Continuous extraction of pectin using twin-screw extrusion

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Introduction

Pectins are soluble fibers found in higher plants, which provide firmness and support the plant tissue's structure. They are a type of hydrocolloid, and they often collected in waste streams of the food industry, such as from juice or sugar production. For this reason, pectins are considered sustainable plant polysaccharides from natural sources,[1] whose use as food ingredients supports a circular economy. This, among other factors, makes pectins a food ingredient well accepted by consumers. [2] Pectins are commonly used as thickening and gelling agents. Besides serving as viscosity enhancers, pectins can also be used as emulsifying agents. With such versatility, pectins have become an important ingredient for the food industry.

The main pectin sources are apples, citrus fruits, and sugar beets. Other plants may be used as sources for extraction depending on their local availability.[1] Pectin extraction involves selecting raw materials like citrus peels and apple pomace, washing and drying them, and treating them with a hot acid solution to release the polysaccharides. The mixture is then filtered, and pectin is precipitated using alcohol. The precipitated pectin is washed, dried, ground into a fine powder, and packaged. The quality and yield of pectin depend on the raw material, extraction conditions, and drying methods. A scheme of the process is found in Figure 1.

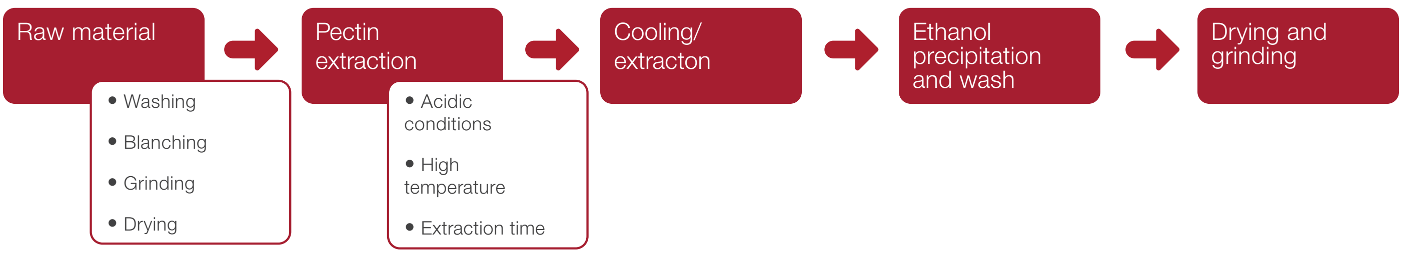

Figure 1. Schematic pectin extraction process.

The extraction of pectin is usually performed with strong acids, such as nitric, sulfuric or hydrochloric acid, and takes several hours to cook in a batch reactor.[3] This kind of process presents many challenges. Strong acids can cause pollution if not managed properly; they require neutralization and careful disposal. Additionally, acidic conditions can corrode equipment, increasing maintenance costs and risking contamination. The high temperatures needed for the process lead to significant energy use and elevated costs. For this reason, research is moving towards “green” extraction methods that use organic acids and aim to increase the extraction efficiency while preserving the techno-functional properties of pectins. This application note showcases the use of the Thermo Scientific™ Process™ 16 Twin-Screw Extruder, a co-rotating twin-screw extruder, as a possible method to obtain pectins in a continuous manner. A twin-screw extruder can help combine the first three processes in a single instrument, reducing the equipment footprint and accelerating the processing time.

Extruder setup

For the extrusion trial, a Process 16 twin-screw extruder was used. For extraction processes, the extruder barrel length is extended by attaching an extraction barrel. Additionally, screws with a 55 L/D ratio are used to combine the grinding and extraction steps within the length of the extruder barrel. An exemplary screw configuration can be found in Figure 2.

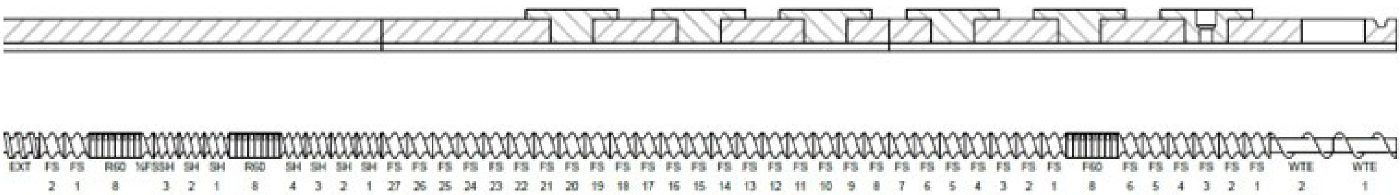

Figure 2. Exemplary screw configuration.

The extraction barrel extension has a split design, allowing it to be easily taken apart for cleaning. The bottom part of the barrel extension is equipped with five holes for inserts, with each insert containing 280 holes with a diameter of 0.5 mm. Users can flexibly attach filter inserts or close the openings depending on their needs. For this application note, trials were conducted with a full set of filter inserts to maximize the extraction capacity of the barrel extension. Figure 3 shows the extraction barrel extension in operation. The extension is attached between the extruder barrel and the die adapter, allowing for the use of different die plates with varying diameters, depending on the desired shape and pressure build-up.

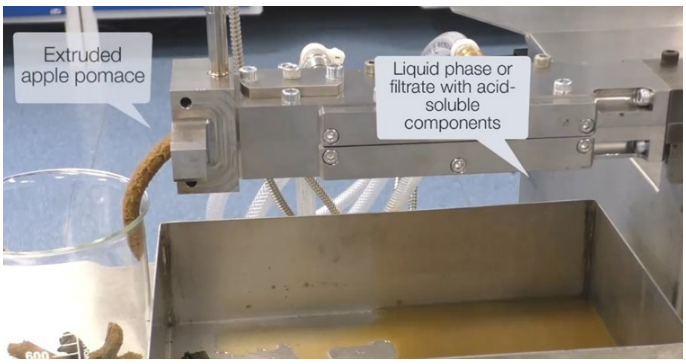

Figure 3. Extraction barrel.

For this application note, dried apple pomace was chosen as the pectin source. A citric acid solution at pH 2 was used as the solvent. The acid-to-solid ratio was set to 3:1, with 2.5 L/h of acid dosed into the second feeding port and 0.5 kg/h of dried apple pomace dosed into the first feeding port. The dried apple pomace was fed into the extruder using a twin-screw gravimetric feeder, equipped with spiral screw elements, which allowed the apple pomace to be fed in whole pieces. Additionally, the extruder screw configuration included wide throat elements at the beginning of the extruder’s barrel (see Figure 2) to accommodate the chunks of dried apple pomace and transport them along the extruder. This setup eliminates the need for an additional milling step to convert the raw material into powder, thereby supporting process intensification.

The extraction barrel extension was connected to a water bath with a separate circulator to maintain the extension temperature at 80 °C. The extraction barrel can also be cooled by the external circulator. This setup can enhance the extraction process, especially for volatile compounds, and provide flexibility to the user.

To assess the yield of soluble compounds under varying conditions, the acid and apple pomace were extruded at two different combinations of speed and temperature settings. The first round of trials was conducted at 200 rpm with all temperature zones set to 100 °C. The second round was conducted at 300 rpm and 120 °C. After extrusion, the filtrate was collected in beakers (see Figure 3). The acid-soluble compounds, including pectin, were then precipitated with ethanol at 90% purity. Figure 4 shows the results of the extruded samples after ethanol precipitation, compared to an untreated sample. As seen in Figure 4, the untreated sample does not show any significant changes after the addition of ethanol. This indicates that compounds like pectin remain inside the cell structure and are not accessible. The filtrate collected from the extruded samples, on the other hand, shows precipitation after ethanol addition. The amount of precipitation strongly depends on the extrusion parameters, with an increase in extracted compounds observed with increasing screw speed and barrel temperature. This indicates that the apple pomace cell structure is disrupted to a greater extent with increasing thermal and mechanical energy, facilitating the extraction of acid-soluble compounds and reflected in the yield. These initial trials prove that extrusion can be used to intensify extraction processes.

To obtain pure pectin, the samples should be centrifuged after precipitation. The insoluble particles are then dried and ground.

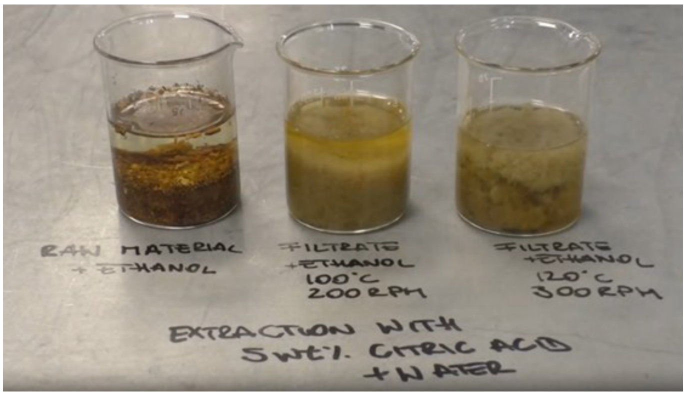

Figure 4. Ethanol precipitation of raw material and filtrates collected during extrusion.

Conclusions

Extrusion, coupled with an extraction barrel, is a suitable option for the extraction of pectin and other compounds. Extrusion provides the necessary combination of thermal and mechanical treatment to disrupt cell structures and facilitate the extraction of solvent-soluble compounds. By choosing the right processing parameters, such as the speed and temperature of the barrel, the yield of extraction can be improved.

Literature

1. [1] Thakur, B. R.; Singh, R. K.; Handa, A. K.; Rao, M.A. (1997): Chemistry and uses of pectin. A Review. In Crit. Rev. Food Sci. Nutr. 37 (1), pp. 47–73.
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