

Supporting Information

Life-Cycle Analysis of Microwave Assisted Pectin Extraction at Pilot-scale

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Additional Experimental details

Degree of Esterification

The degree of esterification was carried out according to the methodology set out by Mizote *et al.*¹ A 2g amount of pectin was added, with stirring, to 200 mL of a mixture of 90 mL water, 10 mL concentrated (37%) HCl and 100 mL isopropanol in a 500-mL flask and the mixture left for 15 min. It was then filtered on Büchner funnel, suction halted, and 300 mL of 65% isopropanol added to the pectin in the funnel with stirring. Suction was resumed and the process repeated until the filtrate was free from chloride. The pectin was then washed with isopropanol and finally with diethyl ether in order to facilitate drying of the pectin. The funnel containing the pectin was kept in a drying oven until the pectin was dry and measuring constant weight. A 0.5g amount of the dried pectin was weighed into a 250mL beaker and moistened with 1 mL of 6% isopropanol. 100 mL of distilled water was added and the beaker placed on a magnetic stirrer and stirred until all of the pectin dissolved (about 15 min). The solution was then titrated with 0.1 M sodium hydroxide solution to pH 7.5, while stirring, the volume of titrant consumed (V_1 mL), which corresponds to the polygalacturonic acid, being noted. The stirring continued, 30 ml of 0.1 M sodium hydroxide solution added and the beaker was covered and left for exactly 30 min. An amount of dilute sulphuric acid, equivalent to 30mL of 0.1 M sodium hydroxide solution, was added and, while stirring, the mixture titrated with 0.1 M sodium hydroxide solution to pH 7.5, the volume of titrant consumed again being noted (V_2 mL). The degree of esterification (DE) is calculated as follows:

$$DE = \frac{100 \times V_2}{V_1 + V_2}$$

Gel Test

The pectin and half of the sugar were added to a buffer solution (pH 3-3.5) with continuous stirring and left to dissolve overnight to ensure the homogeneity of the solution. Afterwards, the solution was heated up to its boiling point and, after cooling, the remaining 50% of sugar was added to make the final ratio 1:60:39 pectin: sugar: buffer by weight. At this point, the solution was heated again (to 100°C) with continuous stirring. After reaching its boiling point, the solution was allowed to cool down to room temperature and stored in the fridge overnight.

Test for Amide Group

The test for the amide group was carried out according to the criteria set out by the World Health Organisation (WHO).² 2 ml of concentrated hydrochloric acid and 50 ml of 60% ethanol was added to 0.5 g of sample, and stirred for 20 min. The sample was then filtered in a sintered glass funnel (grade 4) and washed with 6x10 ml portions of the same HCl/60% ethanol mixture.

The sample was added to 100mL deionised water and 4 ml of this solution was transferred into a 30 mL sample vial. 1 ml of 5 mol L⁻¹ sodium hydroxide was added and mixed to form a gel. Another 30 mL sample was prepared with 2.5 ml boric acid TS and then slowly slid into the other sample vial. The vial was sealed and left overnight at 30°. A colour change from red to green indicates the presence of amide groups due to the release of ammonia.

Loss on Drying

A 0.5g sample of material was weighed into a pre-weighed sample vial and left open for 2 hours and then weighed again to determine the loss on drying according to the WHO criteria.²

Total Insolubles

Total insolubles was carried out according to the criteria set out by the World Health Organisation (WHO) ². A 70 mm glass fiber filter paper (GF/B (Whatman code 1821 070) was dried in an oven at 105° C for 1 h. The filter paper was then transferred to a desiccator containing silica gel, allowed to cool overnight and then weighed (M_1). 1g (S) of the sample was accurately weighed into a 250-ml beaker, 5 ml of 2-propanol was added to disperse the sample and stirred. Whilst stirring, a pre-filtered mixture of 100 ml 0.03 mol L⁻¹ sodium hydroxide containing 0.1% (w/w) sodium ethylene diamine tetra-acetic acid (EDTA, Na salt) was added, stirred for 30 min at room temperature, then heated to boiling. The hot solution was filtered through the prepared glass fiber paper under vacuum. The flask was washed five times with 100 ml of pre-filtered warm (50°) water. The filter paper, along with the residue, was dried in the oven again at 105° for 1 h, then transferred to the silica-containing desiccator, left to cool overnight and re-weighed (M_2). The percentage of total insoluble was calculated as follows:

$$Total\ Insolubles\ (\%) = \frac{(M_2 - M_1)}{S} \times 100$$

Nitrogen Content

Nitrogen content was measured through CHN analysis on an Exeter Analytical Inc. CE-440 analyser. The sample was weighed into a tin capsule then placed in a nickel sleeve and injected into a high temperature furnace (975 °C) and burnt in high purity oxygen under static conditions. The tin capsules used for the sample container allow an initial exothermic reaction to occur, raising the temperature of combustion to over 1800°C. A further dynamic burst of oxygen was added at the end of the combustion process, to ensure total combustion of all inorganic and organic substances.

The resulting combustion products were passed through specialised reagents to ensure full combustion of any methane produced and to remove halogens, sulphur and phosphorous. This process ultimately resulted in the production of CO_2 from the elemental carbon, H_2O from the hydrogen, and NO_x from the nitrogen. The combustion gases were then passed, using helium as a carrier gas, through a tube packed with pure copper wire at 620°C , to remove excess oxygen and to reduce the NO_x to elemental nitrogen. After this stage the gases enter a mixing chamber, to ensure a homogeneous mixture, at constant temperature and pressure, was delivered to the detectors.

The mixture was then passed through a series of high-precision thermal conductivity detectors, each containing a pair of thermal conductivity cells. Between the first two cells is a water trap; the differential signal between the cells being proportional to the water concentration (which is a function of the amount of hydrogen in the original sample). Between the next two cells is a carbon dioxide trap for measuring carbon. Finally, nitrogen is measured against a helium reference and the analyser is then purged with a burst of pure helium, to return conditions to the starting position and thus to avoid carryover.

Table S1. Temperature and pressure settings for the freeze-drying cycle.

| Thermal Treatment Stage | | | | |
|-------------------------|-----------------|------------|-----------------|------------------------------|
| Step | Shelf Temp (°C) | Time (min) | Pressure (mbar) | Description |
| 1 | 20 | 10 | Atm | Hold |
| 2 | -30 | 100 | Atm | Ramp |
| 3 | -30 | 240 | Atm | Hold |
| Primary Drying Stage | | | | |
| 4 | -30 | 120 | 200 | Hold |
| 5 | -10 | 20 | 200 | Ramp |
| 6 | -10 | 9200 | 200 | Hold |
| Secondary Drying Stage | | | | |
| 9 | 20 | 40 | 200 | Ramp |
| 10 | 20 | 3890 | 200 | Hold |
| Actions at End of Cycle | | | | |
| | 20 | N/A | Atm | Backfill with N ₂ |

Power Consumption

Figure A1 shows the power consumption over time for the microwave run, as well as the temperature of the mixture. As can be seen, the power drops rapidly upon reaching the required temperature, meaning the actual power consumption is far less than the projected 9 kWh. Figure A2 shows the same data focussed on the 90-minute run from when the desired temperature is reached. This shows good feedback between the temperature probe and microwave head as the

power shifts immediately upon the temperature deviating from 95 °C. Ultimately, the temperature holds well at ± 4 °C before stabilising at ± 0.5 °C after 50 minutes into the run. Whilst this may seem inefficient or undesirable at first glance (taking close to half the run time) it is important to note that this is currently being carried out as a batch process with the rig being emptied and cooled between each run as opposed to having the material fed through and held at temperature continuously.

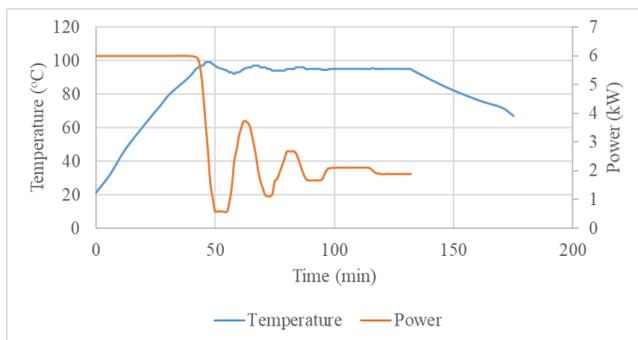


Figure S1. The temperature and power profile.

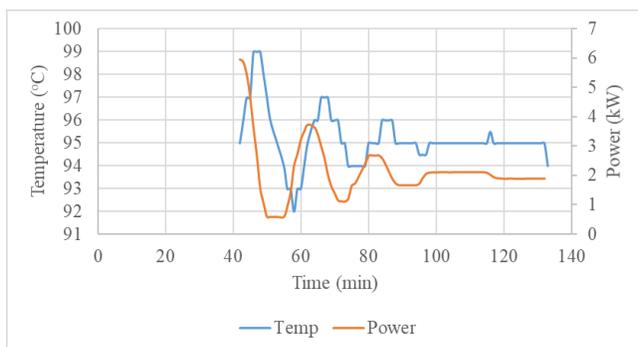


Figure S2. The temperature and power profile zoomed in over the 90 minute run.

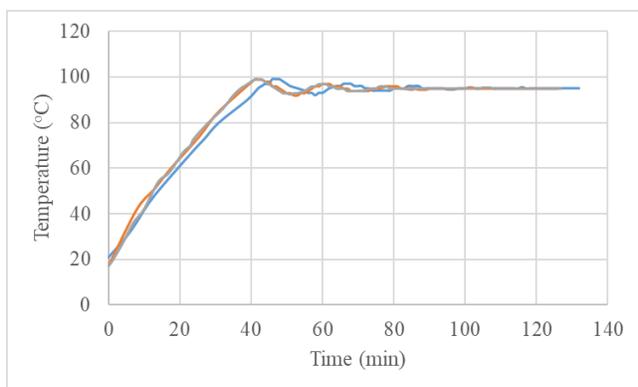


Figure S3. Composite temperature profile of 3 runs on the microwave.

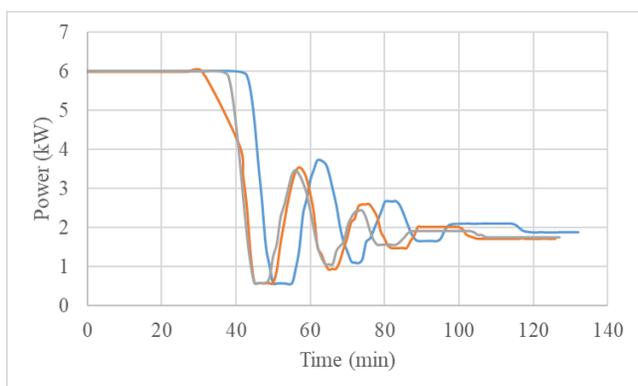


Figure S4. Composite power consumption of 3 runs on the microwave.

1. Mizote, A.; Odagiri, H.; Tôei, K.; Tanaka, K., Determination of residues of carboxylic acids (mainly galacturonic acid) and their degree of esterification in industrial pectins by colloid titration with Cat-Floc. *Analyst* **1975**, *100* (1196), 822-826.
2. FAO, *Evaluation of certain food additives: eighty-second report of the Joint FA*. World Health Organization: WHO Expert Committee on Food Additives, 2016.