POSTER COMMUNICATION

Experimentation and configuring continuous ultrasound assisted extraction pilot plant operation

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Abstract. In this study continuous pilot plant configuration for helical flow type Ultrasound Assisted Extraction (UAE) reactor is described, and carotenoid extraction results from carrot-water extraction system are presented. The pilot plant configuration consists of all necessary unit operations from washed raw material to extracted natural ingredient product. According to our best knowledge the published data from the whole processing line of continuous UAE has not been described. One of the subjects in this study has been patented in-house UAE design which enables to prolong extractable solids material residence time under ultrasound field. Process operation was verified with several hour pilot tests. Extraction results have been presented in case of carotenoids from fresh carrots and strawberry extract from frozen strawberries. The extracted carotenoid concentration was improved 40\% comparison to macerated carrot results, and absorbance ratio of strawberry extract increased by factor 5.

Keywords: ultrasound extraction, continuous process, natural ingredients, pilot process
1. INTRODUCTION

Large amount of process intensified extraction studies using ultrasound have been published during last two decades but only few detailed process configuration studies can be found from continuous large scale processes. The reactor configurations have been circulating loop reactors or flow-through types; solid-liquid separation methods have been decanters, plate-frame filters or membrane filters. (Eom, S.J. et al., 2020; Grillo, G. et al, 2020; Alexandru, L. et al., 2013). Khadhraoui et al. (2021) have reviewed hybrid techniques with UAE. The proposed solutions were based on pressure extruding, where filter cake was mixed with solvent before ultrasound extraction for the rest of the cake. The total process configurations have not been reported. Several scientific peer-reviewed journal articles on continuous UAE processes have described pilot scale equipment and ultrasound effects but the whole process descriptions from raw materials to extracted products are not available (Rodriguez et al, 2022; Dorosh et al, 2020; Clodoveo, M., 2019; Geow et al, 2021). However, process descriptions of full-scale continuous UAE process for virgin oil extracton using flow-through ultrasound based extraction reactor were viewed by Clodoveo et al. (2017) and by Amirante and Clodoveo (2019). The sonication residence times were less than 10 minutes due to installation of US probes directly to main pipe flow. Martinez-Guerra and Gnaneswar-Gude (2015) studied continuous UAE process for vegetable oil transesterification process. The process was described in more detail from oil feed to biodiesel product although oil extraction was not included in the description. In patent survey including keyword “ultrasound assisted extraction” between 2009-2022 70 relevant patents were found related mainly to specific medicinal, pigment, oil and phenolic compounds separation and sonoreactor inventions. Durkacz and Cullen (2020) have patented process alternatives as block diagram level for extraction of cannabis oil in which main sequential unit operations were: raw material washing, shredding/grinding, solvent mixing, pumping, ultrasonication, filtering, solvent recovery and product separation. In another patent Gu&Du (2016) presented their invention of combining US probes into a conveyor system, and reporting example continuous processes at unit operation level.

In our previous work (Tamminen et al., 2022a) flow-through ultrasound assisted extraction (UAE) of chlorophylls and carotenoids was presented. Continuous flow-through reactor improved significantly chlorophyll and carotenoid yields compared to just mixing solvent extraction conditions. The novel reactor geometry is described by Tamminen et al (2022b).
Patented configuration consists of ultrasound source and helical flow channel inside reactor including mixing elements which basically aid radial mixing in the reactor.

In this study pilot plant configuration for helical flow type UAE reactor is described, and carotenoid extraction results from carrot-water extraction system are presented.

2. METHODS

Pilot plant configuration is presented in Fig. 1. The process configuration (see Table 1) consists of feed solvent tank, biomass shredder, high-shear (HS) mixer, hose pump, ultrasound (US) extractor, vibrating sieve for pre-treatment of solid-liquid removal, and screw-press for final mother liquor removal. The equipment configuration is flexible due to the camlock hose connections. For this specific reactor configuration the solids tend to settle on the helical blade, and therefore intermittent fluid pressure variation is used to flush solids which is beneficial for increasing solids residence time while fluid residence time is kept constant. Carotenoid extraction analytics is based on UV/VIS spectrometer (Agilent Cary 8454 UV-Vis), and is described by Tamminen et al. (2022), Perez-Galvez (2020), and Lichtenthaler and Buschmann (2001). Particle sizes were analyzed using Malvern Mastersizer 3000.

Table 1. Main equipment in pilot configuration.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Type and main specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biomass shredder</td>
<td>Aple press ESE-055: motor nominal power 0.55 kW, blade opening 28x8.5 mm, rotation speed 1350 rpm</td>
</tr>
<tr>
<td>High-shear (HS) mixer</td>
<td>Silverson AX-5: motor nominal power 0.75 kW, max rotation speed 6000 rpm, max tip speed 16 m/s, Duplex mixer diameter 56.1 mm.</td>
</tr>
<tr>
<td>Hose pump</td>
<td>LPP-D15: max. pressure 16 bar, max flow rate 2.5 l/min.</td>
</tr>
<tr>
<td>Ultrasound extractor</td>
<td>Weber ultrasonics Sonopush mono: max. nominal power 2000 W, 25 kHZ frequency, LUT in-house design reactor vessel, ultrasound sound dampening housing (60-68 dB).</td>
</tr>
<tr>
<td>Vibrating sieve</td>
<td>Virto VPB 450: Sieve size 0.15 mm, motor power 0.21 kW.</td>
</tr>
<tr>
<td>Screw-press</td>
<td>Angel 20K-GS: motor nominal power 0.4 kW, max. screw speed 1450 rpm, 2x33.5 cm length screws.</td>
</tr>
<tr>
<td>Auxiliary equipment</td>
<td>Lauda WK 4600 thermostat for temperature control, pressurized air operated AKO VMC 25 hose valve for pressure control at US reactor unit.</td>
</tr>
</tbody>
</table>
Figure 1. On-site extraction production line. Processing capacity of pulp is 42 L/h, Power consumption 9 kWh, US power 2 kW, Solid-to-liquid separation efficiency 90%, nominal batch size 120 L. Flexibility in configuring equipment due to hose pipe connections.

3. RESULTS

Pilot-scale extraction tests were performed for carrots as a model compound to extract total carotenoids. Process conditions are collected into Table 2 and in Fig. 2. In addition to carotenoid extraction from carrot-water feed, the pilot has also been successfully tested for production of strawberry extract in 50 vol-% ethanol at 23±1 °C, see Table 3 and Fig. 3.

Extraction results (Fig. 2) show significant increase in total carotenoids concentration 8.5-9.7 mg/l compared to feed tank 6.3 mg/l. The total carotenoid concentration in carrots is is between 14.9-40 mg/l with R/S -ratio 0.15 and data from Khoo et al. (2011). The solids particle size D90 was 1.4-1.6 mm after shredding and HS-mixing. The overall extraction
intensification is due to high shear wet milling and ultrasound reactor effect. Reactor pressure variation cycles with AKO VMC 25 valve from 1 bar to 2.5-3 bar enables solids collection to reactor and flushing from reactor during operation. Solids flushing cycle in experiments was 20 minutes, and solids collecting time to reactor was 35 minutes. In this manner ultrasound treatment time to extractable solids is prolonged compared to solid suspension residence time in reactor zone. However, the carotenoid concentration in screw press filtrate was 13.7-15.8 mg/L which still leaves room for R/S-ratio optimization as vibrating sieve mother liquor flow could be decreased. In addition to carotenoid extraction from carrot-water feed, the pilot has also been successfully used for production of strawberry extracts from frozen strawberries to 50 vol-% ethanol at 23±1 °C, see Fig. 3. Substantial extraction improvement was found based on UV/Vis absorbance with Pilot in comparison to macerated extraction. Particle size in solids outlet was 1.4-1.6 mm (strawberries).

Table 2. Process conditions of carotenoid extraction from carrots.

<table>
<thead>
<tr>
<th>Process parameters</th>
<th>Process values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reactor residence time</td>
<td>10 minutes</td>
</tr>
<tr>
<td>Extraction temperature</td>
<td>31±2 °C</td>
</tr>
<tr>
<td>Raw material-to-solvent volume ratio</td>
<td>0.15</td>
</tr>
<tr>
<td>US power setting</td>
<td>1500 W, US power on 2-4 minutes and 1 minute power off</td>
</tr>
<tr>
<td>Total pilot run-time</td>
<td>180 minutes</td>
</tr>
</tbody>
</table>

Table 3. Strawberry extract in 50 vol-% ethanol. Total pilot run-time 120 minutes.

<table>
<thead>
<tr>
<th>Process parameters</th>
<th>Process values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reactor residence time</td>
<td>10 minutes</td>
</tr>
<tr>
<td>US Power setting</td>
<td>2000 W continuously without pulses</td>
</tr>
<tr>
<td>Raw material-to-solvent volume ratio</td>
<td>0.8</td>
</tr>
</tbody>
</table>
Figure 2. Carrot extraction results, product concentration as average of mother liquor outlets from vibrating sieve and screw press.

Figure 3. UV/Vis absorbances of strawberry extract in 50 vol-% ethanol and macerated extract. Absorbance-ratio: UAE Strawberry extract / Macerated Strawberry extract = 5.
4. CONCLUSIONS

The presented pilot plant has been developed for extraction intensification where combination of high-shear mixing, ultrasound processing and screw pressing takes place. The patented ultrasound device also allows the prolongation of ultrasound time for extracted solids.

ACKNOWLEDGEMENTS

The Authors wish to thank Business Finland for financial support NATINREC -project (1195/31/2019).

REFERENCES


Introduction

Large amount of process intensified extraction studies using ultrasound have been published during last two decades but only few detailed process configuration studies can be found from continuous large scale processes [1, 2, 3]. The reactor configurations have been circulating loop reactors or flow-through types; solid-liquid separation methods have been decanters, plate-frame filters or membrane filters.

In our previous work [4] flow-through ultrasound assisted extraction (UAE) of chlorophylls and carotenoids was presented. Continuous flow-through reactor improved significantly chlorophyll and carotenoid yields compared to just mixing solvent extraction conditions. The reactor geometry is described in [5]. Patented configuration consists of ultrasound source and helical flow channel inside reactor including mixing elements which basically aid radial mixing in the reactor.

Carotenoid extraction analytics is based on UV/Vis spectrometer, and is described in more detail by Tamminen et al. [4]. Particle sizes were analyzed using Malvern Mastersizer 3000. The process conditions are described in Table 1.

Results and discussion

Extraction results (Fig. 2) show significant increase in product flow (mother liquor streams from vibrating sieve and from screw press) carotenoid concentration 8.5-9.7 mg/L, compared to feed tank 6.3 mg/L. The average total carotenoid concentration in carrots is 18.6 mg/L. The solids particle size 0.99 mm. This was 1.4-1.6 mm after shredding and HLS-mixing. The overall extraction intensification is due to high shear wet milling and ultrasound reactor effect. It is noted that reactor pressure variation cycles from 1 bar to 2.5-3 bar enables solids flushing from reactor during operation. Solids flushing cycle in experiments was 20 minutes, and solids collecting time to reactor was 35 minutes.

The carotenoid concentration in screw press filtrate was 13.7-15.8 mg/L which still leaves room for R/S-ratio optimization as vibrating sieve mother liquor stream could be decreased.

In addition to carotenoid extraction from carrot-waster feed, the pilot has also been successfully used for production of strawberry and herb mixture extracts to 50 vol-% and 60 vol-% ethanol at 23±1°C, see Fig. 3. Substantial extraction improvement was found based on UV/Vis absorbance with Pilot in comparison to macerated extraction. Particle size in solids outlet was 1.4-1.6 mm (strawberries), and 2-3 mm (herbs).

Methods

Pilot plant configuration is presented in Fig. 1. The process configuration consists of feed solvent tank, biomass shredder, high-shear (HS) mixer, hose pump, ultrasound (US) extractor, vibrating sieve for pre-treatment of solid-liquid removal, and screw-press for final mother liquor removal. The equipment configuration is flexible due to the camlock hose connections. For this specific reactor configuration the solids tend to settle on the helical blade, and therefore intermittent fluid pressure variation is used to flush solids which is beneficial for increasing solids residence time while fluid residence time is kept constant.

Figure 1. On-site extraction production line. Processing capacity of pulp is 42 L/h, Power consumption 9 kWh, US power 2 kW, S/L separation efficiency 90%, Batch size 120 L. Flexibility in configuring equipment due to hose connections.

Figure 2. Extraction results, product concentration as average of mother liquor outlets from vibrating sieve and screw press.

Figure 3. Herb extract sample in 60 vol-% ethanol (left), and strawberry extract in 50 vol-% ethanol (middle). Pilot strawberry extract comparison to macerated strawberry extract (right). Absorbance-ratio: UAE Strawberry extract / Macerated Strawberry extract = 5.

Conclusions

The presented pilot plant has been developed for extraction intensification where combination of high-shear mixing, ultrasound processing and screw pressing takes place. The patented ultrasound device also allows the prolongation of ultrasound time for extracted solids. The Authors wish to thank Business Finland for financial support NATINREC project (1195/31/2019).

References