Continuous and pulsed ultrasound pectin extraction from navel orange peels

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ABSTRACT

Pectin is a valuable product (up to 30 Skg⁻¹) that makes up 20–30% of an orange’s peel. The commercial extraction is lengthy (up to 6h) and energy intensive as it requires heating aqueous solutions (60–100 °C). Ultrasound speeds up the extraction process reducing processing time by macroscopic and microscopic mixing by acoustic cavitation. We adopted an ultrasonic horn to deliver a rated power of 500 W at amplitudes of 20%, 40%, and 60% with and without pulsation to extract pectin from waste orange peels. These correspond to power densities of 0.08 W ml⁻¹, 0.16 W ml⁻¹ and 0.24 W ml⁻¹, respectively. The extractions operated at a pH of either 2 or 3. The experimental data agree with the fitted values from the statistical model (R² = 95.5%). The model confirms our predictions that yield increases with amplitude/power density and decreasing pH. The highest yield was (11%) at a pH of 2 and with continuous ultrasonic irradiation at a power density of 0.24 W ml⁻¹. There is only a 1.3% difference between this datum and pulse ultrasound mode (1 s on/1 s off) at the same conditions — a Student’s t test confirmed that there was no significant difference in yield between continuous and pulse mode. However, pulsing is more efficient in that it consumes less than half the energy of continuous operation (80 kJ vs. 190kJ).

1. Introduction

Food waste is a growing issue that causes a plethora of problems, including greenhouse gas emissions, water contamination through leaching, and the need for additional landfills [1]. Grizzetti et al. estimated that global food waste outputs 6,300,000 t of nitrogen into the environment per year [2], which engenders other phenomena such as acid rain and eutrophication. Among the massive amounts of food waste generated world-wide, orange peels account for 11,957,000 t [3]. Valorization of food waste is a key strategy in alleviating environmental strain.

The interest in orange and other citrus fruit peels/waste is due to pectin. Pectin is a family of polysaccharides found in the cell walls of plants, which are composed of covalently bonded galacturonic acids [4]. In addition, its composition depends on environmental factors such as moisture, rate of growth, and location [5]. All pectin types contain 1,4-linked α-d-galactosyluronic acids [6,7]. The three main pectins are: homogalacturan, making up 65% of pectin, followed by rhamnogalacturan I at 25%, and the remainder being rhamnogalacturan II, the more complex of the three [6,8]. Pectin is abundant in the middle lamella where it acts as a binding agent for plants’ cell walls [8,5]. Food-grade pectin sources are citrus peels and apple pomace [5] which can have a dry pectin content of up to 30% and 15%, respectively [9]. Pectins’ presence in the food industry is thanks to their ability to form gels in the presence of Ca²⁺ ions or sugar and acid [5,10]. Traditionally, pectin is used in jams, fillings and toppings for baking, glazes for pastries, low-calorie soft drinks, sorbets, chutneys and sauces, in the dairy industry, pharmaceuticals and has healing properties [10].

Pectin’s value is not solely food-related. They also favourably affect lipid metabolism, decrease cholesterol levels in humans [11], improve glucose tolerance [12], and prevent pathogens from attaching to the intestines’ inner wall [13–15]. Pectin demonstrates immunostimulating, anti-metastasis, anti-ulcer, anti-nephrosis activities, and drug-delivering capabilities [16]. Modified citrus pectin (MCP) decreased blood serum levels of lead in hospitalized children; a promising chelating agent [17]. Furthermore, MCP circulates around the body and is anti-cancerous; it activates T-cytotoxic cells, B-cells and NK-cells [18]. The latter inducing

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cancer cell death against K562 leukaemic cells in culture [18].

The pectin market has been growing globally for the last 15 years with production increasing from 30,000 t to 60,000 t per year and sales are predicted to exceed $ 2.4 billion by 2020 [19]. In 2016, the IMR with production increasing from 30,000 t to 60,000 t per year and sales [20]. In a commercial setting, nitric acid extracts pectin from the raw material, in conditions ranging from a pH of 1.5 to 3, 60°C to 100°C, and from 0.5 h to 6 h [10,21,22]. Isopropanol, methanol, or ethanol precipitates the resulting liquid to form a powder [10]. The traditional extraction method for pectin is outdated and being overtaken by newer processes such as ultra-high pressure extraction [23], moderate electric field extraction [24], ultrasound [25,26], microwaves or a combination of these techniques [27]. In addition, the conventional method of extraction (e.g. hydrodistillation), is a lengthy process and requires high amounts of energy (boiling), and the extracted pectin thermally degrades because of the long heating period [24,28-30]. Pectins are interlinked with many polymers in the cell walls, which often prevents its release [31]. Microwave pretreatment aided pectin extraction from orange peels by destroying these links (e.g. parenchymal cells) and inactivating the endogenous enzymes such as pectinesterase [32]; yield increased by 190% and 250% compared to a control group from 1996 and 1997, respectively [31].

Ultrasound is a green technology that increases selectivity, reduces reaction time and induces macro- and micro-mixing through acoustic cavitation; it creates bubbles/cavities that upon collapse release enormous amounts of energy that is made available to the reaction [33-39]. It is applicable in a wide range of fields: materials chemistry [40,41], biomedical uses such as gene and drug delivery [42,43], biofuel and chemical production [44,45], advanced oxidation processes for wastewater treatment [46,47], and dehydroxylation or modification of protein properties (e.g. gelation) in food engineering [47,48]. For example, ultrasound greatly accelerates mass transfer without compromising product stability thus maintaining the quality of fruits and vegetables while minimizing energy losses [49,47]. Gonzalez-Rivera et al. demonstrated that coupling ultrasound with microwave hydro-distillation reduces energy consumption by 60% compared to conventional hydrodistillation [27]. In fruits and vegetables, cavitation creates micro-jets on the surface of vegetable tissue fracturing the cell wall in addition to mixing at the micro-level [50-52]. This allows the solvent to penetrate the internal structure and facilitates pectin release [53,54,25]; yield increased by 190% and 250% compared to a control group from 1996 and 1997, respectively [31].

We mixed the peels ((5.00 ± 0.01) g) with distilled water ((150.0 ± 0.5)ml), in a 200 ml jacketed glass reactor, at a solid-to-liquid ratio of 1:30. Xu et al. tested solid-to-liquid ratios between 1:30–1:70 and found that 1:50 was optimal [25]. However, we were constrained to 1:30 due to the size of our reactor and the mass of our orange peels. Nitric acid 70% from Sigma–Aldrich brought the solution to a pH of 2 or 3 (Table 1). Pectins are most stable between a pH of 3 to 4; at lower pHs the methoxyl, acetyl, and neutral sugar groups degrade [68], so we did not venture beneath a pH of 2. A magnetic stirring plate set at 500 rpm agitated the solution. A Sonics & Materials 500 W ultrasonic horn with a 13 mm diameter replaceable tip sonicated the solution, with and without pulses, at amplitudes of 20%, 40%, and 60% for 2 min to 60 min. Kikuchi and Uchida’s calorimetric method, with water as the liquid medium, determined the power — 11.5W, 23.0W, and 34.2W, respectively — associated to each of our amplitudes [69].

The pulse cycle was 1s on and 1s off. We immersed the horn tip (2.0 ±0.1 cm) into the solution. A Thomas Scientific thermostatic bath set at 15°C, with water as the circulating liquid, kept the temperature below 40°C for all experiments as we wished to avoid overheating that might have evacuated the system from acoustically active gas bubble

| Time min | pH | Pulse | Amplitude % | Pectin yield (mass fraction) |
|----------|----|-------|-------------|-----------------------------|
| 60       | 3  | No    | 20          | 0.04                        |
| 60       | 3  | Yes   | 20          | 0.03                        |
| 60       | 3  | No    | 40          | 0.06                        |
| 60       | 3  | Yes   | 40          | 0.05                        |
| 60       | 3  | No    | 60          | 0.07                        |
| 60       | 3  | Yes   | 60          | 0.06                        |
| 60       | 3  | -     | -           | 0.01                        |
| 60       | 2  | No    | 20          | 0.06                        |
| 60       | 2  | Yes   | 20          | 0.06                        |
| 60       | 2  | No    | 40          | 0.11                        |
| 60       | 2  | Yes   | 40          | 0.08                        |
| 60       | 2  | No    | 60          | 0.11                        |
| 60       | 2  | Yes   | 60          | 0.10                        |
| 60       | 2  | -     | -           | 0.02                        |
nuclei [70]. At the end of each reaction, a porcelain Buchner filter with cellulose filter paper (particle retention of 11 to a pH of 2 eluted the samples in isocratic mode at 0.5 mL min

Table 2

| Time (min) | pH | Pulse | Amplitude (%) | Pectin yield (mass fraction) |
|-----------|----|-------|---------------|-----------------------------|
| 2         | 2  | No    | 60            | 0.07                        |
| 5         | 2  | No    | 60            | 0.08                        |
| 10        | 2  | No    | 60            | 0.09                        |
| 10        | 2  | No    | 60            | 0.08                        |
| 10        | 2  | No    | 60            | 0.09                        |
| 20        | 2  | No    | 60            | 0.09                        |
| 30        | 2  | No    | 60            | 0.10                        |
| 45        | 2  | No    | 60            | 0.11                        |
| 60        | 2  | No    | 60            | 0.10                        |
| 2         | 2  | Yes   | 60            | 0.07                        |
| 5         | 2  | Yes   | 60            | 0.06                        |
| 10        | 2  | Yes   | 60            | 0.07                        |
| 20        | 2  | Yes   | 60            | 0.07                        |
| 30        | 2  | Yes   | 60            | 0.09                        |
| 45        | 2  | Yes   | 60            | 0.10                        |
| 60        | 2  | Yes   | 60            | 0.11                        |

The pectin yield from citrus waste is comparatively lower than other reported values [25,60] (with ultrasound) and the commercial benchmark of 20% to 30% (without ultrasound) [9]. We attribute this to our low power density of 0.23 W mL⁻¹ and our voluntary omission of heating the system to reduce energy consumption. Our conditions were in fact below the extraction threshold recommended by May et al. [10] and our power density was approximately half of the reported power densities for extraction [25].

Some authors report citric acid to be a more suitable acid for pectin extraction than nitric acid as it is the less depolymerizing and de-esterifying [72–74]. However, 0.3 M HNO₃ extracted almost 3 times more pectin than citric acid [75].

We focused on the interactions between varying ultrasound powers, pulsing, and pH. We kept our temperature below 40°C (Fig. 1) for all reactions in an effort to highlight the effect of ultrasound [76].

3.2. Ultrasound effects

Pectin yield increases with increasing amplitude/power (Fig. 2). In other words, the radius/size of active bubbles during cavitation are larger at higher amplitudes [77,78] and thus provide more energy to the reaction.

This increase is almost linear between the three amplitudes, and more so with the pulsed experiments. In addition, albeit the ultrasonic energy input in the continuous US experiments is much higher than their pulsed counterparts (190 kJ vs. 80 kJ — read from the instrument) the yields are similar; a 2.5% difference in yield between the 20% amplitudes and a 1.3% between the 60% amplitudes. A Student’s t test confirmed that there was no significant difference between the continuous and pulsed mode data at a pH of 2 (p = 0.76 with a 95% confidence level). During continuous irradiation, cavitation activity suffers in the presence of many bubbles [25]. This can be attributed to a “saturation effect” in which cavitation bubbles crowd the probe tip and hinder the transmission of energy to the reaction [79]. In addition, more bubbles results in more collisions between them and thus modify their ideal spherical shape and decreasing their energy efficiency upon collapse.
[80], Zhang et al. found that pectin degradation occurs with increasing ultrasound intensity [76]. In fact, the β-elimination rate (depolymerization) increases with temperature [81]. At lower intensities there is less degradation because cavitation can be induced at a minimum power value [82], which is why we can extract pectin and not degrade it. Pulsed ultrasound generates less energy than continuous irradiation — there is a decrease in the number of bubbles during cavitation [83]. However, this increases the availability of power and energy storage capacity for each bubble during expansion [83]. As bubble density decrease entrenches fewer interactions and allows them to maintain a spherical shape into deeper stages of collapse thereby increasing the energy concentration during implosion [83]. In other words, bubbles that maintain their shape into deeper stages of collapse compensate for reduced exposure to sonication. A duty cycle/pulse-mode of 50% led to the highest yields compared to other duty cycles [25,84,76], Xu et al. worked at duty cycles of 33%, 40%, 50%, 60%, 70%, and 80% [25]. At 33% and 40%, the total yield is reduced because the reaction had not been exposed to enough ultrasonic cavitation [25]. On the other hand, they found that at 60% and 70% the “saturation effect” reduced yields [25]. At 80%, the increased sonication time compensated for the more frequent collisions between bubbles [25]. At 50%, there is a balance between the sonication time and shape of bubble to maximize yield.

3.3. pH effect

There is stronger linear relationship in the experiments at pH 3 (Fig. 3) but a lower yield overall as compared to (Fig. 2). In addition, a Student’s t-test confirmed that there was no significant difference between the continuous and pulse mode data at a pH of 3 (0.64 with a 95% confidence level).

Increasing the ionic strength of the reaction mixture increases pectin yield [74]. Venkatanagaraju et al. and Hamidon et al. [85,86] obtained high yields of pectin at pHs of 2 and below. This highlights the importance of a low pH for extraction [74]. Acids are the best extractors of pectin because of their ability to prize heavily bound protopectin from cell walls [74]. Hydrogen ions, in high concentrations, engages pectin hydrolysis from protopectin; acids with a high ionic strength worked at duty cycles of 33%, 40%, 50%, 60%, 70%, and 80% [25]. At 33% and 40%, the total yield is reduced because the reaction had not been exposed to enough ultrasonic cavitation [25]. On the other hand, they found that at 60% and 70% the “saturation effect” reduced yields [25]. At 80%, the increased sonication time compensated for the more frequent collisions between bubbles [25]. At 50%, there is a balance between the sonication time and shape of bubble to maximize yield.

3.4. Statistical model

We regressed the experimental data first with a linear model (without interactions) to identify significant factors then by a power law model to assess the relative importance of each. We applied a pseudo Arrhenius expression to account for temperature (exp(-Ea/(RT))). However, its contribution to explaining the variance in the data was minimal and so we omitted it. The power law model explains 95% of the variance in the data (excluding temperature). The two most significant factors on pectin yield are power, P and pH - it increases with the square root of P and is inversely proportional to pH. Yield increases with the 1/4 power with pulse (either on or off) and increases with the 1/12 power with time for this data set.

\[
y = 0.21 \frac{P^{1/2}}{\text{pH}^{1/4} t^{1/12}}
\]

where,

- $P$ is power density (W mL$^{-1}$) (0.08 W mL$^{-1}$, 0.16 W mL$^{-1}$, or 0.24 W mL$^{-1}$),
- pH at 2 or 3,
- $t$ is time in min,
- $p$ is pulsing (values of 2 and 3 are assigned to pulsing and continuous irradiation, respectively).

We also extended our model to predict yields at the optimal power density (0.4 W mL$^{-1}$) reported by [25]. Despite this, the yield only increased by 2% across the board. Thus, temperature is an inhibitor for extraction below 60°C, i.e. the minimum required temperature during conventional extraction [10]. Furthermore, time’s influence is mediocre because we are operating at low power density and temperature. If we increased the time of our reaction we suspect our yield would rise. However, extending the power density to 0.4 W mL$^{-1}$ and potentially up to 0.8 W mL$^{-1}$ at 25°C can overcome temperature-related inhibitions and intensify the role of time. Yet, Xu et al. found that pectin yield decreases at power densities greater than 0.4 W mL$^{-1}$ combined with heating at 60°C [25]. We attribute this to ultrasound being more performant at lower temperatures (i.e. 25°C) [87] allowing for enhanced extraction above 0.4 W mL$^{-1}$. The combination of high power densities (≥0.4 W mL$^{-1}$) with an elevated temperature (60°C) is cause for degradation [25].

There is a small difference between the experimental data and the values fitted by the statistical model, $R^2 = 95.5\%$ (Fig. 4).

This follows the trends we see in our data and our predictions that pectin yield increases with: increasing amplitude/power, decreasing pH (i.e. more acidic), and a longer reaction time (because our amplitude/power is not high enough for degradation).

![Fig. 3. Pectin yield as a function of pulse mode at pH = 3. All experiments were held for 60 min at constant temperature. Ultrasound pulsed at intervals of 1 s. Symbols & lines represent experimental data and predicted values, respectively. Symbol size represents standard deviation, n = 6.](image3.png)

![Fig. 4. Predicted vs. experimental pectin yield.](image4.png)
4. Conclusions

We adopted an ultrasonic horn at 20kHz to deliver 0.08 W mL\(^{-1}\), 0.16 W mL\(^{-1}\), and 0.24 W mL\(^{-1}\) with and without pulsed ultrasound to extract pectin from waste orange peels at a pH of 2 or 3. The design of experiments was a full factorial. There was no significant difference between continuous irradiation and pulsing at 0.24 W mL\(^{-1}\) despite the great difference in energy consumption (190kJ vs. 80kJ). We believe ultrasound operating on a pulse-mode is favourable to its counterpart, especially if scale-up is envisioned. The maximum yield was 11\% which could have been in line with other reported values if we: heated the system or increased the power density. Future work in comparative studies of pectin extraction from different citrus fruits and exploring pectin formation by monitoring the growing conditions of the fruits would be valuable. In conclusion, pectin is a valuable resource that is accessible in large amounts from orange and other citrus fruit peels — efforts should be taken to valorize what is currently being considered waste.

CRediT authorship contribution statement

N.A. Patience: Methodology, Software, Formal analysis, Investigation, Data curation, Writing - original draft, Writing - review & editing, Visualization. D. Schieppati: Methodology, Validation, Investigation, Writing - original draft, Writing - review & editing. D.C. Boffito: Conceptualization, Methodology, Validation, Investigation, Resources, Writing - original draft, Writing - review & editing, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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