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Effect of different process parameters and ultrasonic treatment during solid osmotic dehydration of jasmine for extraction of flavoured syrup on the mass transfer kinetics and quality attributes

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ABSTRACT

This study investigates the effects of different process parameters (sugar dose, sugar size and ultrasonic treatment time) on the solid osmotic dehydration (SOD) process of jasmine flowers and the physiochemical characteristics of syrup. The kinetics of moisture loss, solute gain and effective diffusivity during SOD were predicted by fitting the experimental data with the Azuara model, Weibull model and Fick second law model. The results showed that the high regression coefficient ($R^2 > 0.9$) and low χ^2 value represented the suitability of the Azuara model for predicting equilibrium water loss and solid gain and the Weibull model for predicting both moisture and solute fraction in jasmine under different process parameters during SOD. A relatively high sugar dose (120% w/w, fresh jasmine flower basis) and moderate sugar size (6-20 mesh) enhanced the moisture loss and solute uptake during SOD and produced more syrup. Furthermore, the high sugar dose (120% w/w) and small sugar size (40 mesh) enhanced the diffusion of polyphenols and flavonoids in jasmine to syrup. The mass transfer rate and syrup yield were increased, and more polyphenols, flavonoids and antioxidant components diffused to the syrup after applying the ultrasonic waves during SOD. Most flavour compounds of jasmine were effectively extracted by SOD under different process parameters, and alcohols (more than 68%) are the main flavour components of syrups, but the amount of major volatile compounds extracted was not affected by SOD process parameters.

Keywords Solid osmotic dehydration · Mass transfer · Ultrasound · Flavored syrup · volatiles

Introduction

With the increasing health awareness of consumers, flavour syrups with typical aromatic compounds and active substances from fruit or flowers are increasingly popular, and are generally used as flavour or functional sweets for beverages, desserts, and catering, such as tea with milk, flavour fruit juice and cakes, etc. in China (Milani and Koocheki 2011; Urbanus et al. 2014). Currently, flavour syrups are mainly produced by thermal concentration technology, in which sugar cane clear juice is heated and concentrated while flavour fruits or substances such as Chinese date, wolfberries, flowers or their aromatic compounds and active substances are added during the concentration process (Alarcón et al. 2020; Mohammed et al. 2021). However, the nutritional and aromatic compounds of the flavour syrups are mostly heat sensitive and are usually damaged during heating, so the nutritional value of the finished flavored syrups is reduced (Letaief et al. 2021). It is of great importance to develop nonthermal preparation methods for flavour syrups with a high concentration of flavour and active substances.

Solid osmotic dehydration (SOD) is a process that partially removes water from food by mixing it with solid sugar or salt and is usually used as a storage technique for foods with a high water content, such as sausages, salami, hotdogs, and cured ham/meat (Blankenship et al. 2018). The working principle of SOD is similar to that of osmotic dehydration (OD) (Wang et al. 2021), which is used as a predrying process for fresh fruits or vegetables, where a hypertonic solution is used to remove partial water from the fresh fruits or vegetables (Ciurzyńska et al. 2016). To date, SOD is also used to prepare flavour syrups (Ramalingam et al. 2021) because of its minimized flavour and active substance loss. This process involves two countercurrents flowing across the semipermeable cell membrane: (i) infusion of sugar into the interior of cellular tissue of the food and (ii) diffusion of water and water-soluble substances (sugars, organic acids, minerals, vitamins) of the cellular tissue to the outside of the tissue, and dissolving the surrounding solid sugars, forming a syrup with many natural nutrients, and aroma compounds from fruits or vegetables (Fernández et al. 2020).

The diffusion rates of water and osmotic solutes of SOD are the key factors that dominate the syrup quality and yield, which mostly depend on the tissue properties (size, shape, porosity, maturity level, composition, etc.) of fruit or vegetable, the particle size and dose of the osmotic medium, and the temperature and of duration of dehydration (Ciurzyńska et al. 2016). Previous studies have shown that several methods, including high hydrostatic pressure (Luo et al. 2018a), high electrical field pulses (Amami et al. 2008), and ultrasound (Geow et al. 2020), could accelerate mass transfer during the osmotic process.

Ultrasound is an energy form that generates mechanical agitation and acoustic cavitation, thus creating microscopic pores and microchannels on the surface of the food samples to be processed, and enhancing the mass transfer of OD (Nowacka et al. 2021). Understanding the mass transfer kinetics during SOD with various process parameters is a crucial factor for improving the flavour syrup quality and yield. Jasmine is highly valued for its distinctive aroma, and is used to produce various foods because of its distinctive aroma and high annual output in China (Issa et al. 2020). However, there are no studies on the mass transfer kinetics of SOD, and the effects of the process parameters on the preservation of aroma compounds and nutrition of jasmine.

The objectives of the study were to (1) study the effects of different technological parameters (sugar dose, sugar size and ultrasonic treatment time) on the mass transfer kinetics of jasmine flowers during the SOD process. (2) study the mass transfer kinetics by using the Azuara's model and the Weibull frequency distribution model to predict moisture content and solute content during jasmine SOD. (3) determine the moisture and solute diffusivity by applying the Fick's second law. (4) study the effects of different technological parameters on the physical properties, nutrition and volatile substances of extracted syrup.

Materials and method

Raw materials and reagents

Fresh jasmine flowers and brown rock sugar were purchased from local markets in Nanning, China. The flowers were selected according to the criteria of same size and no browning, and they were cleaned and refrigerated at −18℃. Before processing, the flowers were removed from the refrigerator, and then placed at room temperature until their temperature equilibrated. The brown rock sugar was sieved by different sieves to collect different sizes of sugar (6, 6-20 and 20-40 mesh).

Gallic acid standard, rutin standard, 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) were purchased from Sigma (Sigma Corporation, Japan). All other chemicals were of analytical grade and were purchased from Tianjin Kemiou Chemical Reagent Co. (Tianjin, China)

Solid osmotic dehydration experiment

The SOD experiments were carried out as shown in Fig. 1 in a refrigerated 5±2 °C warehouse. The effects of sugar dose, sugar size and ultrasonic treatment time on mass transfer, syrup quality and sugar yield were considered. The sugar doses were 40% (SOD 40), 80% (SOD 80) and 120% (SOD 120) (w/w, fresh jasmine flower basis). The sugar sizes were 6 mesh (SODS 6), 6-20 mesh (SODS 20) and 20-40 mesh (SODS 40). Jasmine flowers were mixed with sugar using the layer-layer method, i.e., a layer of flowers was placed at the bottom of the container, then a layer of sugar was placed on the flowers, then a layer of flowers was added again, and so on; flowers and sugar were placed in alternating layers, with the top layer being sugar (20% w/w, sugar dose basis) (Ramalingam et al. 2021). To estimate the mass transfer parameters and amount of solid gain and water loss during 168 h of SOD, every 24 h, the dehydrated jasmine flowers of each group were removed, rinsed with distilled water to eliminate the adhering syrup and undissolved sugar on the flower surface, blotted with tissue paper and weighed gently (Prithani and Dash 2020). All experiments were performed in triplicate.

Design for SOD of jasmine treated with ultrasound (USOD)

The effects of ultrasound on the mass transfer kinetics, syrup quality and yield of SOD jasmine were investigated. During SOD, samples were treated in an ultrasound cleaner bath (KQ-500DE, China) three times, i.e., after 48, 60 or 84 h for 20, 30 and 40 min, with total treatment times of 60 min (USOD 60), 90 min (USOD 90) and 120 min (USOD 120). To maintain the temperature of the samples at 5±2 °C, an ice bath was used. The ultrasonic frequency was 40 kHz, the power was 500 W, the sugar dose was 120% (w/w, fresh jasmine flowers basis) and the sugar size was 6-20 mesh (Fig. 1). During this process, flowers were removed from the syrup every 24 h and weighed to calculate the water loss and solid gain. To evaluate the effect of ultrasound, the same procedure was repeated without ultrasound (USOD0). All flower samples were dried in an oven (101-2A, Shanghai) at 105 °C until achieving a constant weight to determine the moisture content (Song et al. 2020). Each experiment was carried out in triplicate, and the data are presented as the average of triplicate samples.

Water loss (WL) and solid gain (SG) calculations

Water loss (g/100 g) can be expressed as the net loss of water from the fresh samples after osmotic treatment and was estimated by Eq. (1).

$$\mathrm{WL} = \frac{\mathrm{W_0} \times \mathrm{X_0} - \mathrm{W_t} \times \mathrm{X_t}}{\mathrm{W_0}} \tag{1}$$

Solid gain (g/ 100 g) is the amount of solute uptake of the sample from the osmotic solution during dehydration after a time t and was determined by Eq. (2).

$$\mathrm{SG} = \frac{\mathrm{W_t} \times \mathrm{S_t} - \mathrm{W_0} \times \mathrm{S_0}}{\mathrm{W_0}} \tag{2}$$

where 'W′ is the weight of jasmine (g), 'X′ is the moisture content of jasmine on wet basis (g/ 100 g) and 'S′ is the solid content (g/ 100 g). Subscripts of 0 and t indicate the initial value and the value at time t, respectively (Luo et al. 2018b).

Mass transfer models

Azuara's model

The two-parameter model developed by Azuara (2010) can be applied to the initial part of the data obtained over relatively short time intervals during SOD. The initial part of the dehydration curve can be used to predict water loss and solid gain in the equilibrium state. The proposed model for WL and SG during SOD is presented in Eqs. (3) and (4), respectively.

$$\mathrm{WL} = \frac{\mathrm{S_w} \cdot \mathrm{t} \cdot \mathrm{WL_\infty}}{1 + \mathrm{S_w} \cdot \mathrm{t}} \tag{3}$$

$$\mathrm{SG} = \frac{\mathrm{S_s} \cdot \mathrm{t} \cdot \mathrm{SG_\infty}}{1 + \mathrm{S_s} \cdot \mathrm{t}} \tag{4}$$

where S_w and S_s are the model constants. The linearization of Eqs. (3) and (4) for WL and SG leads to Eqs. (5) and (6), respectively.

$$\frac{\mathrm{t}}{\mathrm{WL}} = \frac{1}{\mathrm{S_w} \cdot \mathrm{WL_\infty}} + \frac{\mathrm{t}}{\mathrm{WL_\infty}} \tag{5}$$

$$\frac{\mathrm{t}}{\mathrm{SG}} = \frac{1}{\mathrm{S_s} \cdot \mathrm{SG_\infty}} + \frac{\mathrm{t}}{\mathrm{SG_\infty}} \tag{6}$$

where the equilibrium WL (WL_∞) and SG (SG_∞) can be calculated from the plots of t/WL and t/SG versus t, respectively.

Weibull distribution mode

The Weibull frequency distribution model was implemented to predict the moisture and solute contents of jasmine flowers. The fractional amount of WL and SG during SOD can be expressed in Eqs. (7) and (8), respectively (Sharma and Dash 2019; Dash et al. 2019).

$$1 - \frac{\mathrm{WL}}{\mathrm{WL_\infty}} = \mathrm{MR} = \exp\left[\left(\frac{\mathrm{t}}{\alpha_\mathrm{w}}\right)^{\beta_\mathrm{w}}\right] \tag{7}$$

$$1-\frac{SG}{SG_{\infty}}=SR=\exp\left[\left(\frac{t}{\alpha_s}\right)^{\beta_s}\right] \quad (8)$$

where MR and SR are the moisture and solute ratios, respectively; α_w and α_s are the scale parameters of the Weibull model and are associated with the process rate (the time required for WL/WL_{∞} or SG/SG_{∞} to reach the value $1\text{-}e^{-1}$); β_w and β_s are the shape parameters (dimensionless) for moisture and solute, respectively; and t is the time (h).

Determination of moisture and solute diffusivity

Based on Fick's second law, Crank (1975) proposed a group of analytical solutions for diffusion in different geometries with several initial and boundary conditions: (a) the moisture transfer is predominantly one dimensional; (b) the initial moisture content is distributed uniformly in the product; (c) a negligible shrinkage; and (d) a constant diffusion coefficient during SOD and a negligible resistance to mass transfer at the product surface. Hence, the effective diffusivity was calculated according to Eqs. (9) and (10).

$$MR=\frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left[-(2n+1)^2\frac{\pi^2 D_w t}{4L^2}\right] \quad (9)$$

$$SR=\frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left[-(2n+1)^2\frac{\pi^2 D_s t}{4L^2}\right] \quad (10)$$

where D_w and D_s are the effective diffusivities of water and solute (m^2/s), respectively, and L is the half-thickness of the slab in flowers (m). For long osmotic periods, Eqs. (9) and (10) can be further simplified to only the first term of the series (Lemus-Mondaca et al. 2018):

$$MR = \frac{8}{\pi^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_w t}{4L^2}\right] \quad (11)$$

$$SR = \frac{8}{\pi^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_s t}{4L^2}\right] \quad (12)$$

$$\ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \frac{\pi^2 D_w t}{4L^2} \quad (13)$$

$$\ln(SR) = \ln\left(\frac{8}{\pi^2}\right) - \frac{\pi^2 D_s t}{4L^2} \quad (14)$$

The water and solute diffusion coefficients were calculated from Eqs. (15) and (16). The values of slope were obtained using the trinomial regression of ln(MR) and ln(SR) against time.

$$slope = \pi^2 \frac{D_w}{4L^2} \quad (15)$$

$$slope = \pi^2 \frac{D_s}{4L^2} \quad (16)$$

Physical analysis (pH, total soluble solids, dry weight and syrup yield)

The pH values of the syrup were recorded according to the protocol adopted by Lee et al. (2018), using a pH meter (PHS–3C, Shanghai) at room temperature. The dry weight was determined by drying the syrups in a vacuum dryer (DZF-6034, Shanghai) for 24 h at 70 °C and 100 kPa and expressed as a percentage. A digital refractometer (WYA-2S, China) was employed to determine the total soluble solids content of the syrup (Turkiewicz et al. 2020).

The amount of syrup was calucalted as the weight (g) of the syrup formed by SOD, which was filtered by a gauze filter (200 mesh) and then weighed when the SOD process reached equilibrium. The jasmine flower weight used in the experiments was 200 g, and the equilibrium times of SOD 40 and SOD80 were 96 h and 120 h, respectively, and that of the other samples was 144 h based on previous work.

Antioxidant capacity determined by DPPH radical scavenging

DPPH radical scavenging capacity was measured according to the protocol of Goslinski et al. (2021). A total of 0.1 mL of the syrup solution (8 g·mL^{-1}) was added to 2.9 mL of 0.1 mmol L^{-1} DPPH methanol solution, mixed and left in the dark for 30 min to incubate at room temperature. The absorbance of the syrup samples was recorded at 517 nm with a UV-spectrophotometer (UV-1800, Shanghai, China), and methanol was used as a blank. The results were expressed as Trolox concentration (mg Tx·kg^{-1} dw), using a calibration curve of Trolox, with a linearity range from 0 mg/mL to 0.4 mg/mL (R^2 greater than 0.999). The antioxidant capacity was expressed as milligrams of Trolox equivalents per kg of a syrup sample (mg Tx·kg^{-1} dw).

$$\mathrm{DPPH}(\%) = \left[\frac{(A_{DPPH} - A_{syrup})}{A_{DPPH}}\right] \times 100 \qquad (17)$$

where A_{DPPH} is the absorbance of the DPPH blank solution and A_{syrup} is the absorbance of the sample solution.

Total polyphenol content (TPC)

The total polyphenol content (TPC) was evaluated according to Cheng et al. (2014) with slight modifications. A total of 1 mL of syrup solution (5 mg·mL^{-1}) was added to 1 mL of Folin-Ciocalteu reagent, and the mixture was kept at room temperature for 5 min. Then, 5 mL of sodium carbonate (1 M) was added to the mixture, and the mixture was gently homogenized. The total volume of the mixture was adjusted to 10 mL with distilled water. After the mixture was kept at room temperature for 1 h, the absorbance was read at 760 nm using a UV spectrophotometer (UV-1800, Shanghai, China). Distilled water was used as a blank. The results are presented in mg GAE·kg^{-1} dw.

Total flavonoid content (TFC)

The total flavonoid content was evaluated according to Wang et al. (2015). One millilitre of syrup solution (5 mg·mL^{-1}) was placed in a 10 mL volumetric flask, 0.4 mL of 5% sodium nitrite solution was added, and 0.4 mL of 10% aluminium nitrate was added 6 min later. After 6 min, 4 mL of 4% sodium hydroxide was added, and the total was brought to 10 mL with methanol. The absorbance was evaluated at 510 nm using a UV spectrophotometer (UV-1800, Shanghai, China). The obtained results were expressed in mg equivalents of rutin (mg RU·kg^{-1} dw).

Volatile flavour compound analysis

The flavour components of syrups and jasmine flowers were extracted using the headspace solid-phase microextraction (HS-SPME) technique and analysed using gas chromatography-mass spectrophotometry (GC–MS) (Issa et al. 2020; Rodriguez-Flores et al. 2021), detailed procedures are outlined in the Supplementary Materials.

Scanning electron microscopy (SEM)

Microscopic observation of vacuum-freeze-dried control (USOD0) and ultrasonically treated SOD jasmine petals was carried out by coating the jasmine petals with a thin layer of gold in a sputter coater. Scanning electron microscopy (SEM) images were then obtained using a scanning electron microscope (Phenom Pro, Phenom, Holland) at 5 kV and examined at 2000× magnification for surface analysis (Liu et al. 2020).

Statistical analysis

The experiments were all performed at least 3 times. Statistical analysis of experimental data was conducted using SPSS statistical software (SPSS 20.0) applying the analysis of variance to estimate statistically significant differences for a confidence level of 95% ($p < 0.05$). The determination coefficient (R^2), chi-square (χ^2), and root mean square error (RMSE) were considered the primary criteria to determine the suitability and goodness of fit in the model (Sharma and Dash 2019; Dash et al. 2019). Relatively high R^2 values and low χ^2 and RMSE values represented the best fitting of the mathematical model to predict the behaviour during SOD. The R^2, χ^2 and RMSE vales were evaluated by applying Eqs. (18)-(20).

$$R^2 = 1 - \sum_{i=1}^{n} \frac{\left(Y_{pred,i} - Y_{exp,i}\right)^2}{\left(Y_{pred,i} - \overline{Y_{exp,i}}\right)^2} \tag{18}$$

$$\chi^2 = \sum_{i=1}^{n} \frac{\left(Y_{pred,i} - Y_{exp,i}\right)^2}{Y_{pred,i}} \tag{19}$$

$$RMSE = \left[\frac{1}{n}\sum_{i=1}^{n}\left(Y_{pred,i} - Y_{exp,i}\right)^2\right]^{\frac{1}{2}} \tag{20}$$

Results and discussions

Effect of different processing parameters on WL and SG in jasmines

The experimental results of SOD kinetics in terms of WL and SG over time using various sugar doses, sugar sizes and ultrasonic treatment times are shown in Fig. 2. The results showed that there was a rapid increase in WL and SG during the first 0–24 h, followed by a progressive reduction (24-120 h), and finally, equilibrium was reached after 120 h of SOD. The rapid WL and SG in the beginning was due to the large osmotic driving force between the jasmine flower and the surrounding hypertonic medium. In the SOD process, the chemical potential gradient of water was the main driving force for mass transfer. The decrease in WL and SG rates in the later period was due to a reduction in osmotic driving potentials for moisture and solute transfer caused by sugar dissolution (Dash et al. 2019). Furthermore, the rapid WL and SG near the surface layers of the flower tissue in the beginning of the SOD process resulted in structural changes in the flower; that is, the compaction of the flower surface layers increased, so the mass transfer resistance of water and solids increased, causing a gradual reduction in WL and SG (Prithani and Dash 2020).

The sugar dose and sugar size were found to have significant effects ($p < 0.05$) on WL and SG. The results showed that the WL and SG increased with increasing sugar dose. Comparable results were also reported by Etemadi et al. (2020) when working with apples in different concentrations of sucrose solution during OD. This observation is explained mainly by the relatively high sugar dose causing a high concentration gradients across the cell wall, which might have increased WL and SG (Zúñiga and Pedreschi 2011).

Too large or too small of a sugar size caused a decrease in the rates of WL and SG. The rates of WL and SG were both highest when the sugar size was 6-20 mesh, and after 168 h of SOD, the rates of WL and SG of SODS20 (36.74 g/100 g and 27.72 g/100 g) were greater than those of SODS6 (35.76 g/100 g and 27.11 g/100 g) and SODS40 (35.99 g/100 g and 26.35 g/100 g). This phenomenon could be attributed to the relatively small contact area between the large sugar size and the flowers, resulting in slow WL from the flowers and the slow dissolution of solid sugar, which led to a decrease in the rates of WL and SG. However, when the sugar particles were too small, the sugar layer structure was compact, and the channels between sugar particles were very small; therefore, the mass transfer resistance increased, and the rates of WL and SG decreased (Prithani and Dash 2020; Ramya and Jain 2017).

Higher WL and SG values were found for ultrasonic samples than for USOD0 samples, and the rates of WL and SG increased with the progression of ultrasonic treatment time. After 168 h of SOD, the WL and SG for the ultrasound samples varied in the ranges of 36.73-40.62 g/100 g and 19.71-20.71 g/100 g, respectively, which were 3.55–14.52% and 11.80-17.47% higher than those of USOD0 (35.47 g/100 g and 17.63 g/100 g). This phenomenon can be attributed to the formation of ultrasonic microchannels in the internal structure of the flower, in which the microchannel number might increase with increasing ultrasonic treatment time, which eases moisture and diffusion from the product surface (Sharma and Dash 2019). . Ultrasound treatment enhanced the mass transfer rate in many fruits and vegetables, such as kiwi fruits, garlic, and black jamun fruit (Prithani and Dash 2020; Alolga et al. 2021; Sharma and Dash 2019).

Modelling effect of different processing parameters on the Azuara's model parameters

The two-parameter model developed by Azuara (2010) was used to predict the kinetics of SOD and to determine WL and SG under equilibrium conditions. The coefficients of R^2

values, χ^2 and RMSE for WL and SG varied between 0.9675 and 0.9990, 0.0369-0.3843 and 0.1795 and 0.8820, respectively, suggesting that the Azuara model showed a good fit of the data and was suitable for predicting WL_∞ and SG_∞ for three process parameters. The values of the Azuara model parameters for WL (S_w and WL_∞) and SG (S_s and SG_∞) are presented in Tables 1 and 2, respectively. The relatively large values of WL_∞ and SG_∞ indicate that more flavour syrup was produced during SOD and can be used as a reference to determine preferable processing parameters in practical production to obtain higher syrup yields.

Sugar dose and sugar size were found to have significant effects ($p < 0.05$) on WL_∞ and SG_∞. Tables 1 and 2 show that with increasing sugar dose, WL_∞ and SG_∞ increased from 27.37 g/100 g to 40.12 g/100 g and 23.85 g/100 g to 32.56 g/100 g, respectively, indicating that increasing sugar dose (≤120% w/w) produced more syrup. A similar trend was also reported during the OD of apples in different concentrations of osmotic solution (Etemadi et al. 2020). This observation is explained mainly by the large osmotic pressure gradient across the interface of the flower and the sugar caused by increasing the sugar dose (Pei et al. 2019).

The WL_∞ and SG_∞ of SODS20 were 43.44 g/100 g and 33.53 g/100 g, respectively, higher than SODS6 (42.78 g/100 g and 32.54 g/100 g) and SODS40 (42.12 g/100 g and 32.08 g/100 g), indicating that a moderate sugar size (6-20 mesh) is beneficial to produce more syrup. Such differences in WL_∞ and SG_∞ were due to low water loss, and solute gain resulted in a small contact area resulting from larger sugar or higher transfer resistance because of the compact structure of small sugar particles (Landim et al. 2016).

Ultrasonication was found to have positive effects on WL_∞ and SG_∞ ($p<0.05$), and its enhancement effect on WL_∞ and SG_∞ increased with increasing ultrasound treatment time.

The WL_∞ and SG_∞ of ultrasound-treated samples were 2.35-8.50% and 7.17-11.01% higher, respectively, than those of USOD0 (45.37 g/100 g and 26.46 g/100 g), while the WL_∞ and SG_∞ of USOD120 were 49.23 g/100 g and 29.37 g/100 g, respectively, which were 8.50% and 11.01% higher than those of USOD0; these results indicated that USOD can produce more syrup, and the syrup yield increased with increasing ultrasonic treatment time. Similar findings were reported by Prithani and Dash (2020), who found that WL_∞ and SG_∞ values of kiwi samples pretreated with ultrasound were much higher than those of the samples not treated with ultrasound. The differences in the WL_∞ and SG_∞ between ultrasonic-treatment samples and the control samples were attributed to the fact that ultrasonic waves can weaken the cell adhesion and disrupt the cell structure, then create vacancies and cracks in the cell wall, thereby accelerating moisture and solute transfer (Kroehnke et al. 2021).

The S_w and S_s parameters established by the Azuara model were related to mass transfer rates. The relatively large values of S_w and S_s indicate increased mass transfer rates for WL and SG. It appears that S_w increased from 0.0459 to 0.0523 and 0.0253 to 0.0279 with increasing sugar dose and ultrasonic treatment time, respectively, indicating that the water transfer rate increased when the sugar dose (40-120%, w/w, fresh jasmine flower basis) and ultrasonic treatment time increased, which can shorten the SOD time and improve SOD efficiency. However, the variation in S_w was not consistent with increasing sugar size, and the S_w of SODS20 was the highest (0.0436) and was 15.34% and 4.06% higher than those of SODS6 and SODS40, respectively, which was consistent with WL_∞, indicating that a moderate sugar size is beneficial to water leaching out and to produce more finished syrup. The S_s values increased from 0.0153 to 0.0161 with increasing ultrasonic treatment time, suggesting that solute diffusion was accelerated by ultrasonication during jasmine SOD. However, S_s first increased and then decreased when the sugar dose and size increased, suggesting that solute diffusion is slower when the sugar dose is too low or too high and the sugar size is too large or small; therefore, to control solute diffusion and produce as much syrup as possible, the sugar dose and size must be optimized.

Modeling effect of different processing parameters on the Weibull model parameters

The Weibull model depends on WL_∞ and SG_∞ values to determine the moisture ratio and solid ratio (Sharma and Dash 2019) and exhibits a strong fit to describe the moisture loss during SOD, as indicated by the higher R^2 (0.9861-0.9995) with lower χ^2 (0.0008-0.0378) and RMSE (0.0056–0.0324) values. The scale parameter (α_w) defines the rate and represents the time needed to accomplish approximately one log cycle or 63% of the process. A lower α_w value indicates a lower time requirement for achieving equilibrium conditions in the dehydration process, and it can be used as a reference to determine the optimal extraction time during practical production to improve productivity. The shape parameter (β_w) is also known as the Weibull slope or the threshold parameter and is related to the mass transfer velocity at the beginning of the OD process; a lower β_w value represents faster mass transfer at the beginning of OD (Dash et al. 2019). The values of α_w and β_w of the Weibull model for WL during SOD under different processing parameters are listed in Table 3. As seen in Table 3, α_w and β_w were affected by sugar dose, sugar size and ultrasonic treatment time.

Both the α_w values and β_w values decreased as the sugar dose increased. Hence, the initial rate of WL was faster and required less time to achieve equilibrium conditions at a higher sugar dose. Similar results were obtained by Pei et al. (2019) during the OD of button mushrooms in different concentrations of sucrose solutions. Among the three sugar sizes, the α_w and β_w values of SODS20 were the lowest (33.74 h and 0.5390) followed by SODS6 (48.26 h and 0.5659) and SODS40 (42.93 h and 0.5750), indicating that the initial rate of WL was the fastest and lowest time requirement for achieving equilibrium when sugar size was 6-20 mesh. The α_w values decreased from 69.05 to 65.42 (h) with increasing ultrasonic treatment time, indicating a lower time requirement for achieving equilibrium conditions, and the SOD process was accelerated by ultrasonication. Similar findings were obtained during the osmotic dehydration of kiwi fruits (Prithani and Dash 2020). However, a gradual increase in β_w values was observed, indicating that ultrasonication has no positive effects on the WL rate at the beginning of the SOD process, which is mainly attributed to the ultrasonic treatment carried out after 48 h of SOD.

The scale (α_s) and shape (β_s) parameters of the Weibull model for solute uptake under different processing parameters are listed in Table 4. The fits were good, with R^2 values higher than 0.9783 and χ^2 and RMSE values ranging from 0.0024-0.0217 and 0.0122-0.0724, respectively. This result shows that α_s and β_s were affected by sugar dose, sugar size and ultrasonic treatment time. Along with increasing sugar dose, the α_s values increased from 30.18 to 50.79 (h), while the β_s values decreased from 0.6236 to 0.6043, indicating a lower time requirement for achieving equilibrium conditions at a lower sugar dose, while the initial rate of SG was faster at a higher sugar dose during the SOD process. The α_s value of SODS20 was the lowest (58.50 h), and the highest α_s value (86.05 h) was obtained for SODS6, indicating that a medium-size sugar (6-20 mesh) was beneficial to an efficient SOD process.

The β_s values show a decreasing trend (from 0.6923 to 0.5720) with decreasing sugar size, indicating that the initial rate of SG was faster at a smaller sugar size (40 mesh). With increasing ultrasonic treatment time, the α_s values decreased from 114.22 to 106.99 (h), revealing that the SOD process was accelerated by ultrasonication. However, the β_s values did not show the same changing rule as the α_s values, which indicated that ultrasonication has no effect on the initial rate of solid gain for the SOD process. Furthermore, the β_s values were higher than the β_w values, indicating that the rate of WL was faster than the rate of solid gain due to the semipermeable characteristics of the cell walls. Similar findings were reported in a study of the effects of high pressure on the osmotic dehydration kinetics of granny smith apples (Dash and Balasubramaniam 2018).

Moisture and solute diffusivity during solid osmotic dehydration

The calculated effective moisture and solute diffusivities for different process parameters of SOD are presented in Table 5. It was observed that with increasing sugar dose, D_w values were increased from 8.9005×10^{-15} to 9.8910×10^{-15} m^2/s, which seemed to suggest that the higher sugar dose had a faster moisture diffusion. This phenomenon could be due to increasing the osmotic pressure gradient, which improved the rate of mass transfer during SOD and changed the porosity and cell permeabilization (Koprivica et al. 2014). Similar results were observed by Semenoglou et al. (2020), who reported that the effective moisture diffusivities of sea bass fillets during OD for 40%, 50% and 60% glycerol solutions were found to be 1.9×10^{-9} m^2/s, 2.8×10^{-9} m^2/s and 3.6×10^{-9} m^2/s, respectively. However, compared with SOD40 (8.7326×10^{-15} m^2/s) and SOD80 (9.0908×10^{-15} m^2/s), the value of D_s was observed to be lower for SOD120 (8.0931×10^{-15} m^2/s), which indicated a lower diffusion of solute at a higher sugar dose. This result may be because the formation of a solute film on the surface of the flower samples becomes more prominent as the sugar dose increases and, as a result, reduces the transfer of sugar into flowers (Song et al. 2020). Comparable results were also reported by Etemadi et al. (2020) when working with apple rings in sucrose solution during OD.

The D_w and D_s values of SODS20 were highest (10.2170×10^{-15} m^2/s and 7.4465×10^{-15} m^2/s), followed by SODS40 (8.9672×10^{-15} m^2/s and 7.0949×10^{-15} m^2/s) and SODS6 (8.2488×10^{-15} m^2/s and 5.7947×10^{-15} m^2/s), suggesting that when the sugar size is too small or too large, the effective moisture and solute diffusivities are slow, which was also consistent with the water loss rate and solid gain rate with different sugar sizes. This result is probably because of slow seeping out of moisture from flowers and slow dissolution of solid sugar resulting from the smaller contact areas between the larger sugar and the flowers or increasing mass transfer resistance owing to the compact structure of small sugar particles. Moreover, the small sugar particles wetted by water but not yet dissolved during SOD would agglomerate to form larger particles, resulting in a decrease in the rates of WL and SG because of increasing mass transfer resistance (Ramya and Jain 2017).

Ultrasound treatment prior to SOD witnessed a positive result in terms of higher values of effective diffusivity for both moisture loss and solute uptake. With increasing ultrasonic treatment time, the D_w values and D_s values increased from 6.1900×10^{-15} to 6.4507×10^{-15} m^2/s and 4.2326×10^{-15} to 4.3975×10^{-15} m^2/s, respectively. The enhancement in the mass transfer by the application of ultrasound can be attributed to the formation of microscopic channels, thereby acting as a preferential for water and solute to diffuse, thus increasing the effective D_w values and D_s values (Nowacka et al. 2018). In addition, ultrasound induces cavitation, which enhances the mass transfer rate by reducing the internal and external resistance of the diffusion boundary layer (Nowacka et al. 2021). Likewise, Sharma and Dash (2019) reported that the application of ultrasound during the osmotic dehydration of black jamun fruit enhanced the effective D_w and D_s values by approximately 184–209% and 154–241%, respectively.

It was observed that in the Azuara model, the parameters (S_w and S_s) and effective diffusivity values were relatively low, and the scale parameters (α_w and α_s) and shape parameters (β_w and β_s) of the Weibull model were higher for the USOD samples than for the SOD and SODS samples. On the one hand, this observation may be because the jasmine flowers used for USOD differed in the harvesting period with SOD and SODS, so the difference in the components of jasmine flowers might lead to different mass transfers. On the other hand, frozen jasmine flowers, instead of fresh jasmine flowers (USOD), were used for SOD and SODS, and freezing treatment might affect the cell tissue of jasmine, especially regarding porosity and cell permeabilization (Ciurzyńska et al. 2016).

Physiochemical Changes in syrup

The physiochemical characteristics of the syrup samples are depicted in Table 6. The pH values of the syrup under different sugar doses, sugar sizes and ultrasonic times were 5.56-5.70, 5.50-5.64 and 5.44-5.54, respectively, but they did not follow a clear trend. However, relatively high dry weight (64.34±0.25%) and soluble solid content (64.30±0.36 °Bx) values were observed for SOD120 samples than for SOD40 and SOD80 samples. This result is probably because the relatively high sugar dose resulted in more WL of jasmine, and then more sugar was dissolved (Stojanovic and Silva 2007). With decreasing sugar size, the dry weight and soluble solid content increased. This phenomenon can be attributed to the smaller sugar size being more easily soluble than the larger sugar particles (Ramya and Jain 2017). Furthermore, higher dry weight and soluble solid content values were observed for the ultrasonic-assisted samples than for the USOD0 samples. This observation is probably because ultrasonic treatment promoted more water and water-soluble substances to diffuse from the flowers into the syrup (Rodrigues et al. 2009). The different process parameters were found to have a significant effect ($p < 0.05$) on syrup yield. The change in syrup yield was similar to that of WL; the higher the WL value was, the greater the syrup yield. The syrup yields of SOD 120 and SODS 20 were 28.15-149.53% and 8.41-9.65% higher than those of similar tests, respectively. Therefore, it is necessary to optimize the process parameters. The syrup yields of ultrasonic-assisted samples were 7.41-14.73% higher than USOD0, indicating that application of ultrasound will significantly increase syrup yield, and extending the ultrasonic treatment time is beneficial to increase the syrup yield.

TPC, TFC and antioxidant activities of syrup under various process parameters

The different amounts of syrup were extracted under different process parameters. To compare the effects of different process parameters on the TPC, TFC and DPPH radical scavenging capacity (expressed as Trolox equivalents) for syrups obtained via SOD. This paper used the following equation to calculate these values:

$$\mathrm{T} = \mathrm{Q} \times \mathrm{C} \tag{21}$$

where T represents the total TPC, TFC and DPPH radical scavenging capacity that diffused from flowers to syrup (mg), Q is the TPC, TFC and DPPH radical scavenging capacity (wet base) in syrup (mg/kg), and C is the syrup yield extracted under different process parameters (g).

The results of the bioactive compounds (TPC and TFC) for syrup extracted under the various process parameters are summarized in Table 7. The results showed that the syrup produced by SOD has abundant phenolic compounds and flavonoids, which not only enriched syrup with unique flavours of fruits or flowers but also exhibited potential health benefits. All the process parameters examined had significant effects on the mass transfer of the bioactive compounds (phenolic compounds and flavonoids) during SOD; hence, their concentration and quantity of syrup extracted differed significantly. As the sugar dose increased, the TPC and TFC decreased from 297.25 to 124.04 (mg GAE·kg^{-1} dw) and 347.59 to 151.71 (mg RU·kg^{-1} dw), and both the T_{TP} and T_{TF} values increased. This finding could be due to the higher sugar dose higher concentration gradients between, into and out of cells, which might have led to more TPC and TFC diffused from flowers to syrup (Feng et al. 2019).

The TPC and TFC increased from 112.71 to 128.96 (mg GAE·kg^{-1}dw) and from 141.37 to 160.10 (mg RU·kg^{-1} dw), respectively, while the T_{TP} and T_{TF} values increased with decreasing sugar size. The trends are similar to the mass transfer of WL because phenolic compounds and flavonoids are dissolved in water and then diffuse into the syrup, accompanied by WL from jasmine.

The TPC and TFC of the USOD samples were 162.10-175.18 (mg GAE·kg^{-1} dw) and 186.73-187.92 (mg RU·kg^{-1} dw), respectively, 28.62-39.00% and 11.84-13.55% higher than those of USOD0 samples, indicating that ultrasonication can significantly accelerate the mass transfer of phenolic compounds and flavonoids during SOD. The T_{TP} and T_{TF} of ultrasonic-assisted SOD samples were significantly higher ($p < 0.05$) than those of USOD0 and increased with increasing ultrasonic time. This result is explained mainly by microchannels and cell breakage resulting from ultrasonic treatment, resulting in the release of free or bound phenolic compounds and flavonoids of jasmine to the syrup (Dewanto et al. 2002). Likewise, Tayyab Rashid et al. (2020) reported that an ultrasound treatment application significantly reduced the TPC and total TFC of sweet potatoes compared to the untreated group during OD.

DPPH assays were employed to measure the potential source of antioxidation in the syrup extracted under different process parameters. The influence of different process parameters on the antioxidant capacities of the syrup is presented in Table 7. The. The DPPH assay varied from 158.37 to 421.90 (mg Tx·kg^{-1} dw), 160.70 to 170.26 (mg Tx·kg^{-1} dw) and 230.88 to 313.55 (mg Tx·kg^{-1} dw) for the SOD, SODS and USOD samples, respectively, indicating that sugar dose and ultrasonic treatment time have significant effects ($P < 0.05$) on DPPH, while sugar size has no effect ($P>0.05$) on DPPH. This consistent and statistically significant higher antioxidant activity of the syrup extracted under different process parameters could again be attributed to the influence of the SOD concentration gradient and the acoustic cavitation of ultrasonication, which disrupted the cell structure and created microchannels on the surface of the jasmine tissue, as explained earlier under the TPC and TFC assays. A similar phenomenon was observed by Alolga et al. (2021). Our results further established an association between antioxidation and TPC, since higher TPC is usually attributed to increasing antioxidant activities. Hence, the results of antioxidation of the syrup are consistent with those of TPC determination.

GC-MS analysis of the volatile flavor components

GC–MS analyses of the syrup extracted under different process parameters and jasmine flowers detected various classes of compounds at different concentrations, and the details are provided in Supplementary Tables S1-S2. In total, 81 compounds were identified in the jasmine flowers, including linalool (21.47%), α-farnesene (21.13%), benzyl alcohol (7.52%), cis-3-hexenyl benzoate (7.32%), benzyl acetate (3.04%), methyl salicylate (3.02%), and methyl anthranilate (2.62%), which was consistent with Issa et al. (2020), who found that the compounds mentioned above accounted for the aroma of fresh jasmine flowers.

Fifty to 56 volatile compounds were found in syrup extracted by SOD under different process parameters, suggesting that the flavour compounds of jasmine were effectively extracted by SOD. The volatile compounds extracted could be categorized into 9 groups of compounds viz. ester, aldehyde/furans/phenol, terpenes, hydrocarbons, ketones, alcohols, and acids, with linalool (37.94-46.06%) as a predominant compound, followed by benzyl alcohol (20.80-23.90%), methyl salicylate (8.53-9.51%), trans-3-hexen-1-ol (3.51-4.81%), methyl anthranilate (2.95-5.87%), and cis-3-hexenyl benzoate (2.23-3.90%), which is not exactly the same as jasmine flowers. Alcohols accounted for more than 68% of the extracted aromatic substances, while others accounted for less than 32%, indicating that the aromatic substances of jasmine flowers were selectively extracted, depending mainly on their intrinsic properties, such as hydrophilicity. This was consistent with the findings of Ramalingam et al. (2021) that partial volatile compounds can be extracted into syrup in the

SOD process. There was no significant difference in the contents of major volatile compounds of syrup samples under different process parameters ($P>0.05$), indicating that the amount of major volatile compounds extracted was not affected by SOD process parameters (Tsitlakidou et al. 2019). However, in addition to the intrinsic volatile compounds of jasmine, many new volatile compounds, such as octanal, nonanal, decanal, trans-cinnamaldehyde and acetic acid, were detected at low concentrations in syrup, which may be produced by enzymatic action during the SOD process (Ramalingam et al. 2021). Moreover, compared to the syrup extracted without ultrasound (USOD0), all USOD syrup samples contained new volatile components, including ethyl trans-4-decenoate, methyl 2-acetamidobenzoate, neo-alloocimene, stab, etc. This observation may be because the acoustical cavitation of ultrasonication promotes the decomposition, isomerization, and oxidation of volatile components (An et al. 2019).

Morphological analysis of USOD samples via SEM

The effects of ultrasonication on the structure of osmotically dehydrated jasmine petals during SOD were observed under SEM (Fig. 4). The SEM images (2000× magnification) in the section of osmotically dehydrated jasmine petals showed distinct differences in the microstructure of jasmine subjected to different ultrasonic treatment times. The cell tissue of the defrosted jasmine before SOD looks like fish scales with irregular shapes and shows orderly arrangement, good structural integrity with wrinkles on the surface and clear grooves between cells (Fig. 4 a). After 84 h of SOD, most of the cells decreased in size and wrinkled, and the grooves between the cells widened and deepened (Fig. 4 b), which increased the cell tissue density and resulted in increasing the mass transfer resistance of water and solute, leading to slower rates of WL and SG in the later SOD stage. However, the micrographs of samples with ultrasonic treatment (Fig. 4 b–j) showed different degrees of cellular distortion and tissue breakdown, and the clear grooves between cells were blurred and flattened with the leaching materials. With increasing ultrasonication time, the surface of the cells was severely wrinkled, the cellular distortion and the tissue breakdown became increasingly serious, and the intercellular grooves were widened and filled with leaching materials, resulting in more irregular intercellular spaces. After 120 min of ultrasonication, most of the cells were even buried by leaching materials, and the cell surface appeared porous, especially the intercellular grooves. Ultrasound treatment generated acoustic waves that created cavitation voids near the flower surface. The cavitation voids showed compression and expansion phases and then collapsed. This process releases gases from the intercellular spaces that might be responsible for the spongy effect and changes the flower tissue (Sharma and Dash 2019; Prithani and Dash 2020). The spongy effect caused cellular distortion and tissue breakdown inside the pores and increased the number of microscopic channels and pores on the flower surface, thus enhancing the diffusivity of effective water and solute in the jasmine petals; hence, more polyphenols, flavonoids, and antioxidant components diffuse to the syrup, so the syrup yield and the nutrients of the syrup both increased.

Conclusion

The results showed that a high sugar dose (120% w/w) and moderate sugar size (6-20 mesh) enhanced the WL and SG during SOD and produced more syrup. Furthermore, a high sugar dose (120% w/w) and small sugar size (40 mesh) enhanced the diffusion of polyphenols and flavonoids in jasmine to syrup. The high R^2 and low χ^2 value represented the validity of the Azuara model for determining WL_∞ and SG_∞ values, and the Weibull model was suitable for predicting both moisture and solute fractions in different process parameters. With increasing ultrasonic treatment time, the rupture of jasmine petal tissue became increasingly obvious, and the number of microscopic channels or pores on the petal surface increased. Therefore, the application of ultrasound in the SOD process can enhance mass transfer, thus allowing for more polyphenolic, flavonoid and antioxidant components to diffuse into the syrup, increasing the syrup yield and nutritional value. The partial flavour compounds of jasmine were effectively extracted by SOD under different process parameters, and alcohols (more than 68%) were the main flavour components of the syrups, but the amount of major volatile compounds extracted was not affected by the SOD process parameters. Therefore, SOD technology may become a novel technology to produce flavoured syrup, and the application of ultrasound treatment during SOD can be used to increase syrup yields and nutrients.

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Data availability The datasets generated during the current study are available from the corresponding author upon reasonable request.

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