among such materials are dark flours, wheat bran or broken rice.

As the processing range within an extruder is very wide, and the die and screw geometry is exchangeable, there is almost an infinite number of products (in shape and texture) that can be designed using modern twin-screw equipment. A subsequent treatment of the extrudates such as drying, roasting or coating helps to give polish to the end product.

Typical extrudated products used for human nutrition are*:

| Type of product | Example |
|---|---|
| Directly expanded | Breakfast cereal, corn curls |
| Unexpanded | Pasta |
| Half-products | Potato pellets |
| Co-extruded | Fruit-based cereals, Jelly-filled cores |
| Modified | Starches, fat mimics |
| Texturized | Meat analogs |
| Candy | Licorice, chewing gum |

*Karwe, Mukund V. (2008). "Food extrusion". Food Engineering 3. Oxford Eolss Publishers Co Ltd. ISBN 978-1-84826-946-0.e

The raw materials entering the extrusion process are typically powders, e.g., derived from starch or a protein source. Depending on the desired end product properties, other powder materials, e.g., vitamins or colorants, are added as a dry-mix. Inside the extruder, liquids or semi-solids (such as syrups, fats, oils, water) are added and mixed together.

The shear energy and elevated temperature of the extruder barrel can induce a cooking process that can be precisely controlled at required levels. As pressure also builds up inside the instrument, the required processing time is comparatively small, so sensitive products are less prone to denaturation. This process is usually referred to as hightemperature-short-time (HTST) or extrusion cooking and performs the desired protein denaturation or starch gelatinization.

Being pressed through the extruder die at the end of the process, the material usually puffs out and changes its texture. Then it is cut into desired length and subsequently treated as required for the desired final result. Modern benchtop twin-screw extruders can help to mimic the full potential of extrusion-based food processing at a laboratory scale.

For more information about our extruders, visit **thermofisher.com/foodextrusion** or **thermofisher.com/extruders** for small and pilot scale extruders.

**Watch the video:**
**Thermo Scientific Process 11 Twin Screw Extruder**

# Case study

The role of extrusion in sustainable food research

Read the case study

# An evaluation of meat analog product characteristics

## Combining extrusion with electron microscopy and rheological measurements

**Authors: Valerie Louise Pietsch, Fritz Soergel and Mattia Giannini**

### Key words

Plant-based, meat analog, soy protein, wheat gluten, food extrusion, electron microscopy, rheology

### Introduction

High moisture extrusion offers a great potential to produce a wide range of meat analog products from various plant protein sources. Products that can be achieved from this process are supposed to resemble the product texture of muscle meat. Typically, high moisture extrusion yields intermediate products, which are further processed to produce meat analog, ready-to-eat products using conventional meat processing operations such as mincing, marinating, and mixing.

It has been proposed that the desired muscle meat-like texture can be achieved by imparting an anisotropic, fibrous structure into plant proteins through a shear-induced structuring of a multi-phase system [1]. Such mechanism can take place if a dispersed phase is present together with a continuous phase, as shown in Figure 1. While the exact composition of the dispersed phase depends on the plant protein used as source, it typically consists of free water, water-insoluble proteins, or residual polysaccharides. The continuous phase is usually comprised of water-plasticized plant proteins. During a shear-induced structuring process, such as extrusion processing, these phases are mixed with each other, leading to the formation of a water-in-water type emulsion. Based on the flow characteristics, the dispersed phase is deformed in the direction of the flow. Upon cooling, the matrix solidifies and the deformed structure is retained.

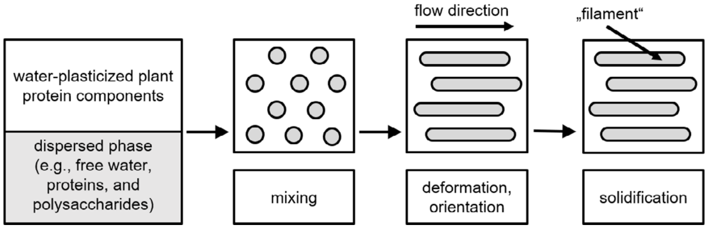



**Figure 1: Scheme of shear-induced structuring of a multi-phase system described by Tolstoguzov [1], [2].**

To achieve the desired anisotropic, fibrous structures, the high moisture extrusion process is generally operated using a co-rotating twin-screw extruder at water contents similar to meat, i.e. between 40 - 80%, and with a cooled slit die attached to the end of the extruder. During the extrusion process, plant proteins are fed to the extruder, mixed with water, and transported along the rotating screws. In the screw section of the extrusion process, the material will be exposed to thermo mechanical stresses due to the shearing of the screws and the heating of the barrel. These stresses can affect the material properties of the plant protein-water matrix in terms of protein denaturation (reaction behavior), miscibility, and rheological properties. The flow characteristics prevailing in the cooling die will determine the deformation and orientation of the dispersed phase in flow direction.

From this general understanding of shear-induced structuring, it can be derived that the final product properties of meat analog products depend on the final morphology of the dispersed phase. The morphology of the dispersed phase further depends on the volumetric fraction and the deformation of the dispersed phase. As current state of research, it is considered that these factors can be influenced by the ingredient composition as well as extrusion process conditions [2-4]. To tailor the product properties of meat analog products to match consumers expectations, it is therefore necessary to consider the influence of ingredient composition as well as extrusion process conditions on the final product characteristics.

Typical ingredients used to produce meat analog products comprise plant protein concentrates or isolates from various plant protein sources. The purity of the protein ingredients as well as their technological properties depend on the processing technology applied. Herein, protein concentrates and isolates typically exhibit a protein content of approx.

70% and 90%, respectively. The most widely used protein sources utilized to produce meat analogs currently are soy protein, wheat gluten, pea protein, or mixtures thereof. Furthermore, interest is rapidly growing in producing meat analogs from plant protein sources that are novel to this application, such as canola seed protein, peanut protein, or algae. Although not vegetarian, even insects are currently considered as promising protein source for proteins for human nutrition that can be imparted into meat analog products.

With the wide range of protein sources available, the applicability of existing as well as novel protein sources to produce meat analog products needs to be evaluated on a regular basis. In terms of material development as well as quality control, the Thermo Scientific™ Process 11 Hygienic Extruder offers a solution to produce meat analog products on a lab scale and test new ingredient compositions with reduced testing time, sample size, and waste [5]. In this report, it is demonstrated how the Process 11 Hygienic Extruder can be applied in combination with rheological tools and electron microscopy to evaluate the influence of ingredient composition on the final product characteristics of meat analog products. Microstructural characterization of the fibrous, anisotropic structure was carried out using a Thermo Scientific™ Quattro S Environmental Scanning Electron Microscope (ESEM), an ultra-versatile high resolution SEM with unique environmental capabilities, which provide the flexibility to accommodate any type of sample. Quantitative measurements of rheological properties were conducted using the Thermo Scientific™ Haake™ MARS™ 60 Rheometer, a research grade rheometer for extended material characterization.

## Material and methods

### Extrusion experiments

Meat analog products were produced with the Process 11 Hygienic Twin-screw Extruder and a cooled slit die. Plant-protein powders were fed into the extruder with a volumetric Twin-screw feeder. A peristaltic pump was used for water feeding. The schematic process setup is shown in Figure 2. The dimensions of cooled slit die were H x W x L: 5 x 20 x 200 mm.

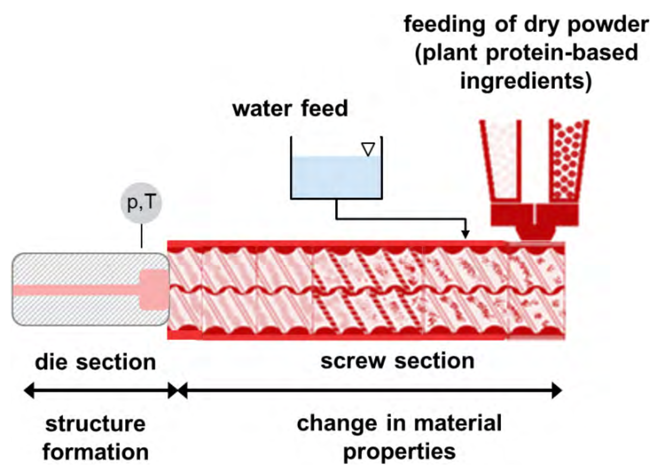



**Figure 2: Schematic setup of the Process 11 Hygienic Twin-screw Extruder with length to diameter ratio of 40 and cooled slit die.**

Three different formulations were analyzed to investigate the influence of ingredient composition on the product characteristics of meat analogs. As indicated in Table 1, wheat gluten and soy protein were used as well as a mixture thereof. The trial was performed at otherwise constant process parameter settings, where screw speed was kept at 400 rpm, total feed rate at 1.1 kg/h, and a water content at 60%. Material temperature of the melt was measured at extruder exit (before entering the cooling die) and accounted for 135 °C for all three formulations. The temperature of the cooled slit die was kept constant at 80 °C.

**Table 1: Ingredient composition used for extrusion trials.**

| Soy Protein Concentrate (SPC) | vital Wheat Gluten (WG) |
|---|---|
| 100% | 0% |
| 70% | 30% |
| 0% | 100% |

After extrusion trials, samples were taken and stored at - 8 °C in airtight plastic bags to prevent drying and spoilage of the samples. For analysis, samples were defrosted overnight and taken out of the plastic bag directly before the measurement.

### Visual appearance and SEM analysis

The typical characteristics of a meat analog production terms of fibrousness, anisotropy, and texture were observed by the visual appearance of a sample. The analysis of the microstructure of the extruded samples was carried out using a Thermo Scientific™ Quattro S ESEM. The samples were prepared for SEM analysis by means of freeze-fracture, a method allowing to expose the cross section of the material without creating any artifacts due to the use of cutting tools. A small piece of the frozen extruded sample was dropped into liquid nitrogen to further cool it and increase its brittleness. Once fully cooled the sample was broken along the flow axis in order to expose a fracture surface along the middle axis of the sample. The fractured sample was then thawed at room temperature and then fixed onto an aluminum SEM stub using carbon tape. Lastly, the sample was sputter coated with Iridium to make the surface electron conducive. The SEM analysis was carried out in High Vacuum mode using Everhart-Thornley Detector (ETD) to collect secondary electron images, which provide topographic information. An acceleration voltage of 5 keV was selected to reveal enhanced surface details.

### Rheological characterisation

Compression and oscillatory measurements were conducted to characterize the rheological properties of the extruded meat analog products. All measurements were carried out at room temperature. As shown in Figure 3, compression tests were conducted with the HAAKE MARS 60 Rheometer equipped with a plate-plate geometry with 8 mm diameter at a compression speed of 0.1 mm/s. For

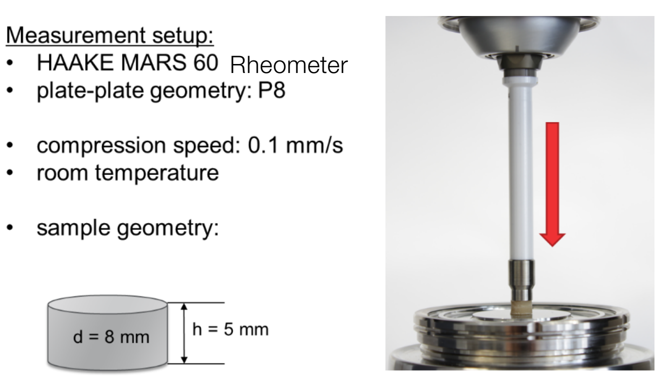



**Figure 3: Measurement setup for compression tests using a HAAKE MARS 60 Rheometer and plate-plate geometry P8.**

the tests, samples were cut with a circular punching iron into a round sample shape with a diameter of 8 mm. The height of the sample resulted from the original height of the cooling die, which was 5 mm.

Using a plate-plate-geometry with a diameter of 35 mm, oscillatory measurements (amplitude and frequency sweeps) were conducted. For this purpose, two stripes of extruded sample were taken and placed under the plate-plate geometry (see Figure 4). The length of the samples was cut to approx. 35 mm. This way it was ensured that the measurement gap is completely filled. Height and width of the sample resulted from the original height and width of the cooling die, which was 5 and 18 mm, respectively. The samples were loaded into the measuring geometry by applying a constant normal force of 20 N. After reaching a constant normal force, oscillatory measurements were conducted at the resulting gap height. The respective strain and frequency settings used for amplitude and frequency sweeps are listed in Table 2.

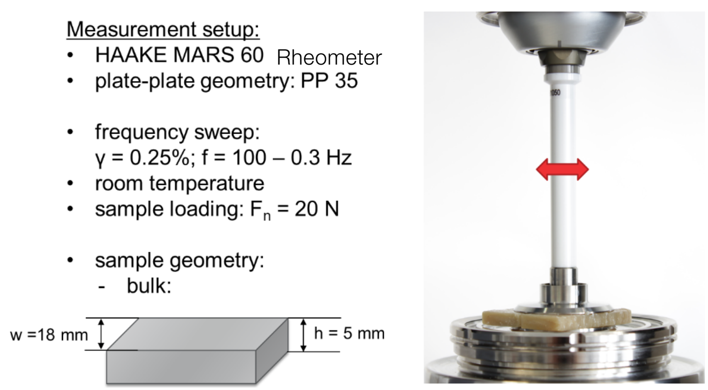



**Figure 4: Measurement setup for amplitude and frequency (bulk) using a HAAKE MARS 60 Rheometer and plate-plate geometry P35.**

**Table 2: Measurement settings for amplitude and frequency measurements (bulk).**

| | Amplitude sweep | Frequency sweep |
|---|---|---|
| Strain γ | $10^{-2}$ - $10^{3}$% | 1 Hz |
| Frequency f | 0.25% | 100 - 0.3 Hz |

## Results

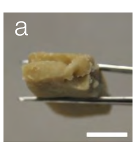
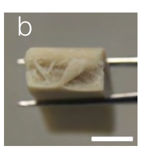
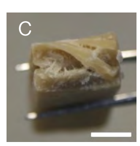
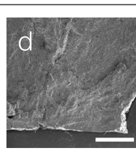
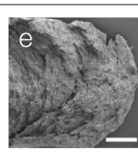
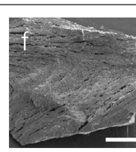
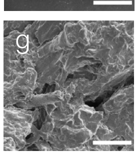
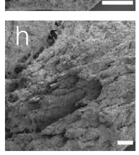
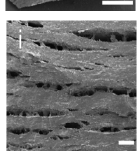
### Visual characterization of macro- and microstructure

The results for the visual characterization of the macro- and micro-structure of the extruded meat analog products are given in Table 3. The view of the macro-structure of all samples shows that all extruded samples exhibit an anisotropic, fibrous structure. With a first visual and haptic analysis it could be observed that the samples exhibit overall different product properties. The samples made from 100% Wheat Gluten (WG), for example, was very elastic and chewy. At the same time, the sample made from 100% Soy Protein Concentrate (SPC) seemed to be very brittle and hard. In contrast to SPC as well as WG, it can be observed that the mixture of SPC and WG with a ratio of 70% to 30% results in an extruded product with a soft texture.

Having a closer look at the micro-structure, the visual appearance of all extruded samples derives from the presence of a multi-phase system. For all samples, a dispersed phase could be observed, which is oriented in flow direction. As explained earlier, this type of structure is considered as a prerequisite for the formation of a meat analog product with a muscle meat-like texture. This is in agreement with previous studies [6], where it has been shown that the SPC to WG ratio significantly affects the structure formation and product characteristics. Moreover, it was suggested that meat analog samples containing 20 to 30% WG are close to the texture of boiled chicken meat [6].

Overall, these results demonstrate that not only the extrusion process parameters, but also ingredient composition affect the product characteristics of meat analog products. In the following, rheological analysis was used as standardized operation procedure for the quality assessment of meat analog products.

**Table 3: Macro- and micro-structure of meat analog samples produced with Process 11 Extruder using varying ratios of Soy Protein Concen-trate (SPC) and Wheat Gluten (WG). The scalebar in the images are 100 mm (a, b, c), 1 mm (d, e, f), 100 µm (g, i) and 200 µm (h), respectively.**

| Ratio of SPC / WG | 100 / 0 | 70 / 30 | 0 / 100 |
|---|---|---|---|
| Macro-structure |  |  |  |
| Micro-structure (SEM) |  |  |  |
| |  |  |  |
| Visual and haptic description | Anisotropic Chewy, elastic | Anisotropic Soft | Anisotropic Brittle, hard |

## Rheological analysis as quantitative measuring tool

The aim was to use rheology as comparative tool to identify the extruded product with a targeted product quality, e.g. similarity to chicken meat [6]. First, compression analysis was conducted by subjecting uniaxial strain to the extruded samples. The result in Figure 5 depict the typical force-distance curve that were recorded for the different meat analog samples. The force distance curves first show a linear increase, which corresponds to the elastic deformation of the material. Then, the applied force approaches a maximum yield point, at which the material cannot sustain its original structure anymore. In the non-linear region of the force-distance curve, the material begins to flow and can rupture. From the Linear Viscoelastic Region (LVR) of these measurements, it is possible to compare the stiffness and elasticity of the extruded samples. The Young's modulus E can be calculated from this part of the force-distance curve. An increase in Young's modulus describes the increase in stiffness of a sample, whereas a decrease in Young's modulus indicates the increase in elasticity of a sample [4].

The comparison between the different samples shows that the Young's modulus increases with increasing Soy Protein Concentrate (SPC) content. These results are in good accordance with the haptic and visual description of the sample. Accordingly, the results show that this method can be used quantitatively to compare the texture of meat analog samples with respect to their elasticity and stiffness.

Second, amplitude sweeps were conducted. The aim was to quantify the structural strength and chewiness of the meat analog samples. The results are depicted in Figure 6. All samples show a distinct range, where storage and loss modulus G' and G" are constant and independent of the applied deformation. This region is typically referred to as the Linear Viscoelastic Region (LVR). The width of the LVR depends on the structural strength of the material. A change in storage and loss modulus indicates that the applied deformation exceeds the LVR, causing a change in the samples structure. In Figure 6, the results show that, after a certain deformation, the non-linear deformation starts with a non-linear increase in loss modulus G" and following the cross-over of G' and G". Typically, the cross-over point is defined by the balance of G' = G". The loss factor tan d can be used as characteristic value to determine the cross-over point. It is given by the ratio of G" and G'. At the cross-over point, tan d equals 1 or $\delta = 45°$.

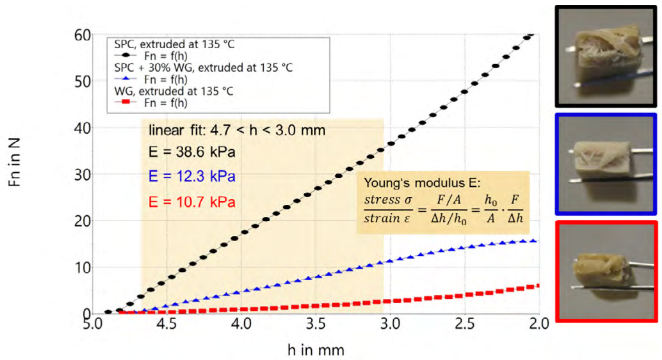



**Figure 5: Change in the elasticity and stiffness of meat analog samples made from varying ratios of Soy Protein Concentrate (SPC) and Wheat Gluten (WG) characterized by compression analysis using a HAAKE MARS 60 Rheometer and plate-plate geometry P8.**

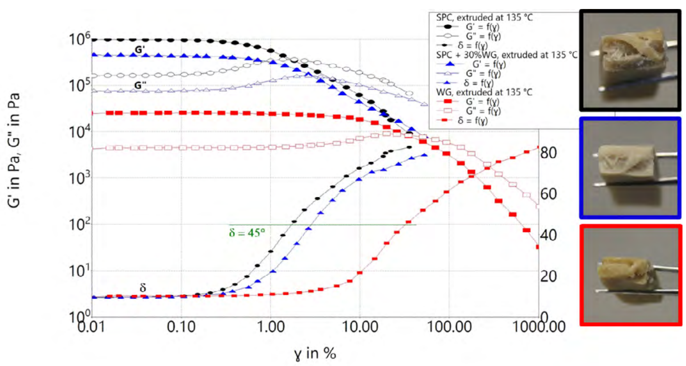



**Figure 6: Change in the structural strength of meat analog samples made from varying ratios of Soy Protein Concentrate (SPC) and Wheat Gluten (WG) characterized by amplitude sweeps (bulk) using a HAAKE MARS 60 Rheometer and plate-plate geometry P35.**

The observed increase in G" before approaching the cross-over point can be caused by the inner friction between structural elements, e.g. between emulsion droplets in liquid systems. In solid systems this increase can also results by the occurrence of micro cracks. To gain further insight on what is happening during the deformation on a micro-structural level, additional amplitude sweep measurements were conducted using the RheoScope module. As explained in [7], this module comprises an optical (polarization) microscope, which can be applied to record microscopic images during shearing of samples. The applied measurement procedure and resulting RheoScope images are depicted in Figure 7. To attain an optical image, it was necessary to cut the samples in thin layer (see Figure 7 a). Sample images were recorded during the deformation of Soy Protein Concentrate (SPC) and Wheat Gluten (WG). Similar to the SEM images depicted in Table 3, the micro-graphs of both samples show a dispersed phase, which is embedded into a continuous protein matrix. The results in Figure 7 b and c depict the microscopic images recorded during an amplitude sweep of SPC at a deformation of 30% and 230%. As highlighted in both images, micro cracks propagate with increasing deformation. Similar findings are observed for WG, where a larger crack appears with increasing deformation from 40% to 120%. Thus, it is very likely that the observed increase in G" (see Figure 7) following the cross-over of G" over G' is related to the break-down of the distinctive microstructure with increasing deformation.

Regarding the characterization of product properties, the results depicted in Figure 7 provide information on the structural strength and chewiness of the extruded meat analog products. While SPC shows a cross-over point at a deformation of 1.7%, the cross-over point of the SPC-WG mixture and WG are shifted to 2.9% and 33%, respectively. The observed shift in cross-over point indicates that the structural strength of meat analog products produced from WG is higher compared to SPC. The fact that the structural break-down of the WG sample requires higher deformation implies that consumers will perceive the texture of meat analogs made from this protein source as chewier.

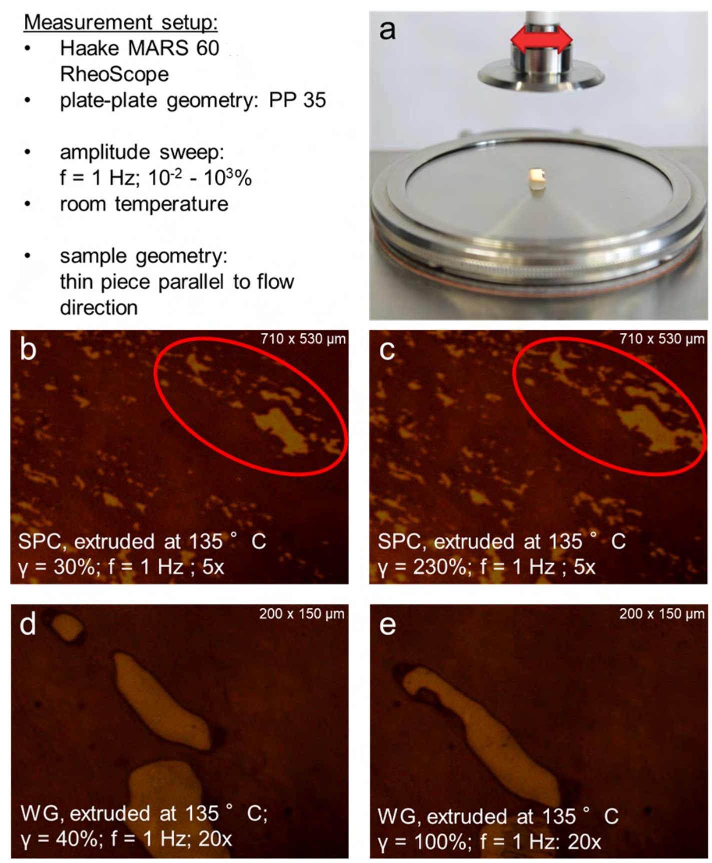



**Figure 7: Change in microstructure elements with increasing deformation, characterized by amplitude sweeps (thin layer) using a HAAKE MARS 60 Rheometer with RheoScope module and plate-plate geometry P35.**

thermo scientific

Thus, the results from the rheological measurements are not only in very good alignment with the visual and haptic impressions of these samples (see Table 3), but they also show that the results (e.g., the applied deformation and rheological properties at cross-over point) from amplitude sweep analysis can be used as quantitative value to compare the product properties (chewiness) of meat analog products.

Finally, frequency sweep measurements within the Linear Viscoelastic Region (LVR) can be used to describe the hardness or softness of the samples. The results for the different meat analog samples are displayed in Figure 8. The material shows shear thinning behavior and is predominantly elastic as G' > G". Moreover, the meat analog sample made from SPC shows the highest viscosity. The complex viscosity further increases from the meat analog SPC-WG mixture to WG. Since these measurements were conducted on samples in bulk, they indicate that the samples with highest complex viscosity are more resistant to shear stresses. Thus, the samples with a higher viscosity are likely to be perceived by the consumer as harder and less easy to bite. These findings are also in very good accordance with the qualitative observations summarized in Table 3. Therefore, these results (e.g., complex viscosity) give a quantitative value to compare the product properties (hardness) of meat analog products.

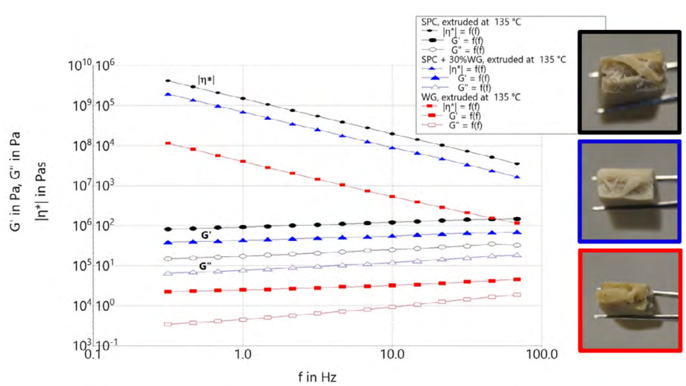



**Figure 8: Change in the hardness/softness of meat analog samples made from varying ratios of Soy Protein Concentrate (SPC) and Wheat Gluten (WG) characterized by frequency sweeps (bulk) using a HAAKE MARS 60 Rheometer and plate-plate geometry P35.**

## Conclusion

The results of this study show how the product characteristics of meat analog products can be analyzed by the combination of microscopy and rheological tools. As summarized in Figure 9, the results from SEM analysis using the Quattro S ESEM show that meat analog products with typical characteristics can be produced using a Process 11 Hygienic Twin-screw Extruder with cooled slit die. Results from rheological measurements using a HAAKE MARS 60 Rheometer allowed to determine quantitative values that describe the elasticity, chewiness, and hardness of the meat analog products. Overall, the product characteristics of the resulting meat analog product depend on the ingredient composition and extrusion process conditions. With the presented analysis tools, the target product quality of the resulting meat analog products can be selected according to the consumers' requirements.

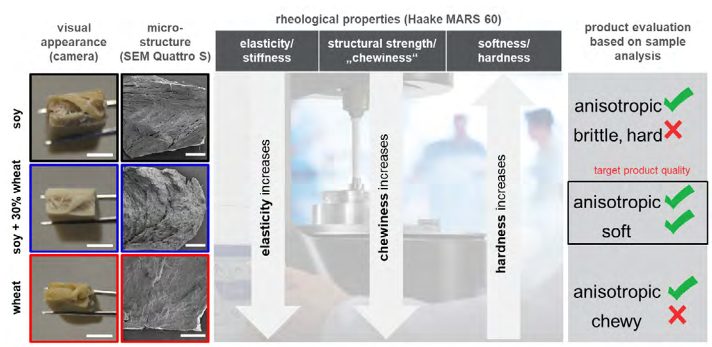



**Figure 9: Evaluation of meat analog product quality based on sample analysis via optical and rheological tools.**

## References

1. Tolstoguzov, V.B., 1993. Thermoplastic extrusion the mechanism of the formation of extrudate structure and properties. Journal of the American Oil Chemists' Society 70 (4), 417–424.
2. Pietsch, V.L., 2019. High moisture extrusion: Investigations on the reaction behavior of plant proteins. PhD Thesis, Karlsruhe Institute of Technology.
3. Osen, R.J.J., 2017. Texturization of pea protein isolates using high moisture extrusion cooking. PhD Thesis, Technical University of Munich.
4. Dekkers, B., 2018. Creation of fibrous plant protein foods. PhD Thesis, Wageningen University.
5. Guy, R., 2000. Extrusion cooking. Technologies and applications. Woodhead Publishing.
6. Chiang, J.H., Loveday, S.M., Hardacre, A.K., Parker, M.E., 2019. Effects of soy protein to wheat gluten ratio on the physicochemical properties of extruded meat analogs. Food Structure, 19, 100102.
7. Oldörp, K., Application Note V-228, What happens when rheological properties change? Looking into Rheological Properties with Simultaneous Collection of Microscopic Images.

thermo scientific

APPLICATION NOTE

No. LR-78

# The influence of extrusion conditions on the processability of starch compounds

**Authors: Matthias Jährling and Bern Jakob**


### Keywords
Food extrusion, twin-screw compounding, process optimisation


### Introduction
Starch is a base material that is widely used in the food industry for snack foods, cereals and pet food products. The processing of starch with twin-screw extruders offers great flexibility in process design and in the final products derived from it.

Carefully choosing extruder parameters like screw set-up and processing temperature, as well as the liquid-to- solid ration of the raw materials enables the operator to greatly influence the resulting product properties.
This application note showcases the different process parameters that play an important role in influenand their influence on the final product quality.

### Experiment
The Thermo Scientific™ Process 11 "Hygienic" Parallel Twin-Screw Extruder was used for extrusion experiment.


**Figure 1: The Thermo Scientific Process 11 "Hygienic" Parallel Twin-Screw Extruder**

The extruder itself is built of hygienic grade steel and is therefore perfectly suited for the processing of food-based materials.

A 30 mm sheet die with a gap height of 1.0 mm was attached to the extruder.
The extruded starch sheet was taken off by a small conveyor belt.

To avoid any loss of sample humidity, 20 mm discs were immediately cut from the extruded sheet, and the rheological properties were determined without further delay.

### Sample material
- Extrusion System:
  Process 11 "Hygienic" Twin-Screw Extruder (see Figure 1)

ThermoFisher SCIENTIFIC

- Cooling Circulator:
  Thermo Scientific™ Polar Series Accel 500 LC Recirculating Chiller
- Feeder for Premix:
  Gravimetric MiniTwin MT0 for Process 11 Extruders
- Feeder for Liquid:
  Thermo Scientific™ Masterflex P/S Pump Systems
- Extrusion Die: 30 mm Sheet Die
- Take-Off System:
  Mini Conveyor Belt for Process 11 Extruders

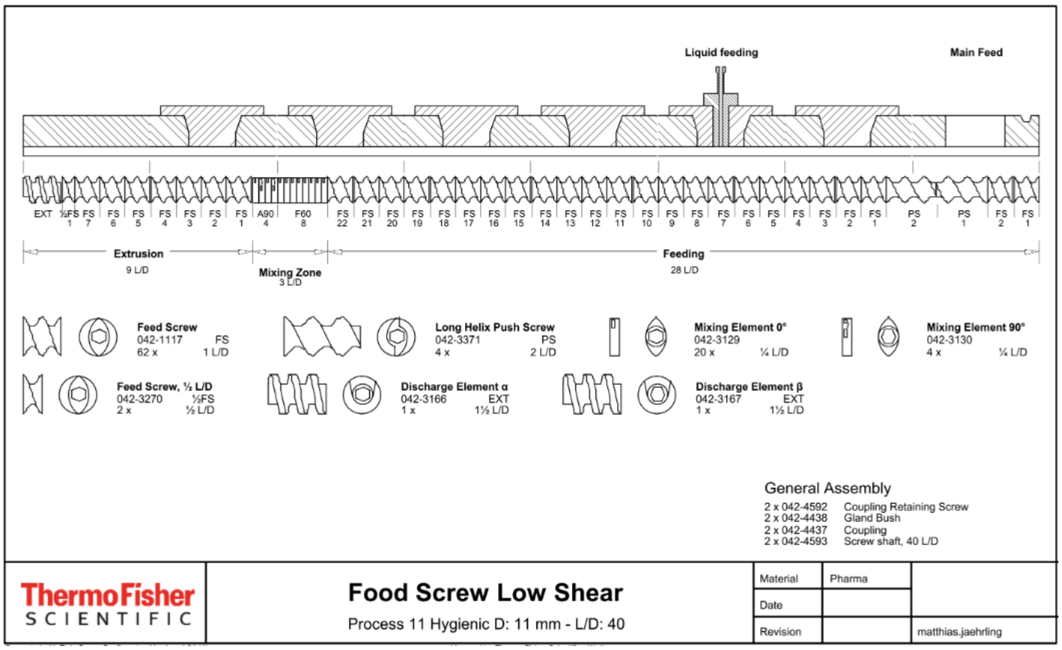
## Rheology equipment

- Rheometer:
  Thermo Scientific™ HAAKE™ MARS™ 60 Rheometer
- Temperature control: Peltier temperature module
- Measuring geometry: 20 mm Parallel plates

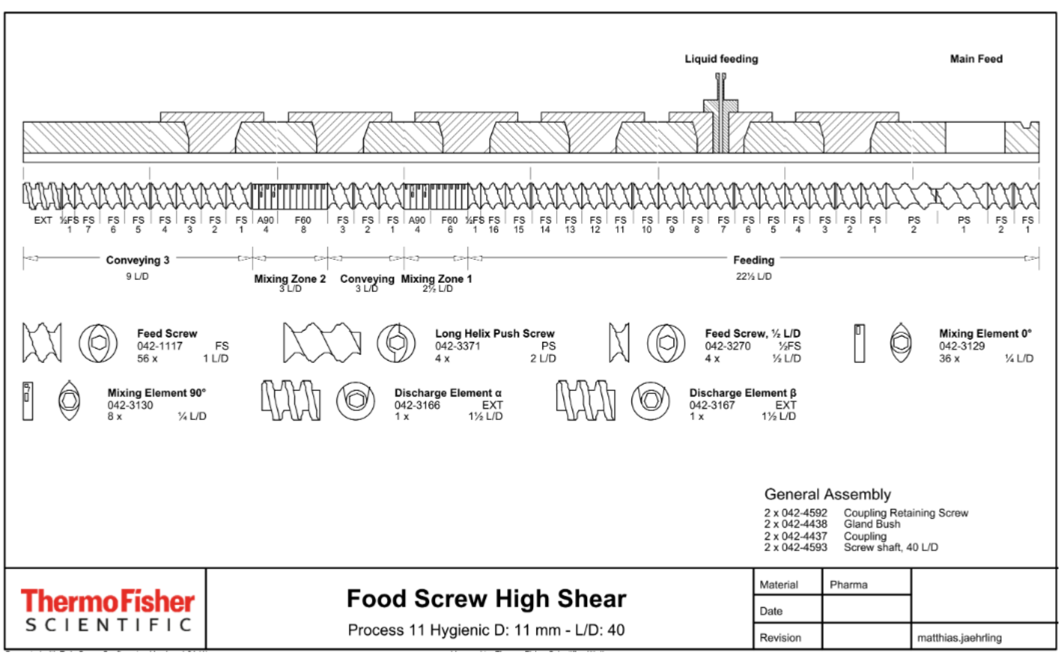
## Extrusion conditions

Variable 1: Screw-Configurations

a) Low shear screw (“LS“): One stage mixing




b) L High shear screw (“HS“): Two stage mixing



### Variable 2: Temperature profiles

Table 1. Low Temperature Profile ("80 °C")

| Barrel Temperature Profile "80 °C" | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Zone | Die | 9 | 8 | 7 | 6 | 5 | 4 | 3 | 2 |
| | 80°C | 80°C | 80°C | 80°C | 80°C | 70°C | 70°C | 50°C | 50°C |

Table 1. Low Temperature Profile ("120 °C")

| Barrel Temperature Profile "120 °C" | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Zone | Die | 9 | 8 | 7 | 6 | 5 | 4 | 3 | 2 |
| | 80°C | 90°C | 120°C | 120°C | 120°C | 70°C | 70°C | 50°C | 50°C |

### Variable 3: Speed extruder screws

a) 200 rpm
b) 400 rpm

### Variable 4: Feeding rate

a) 540 g/h
b) 960 g/h

**Process 11 Hygienic**

| Sample No. | Screw config. | Speed [rpm] | mp [g/h] | Extrusion Temp. | TM [°C] | P [bar] | TQ [%] | RT [sec] | \|η*\| 10 Hz [Pa*s] |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Low | 200 | 540 | 80°C | 87 | 31 | 24 | 95 | 15040 |
| 2 | Low | 400 | 540 | 80°C | 94 | 25 | 25 | 78 | 12170 |
| 3 | Low | 200 | 960 | 80°C | 93 | 41 | 33 | 58 | 15900 |
| 4 | Low | 400 | 960 | 80°C | 99 | 33 | 28 | 47 | 12830 |
| 5 | Low | 200 | 540 | 120°C | 113 | 35 | 26 | 99 | 20990 |
| 6 | Low | 400 | 540 | 120°C | 117 | 32 | 28 | 91 | 20680 |
| 7 | Low | 200 | 960 | 120°C | 107 | 50 | 28 | 63 | 24020 |
| 8 | Low | 400 | 960 | 120°C | 115 | 48 | 27 | 51 | 29090 |
| 9 | High | 200 | 540 | 80°C | 86 | 33 | 37 | 110 | 12960 |
| 10 | High | 400 | 540 | 80°C | 98 | 24 | 32 | 92 | 9194 |
| 11 | High | 200 | 960 | 80°C | 93 | 39 | 44 | 70 | 13970 |
| 12 | High | 400 | 960 | 80°C | 99 | 31 | 39 | 56 | 12250 |
| 13 | High | 200 | 540 | 120°C | 113 | 33 | 28 | 110 | 17300 |
| 14 | High | 400 | 540 | 120°C | 119 | 38 | 27 | 75 | 13160 |
| 15 | High | 200 | 960 | 120°C | 103 | 44 | 36 | 68 | 16830 |
| 16 | High | 400 | 960 | 120°C | 115 | 37 | 32 | 57 | 15840 |

Figure 2: Test-Matrix and results of the extrusion tests.

## Discussion of the extrusion results

a) Residence time

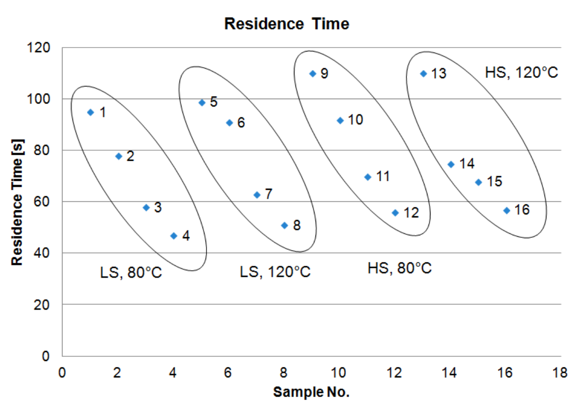



Figure 3.

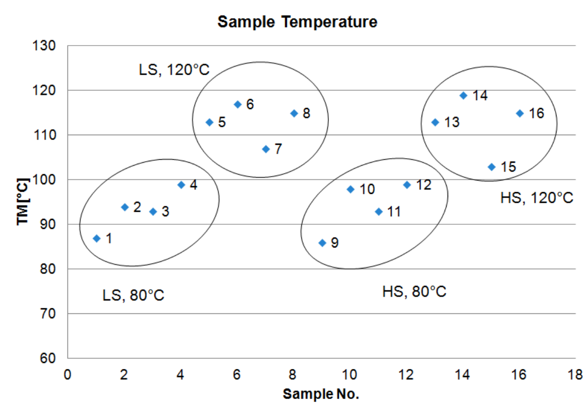



Figure 4.

The residence time was measured by means of a color tracer that was added into the main feed port. The time was stopped until a color change could be seen on the extruded sheet.

Figure 3 shows the results of the residence time measurements.
It can be clearly seen that the residence time gets shorter-with increasing screw speed and with increasing feed rate,

whereas the effect of the higher feed rate proved to have a much larger effect on the residence time.

It also can be seen, that the extrusion temperature had nearly no effect on the residence time. Finally, the effects of the different screw configurations are not very significant.

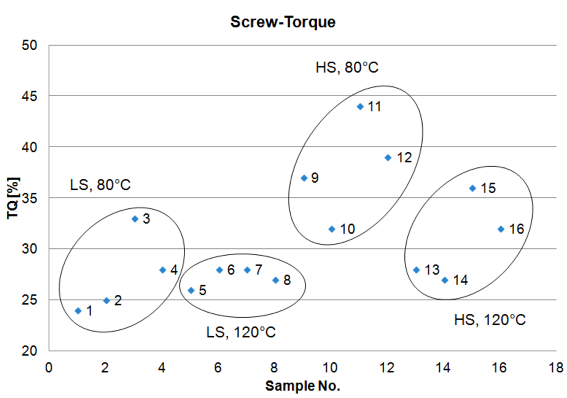
b) Sample temperature

The sample temperature was measured with a melt thermocouple, which was placed at the die adapter at the end of the extruder.

As expected, Figure 4 shows that the sample temperature increased, with a higher extruder temperature. In addition, the higher screw speed resulted in a higher sample temperature. The different screw configurations seemed to have no significant effect on the sample temperature.

c) Extruder torque

Figure 5.

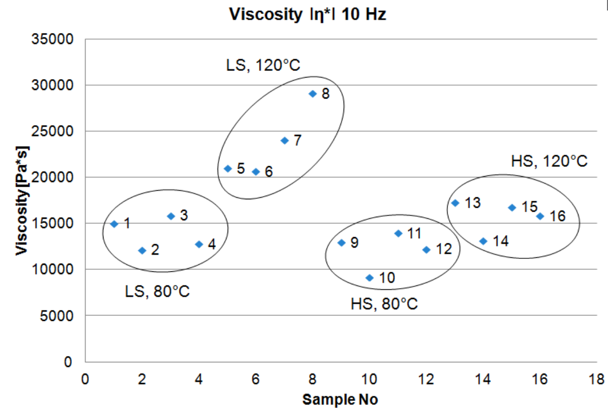



The measurement of the extrusion torque showed a clear increase in torque when using the high shear screw confi- guration with the two mixing zones. It also can be seen that the torque is higher at lower temperatures. Higher feed rates with the same screw speed generate a higher torque where- as an increase of the screw speed decreases the torque.

d) Sample viscisity

The measurement of the sample viscoelastic behavior gave some interesting and unexpected results. The highest sample viscosity could be found with samples extruded at 120 °C with the low shear screw configuration. The viscosity measurements at 80 °C with the low shear screw configuration showed no significant difference to the samples prepared with the high shear screw at 80 °C and 120 °C. A possible ex- planation of this result may be that the sample wasn't yet fully gelated at 80 °C, but it was over-sheared with the high shear screw configuration so the structure already suffered a structural damage.

Figure 6.




The test results indicate that extrusion with the low shear screw configuration, at 120°C, at the high feed rate, and at the high screw speed delivered the best gelation of the product.

## Rheological results

Rheological tests were performed on the samples with a HAAKE MARS 60 Rheometer with a Peltier temperature module. All tests were conducted at 20 °C with a parallel plates measuring geometry: P20/Ti. All samples collected from the extruder were measured immediately. Test spe- cimens were cut out of the extruded sheet with a 20 mm punch hole. First an amplitude sweep from γ 0.5 to 50% at 1 Hz was performed to determine the linear viscoelastic range.

It is obviously in Figure 7 that linear viscoelastic range extends to a deformation of about γ = 10%. For all the successive frequency sweeps, a deformation of γ =2% was the set value for the tests with a frequency range of 0.02 to 46 Hz. To were repeatability, two fresh test specimens extruded at 80 °C were measured.

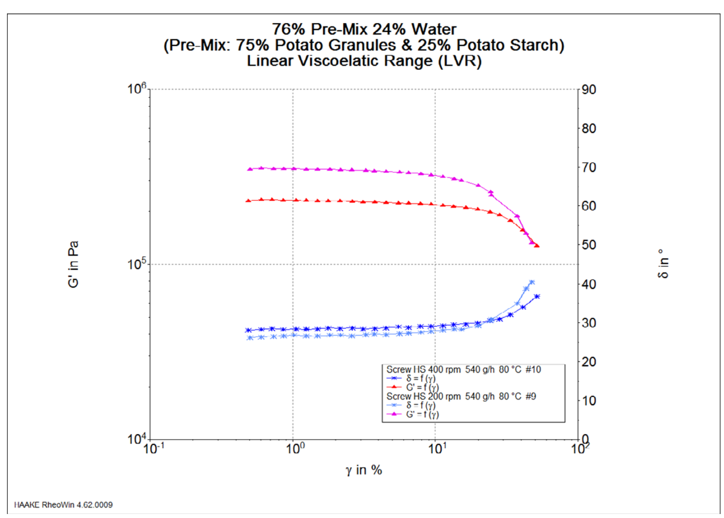



**Figure 7: Determination of the linear viscoelastic range. High shear screws, feed rate 540 g/H at 80 °C.**

In Figure 8 the results of the two tests are plotted and show a reasonable repeatability. All test specimens show the same viscoelastic behavior; the elastic component G' is always higher than the viscous part G". In the frequency dependence of the complex viscosity |η*| a shear thinning of the sample is visible. This is also the typical trend of thequantities for all the other tests.

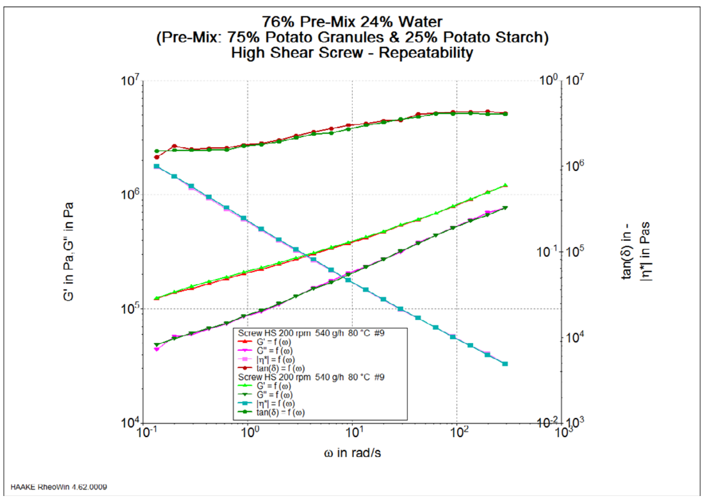



**Figure 8: Frequency sweep repeatability. High shear screws, feed rate 540 g/H at 80 °C.**

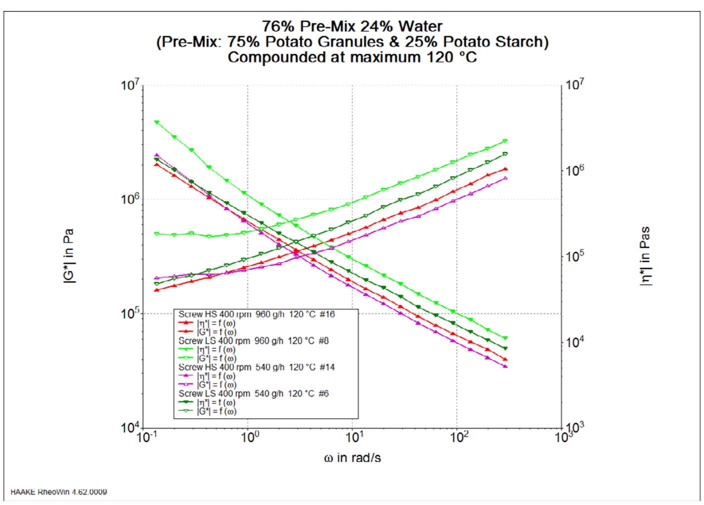



**Figure 9: Frequency sweep - high shear screws, different feed rates at 120 °C.**

The four tests of the samples with low shear screws and the different feed rates at 120 °C are plotted in Figure 8. The complex viscosity |η*| and modulus |G*| of the samples with a feed rate of 540 g/h is almost independent of the screw speed. With an increase of the trough put to 960g/h, the values increase and the expected effect the of higher screw speed is visible. High feed rate and high screw speed of the low shear screws result in the highest viscosity and modulus which is an indication of the best gelation (see also Figure 6).

## Conclusion

The gelation of starch is a complex process that is dependent on several variables. For water concentrations between 20% and 60%, the degree of gelatinization shows a strong dependency on the processing temperature [1]. The higher the temperature, the more complete the gelatinization is.

This effect can be observed in Figure 6 where, using a liquid content of 26%, the viscosity is highest at 120 °C. Under the same conditions, the higher shear energy introduced into the system by the screw setup with two mixing zones degrades the three-dimensional network during the gelatinization process.

In summary, using twin-screw extrusion for production of a starch matrix offers a range of processing variables that enables the user to more adeptly design a starch matrix to required product properties. Twin-screw extrusion offers the user the ability to influence texture, stability and further processability of the final product. Combining extrusion with oscillatory rheology allows for defined, precise analysis of the end product and thus provides a workflow that enhances capabilities in today's food design.

### Reference

1. Fechner, Petra M. „Charakterisierung pharmazeutischer Hilfsstoffe" 2005; Dissertation at Martin Luther Universität Halle-Wittenberg

Find out more at **thermofisher.com/extruders**

ThermoFisher SCIENTIFIC

# On-demand webinar

How extrusion conditions influence the properties of starch compounds

Watch the webinar

APPLICATION NOTE

No. LR-76

# Encapsulation of flavors and ingredients using a twin-screw extruder

Authors: Matthias Jährling and Dirk Hauch

**Keywords**
Flavor encapsulation, continous processing, twin-screw compounding

## Introduction

Flavors are sensitive and expensive additives used in different industries such as pharmaceutical, chemical, cosmetic and food. Over the last decades these flavors and active ingredients have been encapsulated in a polymeric matrix for various purposes such as protection against oxidation, loss of flavor, taste masking, controlled release, or better product handling.

Possible matrix polymers include starch, different sugars, cellulose derivatives, lipids, proteins and special rubbers. The largest shares have of course starch and sugars. The traditional method of encapsulating flavors is based on a batch process, but it can be improved upon with twin screw melt extrusion.

## Traditional processing

The polymers are molten with the addition of water. Then the flavors or active ingredients are added, and mixed by vigorous kneading. Depending on the formulation, excess water may need to be removed under vacuum. Thereafter, the melt is cast as a plate and cooled down.

This process is very cumbersome and time-consuming. Also the required material amount is not flexible because it is predetermined by the size of the batch mixer.

Another popular traditional method for encapsulation of flavors  is spray drying. A drawback of this complex, continuous process is the loss of flavors and active ingredients due to high process temperatures. The materials may oxidize, may have a shorter shelf life, and explosion protection measures may even have to be taken for some materials. The high energy consumption for drying in this process also makes it less favorable from an economic standpoint.


Figure 1: The Thermo Scientific Process 11 "Hygienic" Parallel Twin-Screw Extruder

## Encapsulation using twin-screw melt extrusion

Polymers are frequently processed with extruders, so it is an obvious choice to extend this technology for the encapsulation of flavors.

The flexible combination of dispersive and distributive mixing in a twin-screw extruder is perfectly suitable for continuous encapsulation of flavors. The twin screw extruder allows the temperature to be changed throughout the barrel zones, and it has a modular screw design to induce only the amount of shear and thermal energy needed for the process of encapsulation. This prevents unwanted degradation of the sensitive materials.

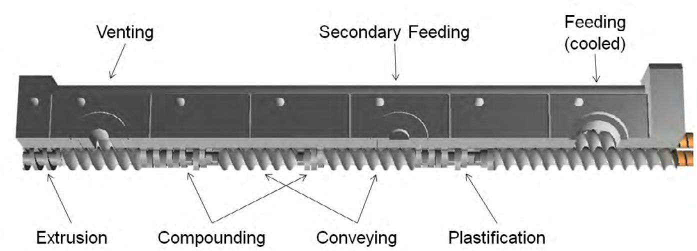



**Figure 2: Schematic of a twin-screw extrusion.**

In the feeding section of the extruder (see Figure 2; material flows from right to left) the polymer matrix material is metered and conveyed into the first mixing zone. Due to the heat and shearing, the polymer is transformed into a homogenous melt. In a secondary feed zone, the flavor is added by means of a liquid feeding pump.

In a further mixing zone, the flavor is now dispersed and evenly distributed into the polymer matrix. At the end of the extruder, the sample pressure is built up to press the compound through a die, and shape it to large number of small strands which are then directly cut into fine pellets by the rotating knife of a facecut pelletizer. Another option is to extrude the melt directly onto chill-rolls which freeze it down and shape the material into flakes.

The Thermo Scientific™ Process 11 "Hygienic" Extruder is the ideal instrument for testing the encapsulation process on a laboratory scale, because it combines the advantages of a compact bench-top extruder, with the full functionality of a production setup. Its modular design enables the optimal adjustment of the extruder barrel and screws to match the application and product needs. All product contact parts are made from stainless, hygienic-grade steel.

As food products are often cleaned with water-based detergents, the high-grade steel provides an advantage over regular extruders which are normally used in polymer processes.

## Testing Equipment

- Extrusion System: Process 11 "Hygienic" Extruder
- Cooling Circulator:
  Thermo Scientific™ Polar Series Accel 500 LC
- Feeder for Premix:
  Gravimetric MiniTwin MT0 for Process 11
- Feeder for Liquid:
  Thermo Scientific™ Masterflex P/S Pump Systems
- Downstream System: Face-Cut Pelletizer

For an encapsulation of a flavor in a sugar matrix, the Process 11 "Hygienic" equipment setup was designed in a way that the sugar was metered into the cooled, first feeding zone of the extruder, with a gravimetric twin-screw feeder. The sugar was then conveyed by the extruder-screw, into the first mixing zone. There the sugar was molten due to the shear and heat generated by the kneading elements. These kneading elements were followed by conveying screw elements. In a co-rotating twin-screw extruder the conveying elements are not totally filled and the melt is not pressurized. As a result the extruder could be opened again and the flavor was added into the molten sugar by the Masterflex P/S peristaltic pump.

Conveying screw elements then transported the mixture into two subsequent mixing sections, where the flavor was dispersed and evenly distributed in the sugar matrix. At the end of the extruder the pressure was built up, and the final compound was pressed through the die head, into the face-cut pelletizer.

## Designing the final product

Figure 3 shows the final product, collected after cooling in the cyclone of the face-cut pelletizer.

**Figure 3: Encapsulated flauror - pelletized.**

thermo scientific

Once the process is developed on the extruder, it is very simple to exchange of the downstream accessories to obtain differently shaped material. Figure 4 shows flakes produced from the same process using a chill-roll (see Figure 5) instead of the face-cut pelletizer. The molten material leaving the extruder is compressed and cooled down between two temperature-controlled rolls and formed into a thin sheet. The cooled sheet is then broken down into flakes by a kibbler device at the end of the chill-roll.

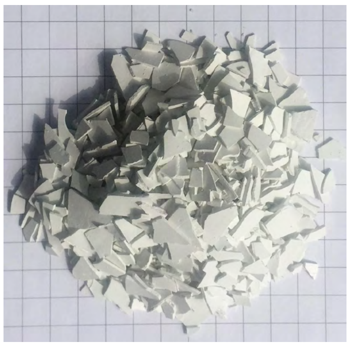


**Figure 4: Encapsulated flauror - pelletized.**


**Figure 5: Chill-roll with kibble.**

## Conclusion

Using twin screw extruders for encapsulation of flavors and ingredients into a sugar or polymeric matrix offers several advantages over traditional processes.

The extruder is a continuous working instrument by nature so the amount of end-product is determined by run time and does not require adaptation via different sized production equipment as traditional batch operation does. Compared to the energy-hungry process of spray-drying, extrusion has milder process conditions and reduces the risk of product denaturation.

Finally the choice of downstream equipment (face-cut pelletizing or chill-roll) can help produce application specific end-product as required.

Find out more at **thermofisher.com/extruders**

ThermoFisher SCIENTIFIC

thermo scientific

# Further information and services

### Customer service

We are committed to delivering top-notch customer support, including tailored service products and fast response times. We offer a comprehensive range of services that can quickly and flexibly respond to various service needs and requests.

### Application laboratories and support

Our fully equipped application laboratories are in constant demand for testing customer samples, and developing and optimizing pioneering applications. We also provide a broad range of product and application solutions, and our team of application specialists is on hand to answer your questions.

### Seminars and training courses

You will find comprehensive training programs, in-house seminars, and practical rheology courses in various locations around the world. **Visit our seminar page** for more information.

### Food rheology and extrusion on the web

Learn more about polymer rheology on our **dedicated web pages**. You'll find application information, videos and more to help engineers and scientists in their daily work.

### Product & application information

Register for application and product information at **thermofisher.com/specoptin** to gain access to latest resources to accelerate your research and improve laboratory productivity.

Find out more at **thermofisher.com/foodextrusion**

ThermoFisher SCIENTIFIC