Simmondsin concentrate from defatted jojoba meal

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1. Introduction

Jojoba, Simmondsia chinensis L. Schneider is native to the desert southwestern United States and adjoining northern Mexico. It is currently

Abstract

A water-extract of defatted jojoba meal was filtered and concentrated from 2.6 to 23% solids on a pilot scale in a reverse osmosis concentration apparatus and then freeze-dried. The characteristics of the membrane and new concentrator were determined with both glucose and the water-extract from jojoba meal. Permeate flux was not significantly affected by the change in total flow within the controllable limits at 1, 2, 10, or 20% glucose concentration. With the total flow past the membrane maintained at 57 l/min, the permeate flux was measured at various concentrations and at different pressures across the membrane. A permeate flux rate of $2.21 \times 10^{-5}$ m/s could be maintained for glucose concentrations up to 20%. For the extract, pressure across the membrane was adjusted to maintain a permeate flux of $1.24 \times 10^{-5}$ m/s (3.8 l/min for a 5.1 m$^2$ surface area membrane) not exceeding the system limit of 6.9 MPa. Using this method, 193 l of 2.6% solids jojoba extract could be concentrated to 25.3 l of 19.7% solids in 45 min. Permeate flux decreased with time because maximum pressure could not maintain a flux rate of $1.24 \times 10^{-5}$ m/s at the higher solids concentrations. The average permeate flux over the entire experiment was $0.99 \times 10^{-5}$ m/s. Based on the pilot scale tests, 568 kg of meal were extracted and processed on industrial equipment. The process included a vacuum drum dryer coated with diatomaceous earth with spray-drying of the concentrate. A powdery solid containing 42% simmondsin and related analogues was obtained. The results of the industrial trials and recommendations for process improvements are discussed. © 1997 Elsevier Science B.V.

Keywords: Simmondsia chinensis L. Schneider; Simmondsin; Jojoba; Membrane
being grown on 3000–5000 ha in the US and elsewhere worldwide. Jojoba has a unique wax ester oil which is 50–60% of its seed weight. Jojoba oil has good markets in cosmetics and lubricants. The solvent-extracted seed meal is not used as much as the oil although it contains about 25% crude protein. The defatted meal also contains sugars and 11–15% of a unique group of natural products, all structurally related to simmondsin. Cokeleaere has shown that simmondsin is an effective hunger satiating agent for obtaining an ideal growth rate in chickens (Cokeleaere et al., 1995). Jojoba meal has been used for the simmondsin's effect of regulating food intake of animals. In addition, the meal also contains other antinutritional factors such as trypsin inhibitor, polyphenols, bitter taste, nonnutritive protein and indigestible jojoba oil.

Preparative silica gel column methods have been proposed for solvent-based extraction and isolation of simmondsin on a kilogram scale (Van Boven et al., 1993). Membrane separation processes for isolating both the protein and the low molecular weight components have been reported (Abbott et al., 1991, 1996; Nabetani et al., 1995). Jojoba meal defatted by hexane extraction has little or no soluble protein because the protein is denatured during the recovery of hexane by a steaming process. The purpose of this research was to scale-up the water extraction of defatted meal and enrichment of simmondsin and its analogues in a simmondsin concentrate. For large-scale recovery of simmondsin concentrate, the extract must be dewatered and dried by the most energy efficient means without damaging the product.

Reverse osmosis concentration systems are currently being used to remove water from maple syrup, to concentrate fruit juices and to recover sugar, salts or detergent from various process waste streams. The advantages of this system are that they are energy efficient, operate at ambient temperature and do not degrade low molecular weight salts and sugars. Spray dryers are used for the final drying of aqueous extracts, but they operate best when the feed solution concentration is between 10 and 20% for optimum product quality. Thus, reverse osmosis can be used to concentrate the dilute extract for spray-drying.

2. Materials and methods

2.1. Materials

Defatted jojoba meal was provided by Purcell Natural Jojoba (Avila Beach, CA). Diatomaceous earth was Celaton Diatomite, FW 20 particle size, from Eagle-Picher Minerals (Reno, NV). Glucose was dextrose from Difco Laboratories (Detroit, MI).

2.2. Equipment and procedures

2.2.1. Laboratory tests

Slurries (200 ml) of ground jojoba meal at 6, 10, 15 and 20% in water were filtered through filter paper (Whatman No. 1, Maidstone, England) or filter paper covered with filter aid on a Buchner funnel under vacuum and washed once with water (100 ml). The same diatomaceous earth used in the industrial trials (See Section 2.1) was tested as a filter aid at 1.5 and 3 cm thickness on top of the filter paper. In addition, a 1.5 cm diatomaceous earth layer was tested with a 1.5 cm top layer of equal parts of the jojoba slurry and diatomaceous earth. The time to filter through each filter layer, the volume recovered and the concentration of the filtrate were measured for 200 ml of slurry.

2.2.2. Pilot plant scale (250 l) tests

Initial pilot plant studies were performed as described previously (Nabetani et al., 1995) except that the extraction water was not adjusted to pH 8 and the microfiltration permeate was concentrated by reverse osmosis to 23% solids and then freeze-dried. The reverse osmosis concentration apparatus (ROC) is a Seprotech (Seprotech Systems, Ottawa, Ontario, Canada) model built to our specifications. It has a 10 cm dia x 102 cm long spiral-wound, composite polyamide membrane element (Hydranautics 4040-HSA-SWC1) with 5.1 m² of surface area and rated for a maximum cross membrane pressure of 6.9 MPa (Fig. 1). Other important features include a high pressure, constant output (19 l/min) pump, a feed pump, a continuous circulation pump and a clean-in-place tank and system. A pressure-sensi-
Concentrate bypass line if temperature is too high for membrane.

* Not all details such as pressure and temperature gauges are shown.

Fig. 1. Schematic diagram of the reverse osmosis concentrator (ROC).

The ROC membrane was cleaned by rinsing with hot water (40–45°C), then rinsing with a pH 9 detergent solution (0.52% Ultrasil 56, Ecolab, St. Paul, MN) and with water again.

2.2.3. Industrial trial (568 kg jojoba meal)
Jojoba meal (568 kg, 4.9% moisture) was ground until 95% passed through a 20 mesh screen. Five batches of 114 kg meal were mixed with 1185 l water and then filtered through a 60 mesh screen (Fig. 2). The combined solids retained on the screen were washed again with 2375 l water. Both water extracts were combined and filtered through a rotary drum vacuum filter coated with 5 cm of wet diatomaceous earth. The clear filtrate was adjusted to pH 7 and concentrated in the Seprotech ROC. A 17% solids solution from the ROC was fed to a spray-dryer with an inlet temperature of 228°C and outlet temperature of 76°C, drying at 19 l/h.

In a second industrial trial, four batches of 114 kg of ground jojoba meal were mixed with 756 l water and then filtered through 5 cm diatomaceous earth on a rotating drum vacuum filtration apparatus. Water used in layering the diatomaceous earth on the filter was discarded as clear filtrate until the colored filtrate from filtering jojoba meal extract appeared. The jojoba extract was concentrated to 14% in the same ROC as described previously and then spray-dried.

Fig. 2. Flow diagram of the industrial extraction of simmondsin concentrate.
2.2.4. Analytical tests

Solids content was followed by refractive index using a hand held Refractometer (Atago Co., Ltd., Model 500, 0–90%, Japan) and calibrated against oven drying and moisture balance methods.

For simmondsin and its analogue analysis, samples of jojoba meal (10 mg) or aliquots of sample solutions containing 1 mg of simmondsin and analogues were mixed with deionized, organic-free water (10 ml) and sonified three times for 90 s. The samples were centrifuged and the supernatant decanted into a Teflon-lined capped vial. Autoinjection vials were filled with 1 ml of the supernatant that had been filtered through a 0.45 μm micro porous filter. Extracts should be kept out of direct sunlight because simmondsin ferulate is isomerized to a mixture of cis and trans isomers by UV exposure (Van Boven et al., 1996). Samples (0.5 ml) were injected through a 20 μl loop injection port and a 20 μl sample automatically injected onto a 4.6 mm x 10 cm Econosil C-18 3U reversed phase column (ALTECH Associates, Deerfield, Illinois). Solvent flow was programmed from 5 to 100% methanol in an aqueous mix over 10 min at a flow rate of 0.75 ml/min, then held at 100% methanol for 15 min. The HPLC system consisted of a Spectra Physics SP 8800 ternary pump (Thermo Separation Products, Schaumburg, IL), the loop, column and a Isco UV detector (Isco, Lincoln, NE) set at 220 nm and a 10 mm pathlength flow cell. Analysis time per sample, including re-equilibration, was typically 45 min. Retention time of simmondsin was 8.9 min, demethylsimmondsin 3.9 min, didemethylsimmondsin 2.9 min and simmondsin ferulate 14 min. Peak areas were compared to a calibration curve made from authentic samples and concentrations in the original sample were calculated.

3. Results and discussion

3.1. Laboratory tests

The results of the filtration trials are listed in Table 1. The goal was to recover the maximum amount of soluble solids at a reasonable filtration rate. The filter paper alone clogged quickly and the filtration became too slow to measure for 15 and 20% slurries. The 20% slurry was too slow to filter regardless of filter aid. The 1.5 cm thick diatomaceous earth and the combination of 1.5 cm diatomaceous earth with an additional 1.5 cm layer of equal mixtures of slurry and diatomaceous earth filtered well with the 15% slurry. The highest recovery of soluble solids (31.1% of dry meal weight) at an overall concentration of 2.4% for the combined washes was obtained with a 1.5 cm layer of filter aid. Significant loss of soluble extract with little reduction in the solids load on the vacuum filter was encountered when the slurry was sieved before vacuum filtration (data not shown). The sieving step was eliminated in the second industrial trial to test whether the laboratory result could be applied on an industrial scale. Foaming was a significant problem in the second industrial (Table 1). Based on those experiments, at least 0.7% defoaming agent was needed even when the pH was adjusted to 7.0 in the extraction.

3.2. Pilot scale tests

The first experiment with 1% glucose involved a batchwise concentration to 23% by discarding the permeate and recycling the retentate, and maintaining the temperature at 23°C. The decrease in flux rate with sugar concentration and time is shown in Fig. 3. After a concentration of 25.3%
<table>
<thead>
<tr>
<th>Slurry (%)</th>
<th>Parameter measured</th>
<th>Filtration media</th>
<th>Filter paper</th>
<th>1.5 cm</th>
<th>1.5 cm plus DE in slurry</th>
<th>3 cm</th>
<th>3 cm plus DE in slurry</th>
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<td>Slow &gt; 8 h</td>
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<td>60 s</td>
<td>10 min</td>
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<td>6</td>
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<td></td>
<td>1.2%</td>
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<td>6</td>
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<td>275 ml</td>
<td>265 ml</td>
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glucose is reached, the ROC system shuts down because the limit of pressure on the membrane of 6.4 MPa is reached.

The second series of experiments started with 1% glucose. Permeate was discarded to increase to set concentrations and then recirculated. At each set concentration the pressure across the membrane or flow rate past the membrane was varied. A unique feature of the ROC is a separate in-line recirculating pump (Designated R.P. in Fig. 1) which maintains a constant flow into the membrane regardless of feed pump or pressure pump flow rates. Pressure pump flow rate is constant at 19 l/min and the recirculating pump can be varied between 19 and 42 l/min. The total flow past the membrane was varied by adjusting recirculation pump rate without changing the pressure across the membrane (by restricting the retentate flow from the membrane). Total flow was varied between 45 and 61 l/min. Permeate flux was not significantly affected by the change in total flow within the controllable limits at 1, 2, 10, or 20% glucose concentration. With a constant total flow past the membrane of 57 l/min, the pressure across the membrane was varied and the permeate flux measured at various concentrations (Fig. 4). We conclude that a permeate flux rate of $2.21 \times 10^{-5}$ m/s could be maintained for concentrations up to 20% (e.g. 6.7 l/min permeate flow rate at 20% glucose concentration, and $\Delta P = 5.52$ MPa across the membrane) (Fig. 4). Membrane rejection of glucose was measured as the glucose was concentrated batchwise from 6 to 12%. In the permeate 0.0876% glucose was measured which represents a 99.9% rejection rate for glucose.

The aqueous jojoba meal extract, after microfiltration, (193 l, 2.6% solids, pH 4.75) was processed in a continuous batchwise concentration to 19.7% solids at 25°C. Pressure was adjusted to maintain a permeate flux of $1.24 \times 10^{-5}$ m/s. Fig. 5 shows the effect of changing pressure on permeate flux ($J_v$) at constant glucose concentration.

Fig. 4. The effect of changing pressure on permeate flux ($J_v$) at constant glucose concentration.
3.3. Industrial scale tests

3.3.1. First trial

The solid content in the extract was expected to be 2.6%, but the water added (2000 L) to layer a 5 cm thick coating of diatomaceous earth on the vacuum filter changed this value. Thus, 10,300 L of water were added to extract the meal and layer the clay resulting in 8700 L of 1.1% solids in the clear filtrate to be concentrated on the ROC before the spray-drying. Possible explanations for obtaining such a dilute solution are the added water from layering the clay, a more complete recovery of extract water in the filtration process compared to the pilot process, less extractable solids in the meal used in the industrial trial, and losses from foaming before the pH was adjusted. The ROC was used to concentrate 6700 L from 1.1% to 14 ± 3% in 245 L batches. Initially, the membrane had to be cleaned after every second batch. This problem was traced to insufficient flow to the feed pump using gravity feed. A transfer pump was placed in-line before the ROC feed pump and 9 batches could be run between membrane cleanings. Membrane cleaning took about 2 h. The permeate flow rate was 8.5 × 10^{-6} m/s during these nine batches.

The concentrate was spray-dried to produce 40 kg of tan to white flowable powder. The powder had 2.4% water, 13.9% simmondsin, 5.5% simmondsin ferulate, 5.9% demethylsimmondsin and 16.0% didemethylsimmondsin. Significant losses were encountered from the spray-dryer collection system. Not all of the extract was concentrated and 38 L of reverse osmosis concentrate (17% solids) was not spray-dried because of time constraints. The net yield of 40 kg of concentrate from 568 kg meal is not a realistic estimation of the expected yield from a properly sized, continuous operation. The 6700 L of 1.1% solids processed in the ROC contained 73.7 kg of solids and the total water-soluble solids before filtration was 113.3 kg or 20% of the starting material. Based on the 73.7 kg in the extraction solution that was processed and the 7 kg in concentrate from the ROC that were not spray-dried, 64% of possible extract was recovered as solid for this operation.

3.3.2. Second industrial trial

Based on laboratory experiments completed between the two industrial trials, the concentration of initial slurry was increased to 15% and screening of the slurry (before vacuum filtration) was eliminated. However, the diatomaceous earth thickness could not be decreased from 5 cm because of potential damage to the cloth filter on an industrial scale apparatus. After initial filtration on the rotating vacuum drum filter, a foaming
problem that had not occurred in the first trial was encountered. Approximately 50% of the total extractables was pulled through the vacuum system as foam and into the drainage system before the operation was finished. Foam was being removed at 19 l/min. The foam had a high (80%) liquid content when it was set aside and the foam dissipated. If the system was physically rearranged with the vacuum pump and trap higher and foam collected in a settling tank, about 53% of the extract could have been recovered. Alternative solutions such as a foam breaking device or defoaming agent could be tried. However, the foaming was not anticipated, and in the second industrial trial, the trap after the vacuum pump was too close to the floor drain to collect the foam into a settling tank.

The initial filtrate through the diatomaceous earth had a 2.93% calculated concentration and measured 3.0% by refractometry. Water (1140 l) was combined with the 760 l of slurry remaining in the reservoir of the rotating vacuum filtration apparatus as a wash of the filtration system. This produced an additional 1140 l containing 1.2% solids. The reservoir had a minimum volume of about 760 l, which prevented the filter aid from drying out and falling off. Combined filtrates totaled 2280 l containing approximately 2% solids and some foam. In the ROC, 2025 l were concentrated to 288 l (14% solids) and spray-dried to give 22 kg solids. Approximately 2 kg (dry basis) of solids were washed from the walls of the spray-dryer, which means that 16.3 kg were lost as fines. Accordingly, a filter bag at the spray-dryer outlet would be beneficial to recover the fines.

3.4. Energy usage

The energy efficiency of removing water by the ROC was measured by comparing electrical energy usage for the ROC to the energy needed to evaporate the same amount of water. The three motors driving the system consumed 14 A at 208 V which converts to 186 kJ to remove 6.7 l of water from the glucose solutions in 1 min, independent of glucose concentration between 2 and 20% concentration. The equivalent amount of energy to vaporize 6.7 l of water would be 15 131 kJ. Identical values were observed during the concentration of the simmondsin concentrate.

Spray-drying under the conditions of our tests required 466 kJ to remove 6.7 l of water in 1 min. Thus, to minimize overall energy usage, the extract should be as concentrated as possible, consistent with maximum recovery of water-soluble solids. Reverse osmosis should proceed to 15 to 17% before spray-drying. In addition to the energy savings from using the ROC rather than other means of water removal, the permeate from the ROC can be recycled into the extraction process, providing a clean source of extraction water.

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References


