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RESEARCH

Stability of Fructooligosaccharides in Convectively Dried Fruits After Initial Osmoconcentration

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Abstract
The aim of this study was to determine the effect of temperature and time of convective drying on the content of fructooligosaccharides (FOS) in apples, plums and strawberries to which FOS had been introduced by osmoconcentration. The share of oligosaccharides in total sugars was analyzed. In apple tissue, fructooligosaccharides were stable in the temperature range 40–80 °C during drying for up to 8 h. Convective drying of osmotically dehydrated strawberries caused FOS losses; the FOS retention after 8 h at 80 °C was 40%. In the case of plums, 40% retention was recorded after just two hours at 80 °C. Therefore, in the case of some fruits, obtaining a satisfactory level of fructooligosaccharides in the dried material with the assumed level of dry substance requires the determination of appropriate process parameters.

Keywords Fructooligosaccharides · Stability · Convective drying · Apple · Strawberry · Plum

Introduction

Dried fruits are a very popular addition to many food products, including cereals, muesli, yoghurts, and desserts. The principal technique of fruit dehydration is convection drying using heated air, the temperature of which may lie in the wide range of 35–80 °C (Onwude et al., 2022). The process may take several hours or even more than 10 h. In such conditions, drying increases the microbiological stability, but at the same time causes the degradation of some of the plant material components (Petikirige et al., 2022; Raveendran et al., 2022).

Components of fruit include fructooligosaccharides. The content of these in fresh material is usually not high. Jovanovic-Malinovska et al. (2014) determined the FOS content in a number of fruits (and vegetables). For most of the tested fruits it was below 0.5%, and for a significant group (including apples, plums and strawberries) it was even below 0.1%. Fruits with a significant content of fructans include bananas, which are rich in inulin. Pongmalai and Devahastin (2020) determined the content of this substance to be over 37 g/100 d.b. However, the content of fructooligosaccharides (the sum of kestose and nystose) was many times lower, at 1.34 g/100 g d.b.

Because of their health-promoting properties, fructooligosaccharides are desirable in the human diet; they are a breeding ground for lactic acid bacteria, which inhibit the development of pathogenic microflora. The consumption of fructooligosaccharides contributes to improving the absorption of magnesium and calcium in the intestines, reduces constipation, and reduces the risk of colon cancer (Costa et al., 2021; Garbacz, 2022; Iannitti & Palmieri, 2010).

Fruit can be enriched with FOS by means of osmoconcentration in hypertonic solutions containing those saccharides. Klewicki and Uczciwek (2008) obtained FOS contents in osmotically dehydrated apples and plums in the range 7–25%. In a study by Matusek et al. (2008), dehydrated apples contained about 15% fructooligosaccharides (the sum of DP3–DP6). Ramesh et al. (2013) dehydrated carrots and papaya. They did not report the oligosaccharide content after osmotic dehydration alone (dry matter content 34–50%), but only after supplementary convective drying (to 92–95% dry matter). Contents of fructooligosaccharides in products that contained them (in some cases FOS were not detected) ranged from 7.7% to 14.2%.

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Osmotic dehydration is often a pre-convection step carried out prior to convective drying to achieve an initial reduction in the water content in the raw material and to modify the tissue structure to make air drying faster and more effective (conventional hot air drying is time-consuming and therefore energy-intensive and costly) (Mundada et al., 2010; Sakooei-Vaygham et al., 2020). Of course, an important issue is the resistance of the oligosaccharides introduced into the fruit tissue to the unfavorable conditions of convective drying.

The stability of FOS depends very much on the conditions in which they reside. Therefore, studies have been made to determine the stability of these saccharides depending on the environment, and on the method and parameters of the processing, preservation or storage of the material containing them [Benkeblia et al., 2005; Huebner et al., 2008; Matusek et al., 2011; Vega & Zuniga-Hansen, 2015; Almeida et al., 2017; Moro et al., 2022). The results obtained range from 100% stability (Cascales et al., 2021) to complete degradation (Cao et al., 2002). These results concern various products; however, in the existing literature data there is no information on the stability of fructooligosaccharides in convectively dried fruits that have been subjected to initial osmoconcentration.

Although osmotic dehydration was used as a pre-treatment of the plant material, the purpose of those experiments was different from the purpose of the research presented in this article. For example, Turkiewicz et al. (2020) determined the effect of osmoconcentration of Japanese quince fruit in fruit concentrates, before convective drying and vacuum-microwave finish drying, on the dehydration kinetics and on physiochemical parameters such as dry weight, water activity, content of ascorbic acid, organic acids, and polyphenols. Sugars were also determined, but excluding fructooligosaccharides. In another experiment (Ahmad-Qasem et al., 2015), apple cubes were impregnated with olive leaf extract in order to infuse polyphenolic compounds into the tissue (which improves the antioxidant capacity). Next, the retention of infused polyphenols after convective drying was evaluated. Bchir et al. (2012) performed osmo-convective dehydration of pomegranate seeds using a sucrose solution. The drying kinetics and the effect of combined drying on antioxidant capacity, total polyphenols, color and texture were determined. Salim et al. (2019) determined, among others, vitamin C and chlorophyll. A sucrose solution was also used by Noshad et al. (2012) to dehydrate quince before hot-air drying. They determined the rehydration ratio and shrinkage. Other substances have also been used to prepare solutions for such experiments, for example sodium chloride (melon dehydration; mass transfer evaluated by Aminzadeh et al., 2012) and trehalose (eggplant dehydration; shrinkage, browning and loss of polyphenols evaluated by Adiletta et al., 2016). Osmo-convective fruit drying was also evaluated for its effect on the organoleptic properties of the product (Nowicka et al., 2015).

The analysis of the literature shows that existing research on combining osmoconcentration (including infusion) with convective drying is multidirectional. It includes the study of the effect of various solution-forming substances on the course of dehydration, the content of various substances in the tissue after drying, and rheological and organoleptic properties. However, it does not cover the question of the stability, during air drying, of fructooligosaccharides introduced into the plant material by osmoconcentration in order to improve its health-promoting properties. The data presented in this article serve to fill this gap.

Materials and Methods

Materials

Fruit purchased in a supermarket in Łódź (Poland) was used in the research. The tested apples are of the Idared variety, grown in Poland. The fruit with the skin, without seeds, was cut into cubes of size 1 cm × 1 cm × 1 cm. The strawberries used in the research came from Spain. Smaller fruits without stalks were cut lengthwise into halves, and larger fruits were cut into quarters. Polish-grown peach plums, with the seeds removed, were cut into quarters, and then in half crosswise.

The following substances were used in the experiments and analyses: sucrose (Krajowa Spółka Cukrowa S.A., Toruń, Poland); Amberlite IR-120 cation exchanger and Amberlite IRA-67 anion exchanger (Sigma-Aldrich Co, Saint Louis, MO, USA); Fructozyme enzyme preparation containing fructosyltransferase and β-D-fructofuranosidase from *Aspergillus niger* with an activity of 7840 U/ml (Novo Nordisk A/S, Bagswaerd, Denmark); glucose, fructose, calcium carbonate and sodium hydroxide pure p.a. (POCh S.A., Gliwice, Poland).

Methods

Osmotic Dehydration

Preparation of fructooligosaccharide solution

Quantities of 880 g of sucrose and 720 g of water were weighed into a 2 L beaker. After the sucrose had dissolved, the solution was heated to 57 °C and then placed in a water bath at that temperature. The solution was constantly stirred with a stirrer. The pH of the solution was adjusted to 7.0 ± 0.1 with 0.05 M NaOH. Next, 5.6 ml of the enzyme preparation was added, and the reaction took place for 3 h with constant control of the pH value; if the pH dropped, NaOH solution was added to restore a value of 7.0. After 3 h, the solution was brought to the boil to stop the reaction, and then cooled to room temperature.

Dehydration process

A quantity of 400 g of fruit was added to the prepared solutions at a temperature of 22 ± 1 °C. The dehydration process lasted 2.5 h. During this time, the solutions together with the fruit were stirred with a glass baguette (for 1 min every 5 min). After the specified time, the fruits were placed on a sieve and then rinsed by immersing them twice in demineralized water. After drying on filter paper, the samples were ready for convection drying.

Convection Drying

Air drying was carried out at three temperatures: 40 °C, 60 °C and 80 °C (respectively: MEMMERT GmbH + Co. KG, Schwabach, Germany; ELKON, Łódź, Poland; POL-EKO-APARATURA sp.j., Wodzisław Śląski, Poland) for 8 h. Two samples were taken every 2 h.

Dry Substance Determination

Samples (ca. 2 g) were placed in dried weighing bottles, weighed, and then placed in a vacuum oven (MEMMERT GmbH + Co. KG, Schwabach, Germany) set at 70 °C for 24 h. After this time the bottles were cooled in a desiccator (1 h), and they were weighed again. On this basis, the percentage content of dry substance was calculated.

HPLC Analysis of Sugars

Samples for analysis were prepared by extraction from the collected samples during drying. The sample (approx. 3 g) and 0.5 g of calcium carbonate were weighed into approximately 50 ml of water in a beaker. The whole was brought to the boil and then cooled. After quantitative transfer to a 100 ml volumetric flask, the solution was made up to volume with water, left for 10 min, and then passed through a filter paper. For desalting, the solution was passed through a column (9 × 64 mm) filled with a mixture of cation exchanger and anion exchanger in a ratio of 1:2; the first 3 ml was discarded, and the next 2 ml was collected. The samples prepared in this way were subjected to chromatographic analysis.

HPLC analysis was performed using a Knauer (Germany) chromatograph equipped with an Aminex HPX-87C, 8 × 300 column (Bio-Rad, Richmond, USA). The separation temperature was 85 °C. The mobile phase used was water, the flow rate was 0.5 ml/min, and the sample volume was 20 µl. A refractometric detector was used. EuroChrom software was used for data processing.

Acidity Determination

Samples for the determination of acidity were prepared by extraction from fresh and dehydrated fruits. The sample (5 g) was weighed into approximately 50 ml of water in a beaker and then brought to the boil. After cooling, it was quantitatively transferred to a 100 ml volumetric flask, topped up with water, and then passed through a filter paper. Next, a 40 ml sample was taken and placed in a beaker with a stirrer and a pH electrode. Titration was carried out using NaOH solution; the titration was completed when the pH reached 7.0. The acidity was calculated according to the formula:

$$X = \frac{a \cdot n \cdot k}{m}$$

where X is the acidity [g/100 g]; a is the amount of NaOH solution used for titration [ml]; n is the concentration of the NaOH solution used for titration (0.0114 M); k is a factor (0.067 – acidity expressed as malic acid); m is the mass of the sample [g].

Statistical Analysis of Results

The analysis was performed with Statistica 10 software (Stat Soft, Tulsa, USA) using Duncan's post hoc test.

Results and Discussion

The solution used for fruit osmoconcentration contained 3.64 g/100 g nystose and 17.58 g/100 g kestose (Table 1). The content of these two fructooligosaccharides (as a sum) was then determined in the dried fruit.

Table 2 shows the dry substance content of fruit during convection drying at different temperatures. The fruits after osmotic dehydration, before convective drying, have a dry substance content two to three times higher than that of the fresh material, which confirms that the initial dehydration process, aimed at shortening the time of convective drying, is successfully conducted. The process of drying with hot air can be considered satisfactory – the dry substance content in

Table 1 Saccharides determined in hypertonic solution

Saccharides	Concentration [g/100 g]
nystose	3,64
kestose	17,58
saccharose	11,29
glucose	17,36
fructose	3,58

Table 2 Dry substance content in fresh, osmotically dehydrated and convectively dried apples, plums and strawberries

Drying temperature [°C]	Dry substance [g/100 g]					
	F	OD	CD2	CD4	CD6	CD8
	apple					
40	15.64 ± 0.10	31.22 ± 0.85	33.69 ± 0.26	67.11 ± 2.80	81.79 ± 0.66	86.06 ± 0.63
60			64.33 ± 0.05	87.98 ± 0.36	91.93 ± 0.47	92.60 ± 0.08
80			88.65 ± 0.23	94.77 ± 0.30	97.17 ± 0.22	98.56 ± 0.21
	plum					
40	7.22 ± 0.32	17.66 ± 0.32	24.90 ± 0.52	31.63 ± 0.40	55.14 ± 0.25	69.14 ± 0.53
60			51.13 ± 0.08	60.90 ± 0.03	80.11 ± 0.38	89.84 ± 0.80
80			60.31 ± 1.80	87.26 ± 0.68	94.36 ± 0.19	95.38 ± 0.35
	strawberry					
40	5.04 ± 0.07	15.83 ± 0.94	18.88 ± 0.41	23.49 ± 0.84	30.36 ± 2.81	65.26 ± 0.46
60			17.86 ± 1.42	34.18 ± 2.37	48.92 ± 0.27	72.52 ± 0.59
80			26.51 ± 0.42	49.60 ± 2.64	84.07 ± 4.20	91.41 ± 1.15

F – fresh fruit; OD – osmotically dehydrated fruit; CD2 – OD + convective drying for 2 h; CD4 – OD + convective drying for 4 h; CD6 – OD + convective drying for 6 h; CD8 – OD + convective drying for 8 h

osmotically dehydrated apples increased from about 31% to over 80% (commercial dried fruit contains from about 70% of dry matter) after six hours at 40 °C. At 60 °C, a level of 88% was reached after 4 h, and at 80 °C, a similar level was reached after only 2 h. The convective drying of plums was slower. After 8 h at 40 °C the dry substance content had increased to nearly 70%; at 60 °C it reached 80% after 6 h. At the highest temperature, after 4 h, the dry substance content reached 87%. Strawberries were the fruit that released water the slowest. At the lowest temperature, after 8 h, the dry substance content was approximately 65%; increasing the temperature to 60 °C raised the value to over 72%. At the highest temperature, it took 6 h to obtain over 80% dry substance.

Figure 1 shows the proportion of saccharides in total sugars in dried apples. Even at the highest temperature used, no downward trend was observed for FOS (although some fluctuations occurred, which can be attributed to the natural heterogeneity of biological material, in this case the fruits). The content of FOS was in a range of 30.3–33.4% of total sugars. The stability of saccharides containing fructosyl residues in the tested apples after osmoconcentration (acidity 0.20 g/100 g) is also evidenced by the almost unchanged level of sucrose (16.9–17.8% of total sugars); no statistically significant differences were noted in any case. Also, the proportions of glucose and fructose do not indicate an increase in the content of those monosaccharides, which would certainly occur in case of hydrolysis of the fructooligosaccharides.

Figure 2 shows the proportions of saccharides in dried strawberries. Their acidity was twice as high as that of apples. In the case of drying at 40 °C, the shares of fructooligosaccharides are at a similar level at each hour of drying. The values range from 15.2% to 19% and are not statistically different, although there is a slight downward trend. Significant differences were noted in the content of sucrose, the value of which dropped from 24.6% to 8% (statistically significant differences). At the same time, a significant increase in glucose was observed (statistically significant differences) from 33.1% to 46.2% (after 8 h of drying), as well as an upward trend in fructose, from 23.3% to over 30% (there was significant fluctuation but the statistical test did not show significant differences). The results indicate that FOS hydrolysis can occur even at the lowest tested temperature. The main factor causing the degradation of FOS is considered to be a sufficiently high proton concentration (Bolin et al., 1983; Matusek et al., 2011). However, some contribution may also come from invertase, which is present in fruits (Kim et al., 2015; Peng et al., 2023; Topcu et al., 2022).

At 60 °C, the share of FOS was found to decrease from 18.9% to 11.3% (a statistically significant difference). In the case of sucrose and monosaccharides there were significant fluctuations; no clear downward or upward trend was observed. However, the changes were more visible after drying at 80 °C. The share of FOS in total sugars decreased from 18.9% to 13.5% after 4 h of drying (the difference is statistically significant). The share of sucrose also decreased from 24.6% to 12.7%. The above data indicate a significant decrease in the stability of fructooligosaccharides in the tissue of dehydrated strawberries at temperatures of 60 °C and above. Considering that the goal would be to obtain a product with a dry substance content of approximately 70%, the time necessary to achieve this goal (Table 2) may already be sufficient to cause losses of fructooligosaccharides (a loss of 40.3% of FOS after 8 h at 60 °C, and a loss of 33.4% of FOS after 6 h at 80 °C).

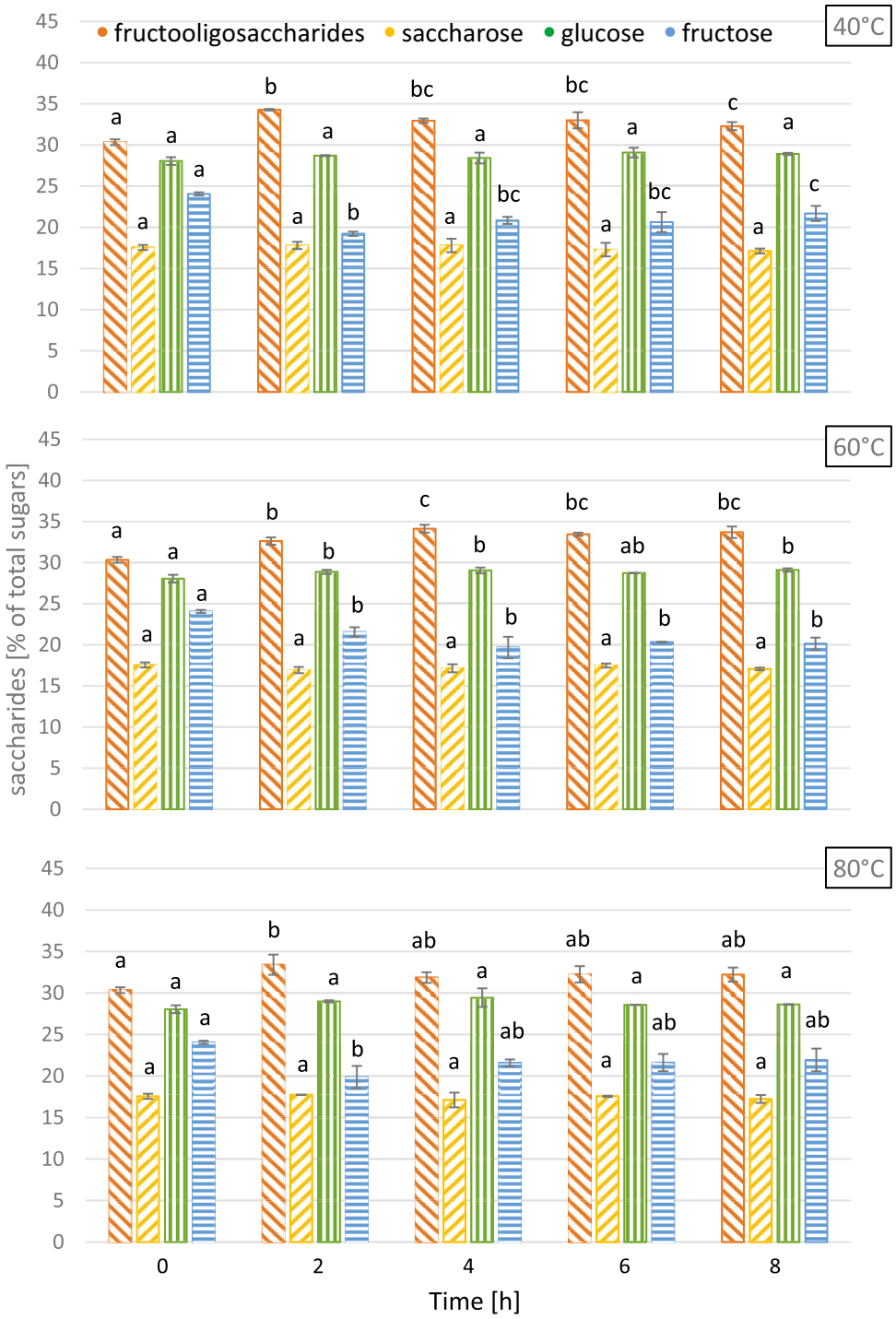

Fig. 1 Share of individual saccharides in total sugars in apples after osmoconcentration convectively dried at different temperatures (acidity of osmo-dehydrated material 0.20 g/100 g)

The saccharide profile of dehydrated plums (acidity 0.77 g/100 g) is shown in Fig. 3. A loss of fructooligosaccharides was recorded as early as two hours at 40 °C (from 28.2% to 18.9% of total sugars). Later, the share of FOS was quite stable (18.9–19.9% of total sugars; at this stage the differences were not statistically significant). The share of sucrose in total sugars also decreased – from 17.7% to 12.3% after 6 h. Glucose values showed a slight upward trend, but with no statistically significant differences. On the other hand, there was a clear increase in fructose after the second hour of drying (from 21.1% to 28.2%), which is consistent with the decomposition of FOS.

An increase in temperature to 60 °C caused a higher loss of fructooligosaccharides. The share of FOS in total sugars significantly dropped from 28.2% to 13.5% after 6 h, with 17.3% recorded after 8 h (there were no significant differences

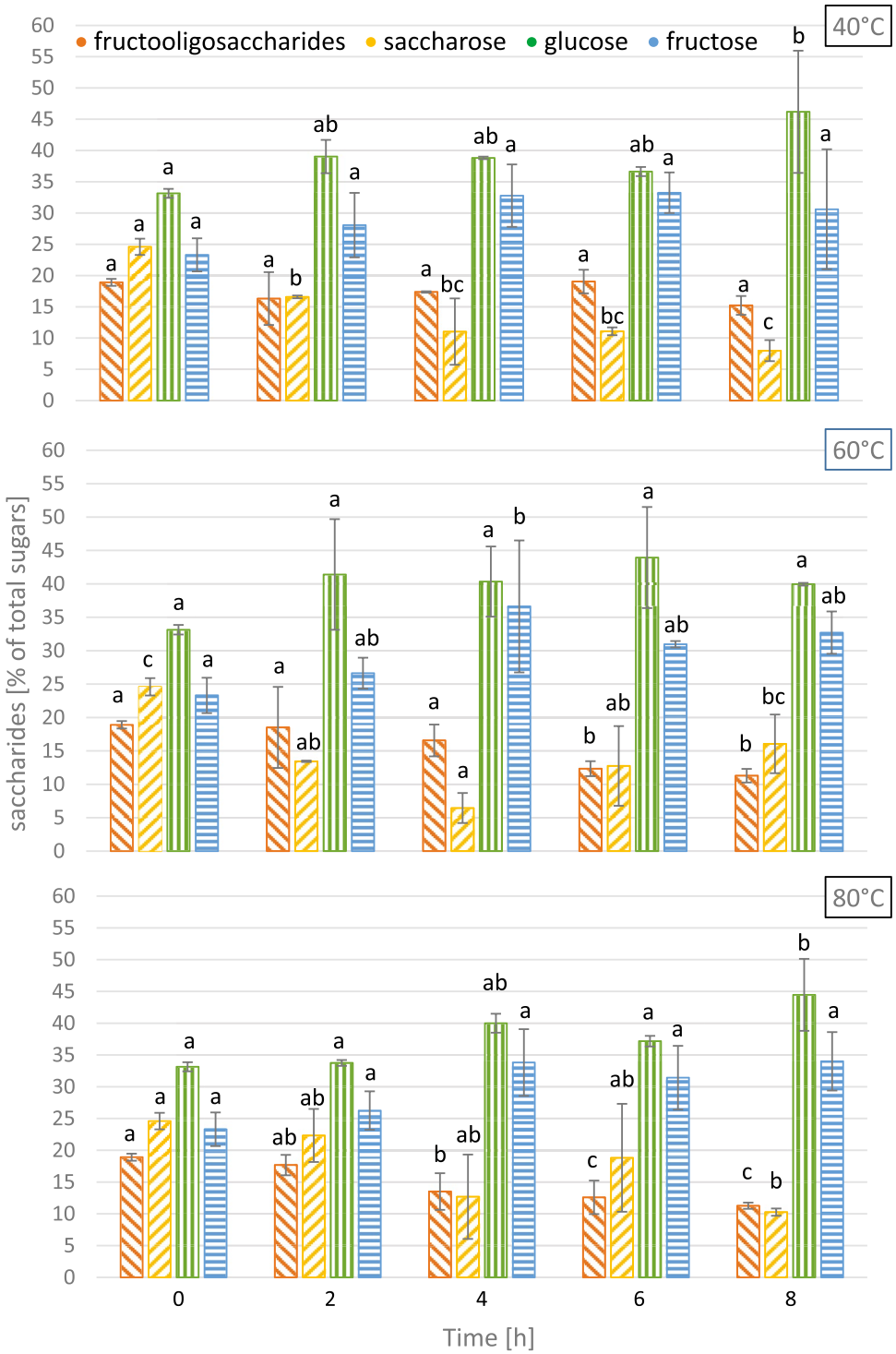

Fig. 2 Share of individual saccharides in total sugars in strawberries after osmoconcentration convectively dried at different temperatures (acidity of osmo-dehydrated material 0.40 g/100 g)

between 6 and 8 h). The values for other saccharides also differed significantly; there was a decrease in the amount of sucrose (from 17.7% to 10.7% of total sugars) and an increase in the amount of glucose (from 33.1% to 39.1% of total sugars). In the case of fructose, a large increase was noted in the first hour (from 21.1% to 31.1% of total sugars), after which its content was fairly stable (29.7–34.1% of total sugars).

Fructooligosaccharides were least stable at 80 °C; the share of FOS in total sugars decreased from 28.2% to 8.6–13.7% depending on the drying time. There was also a decrease in sucrose, from 17.7% to 12.1% of total sugars. The decomposition of these saccharides caused significant increases in glucose and fructose.

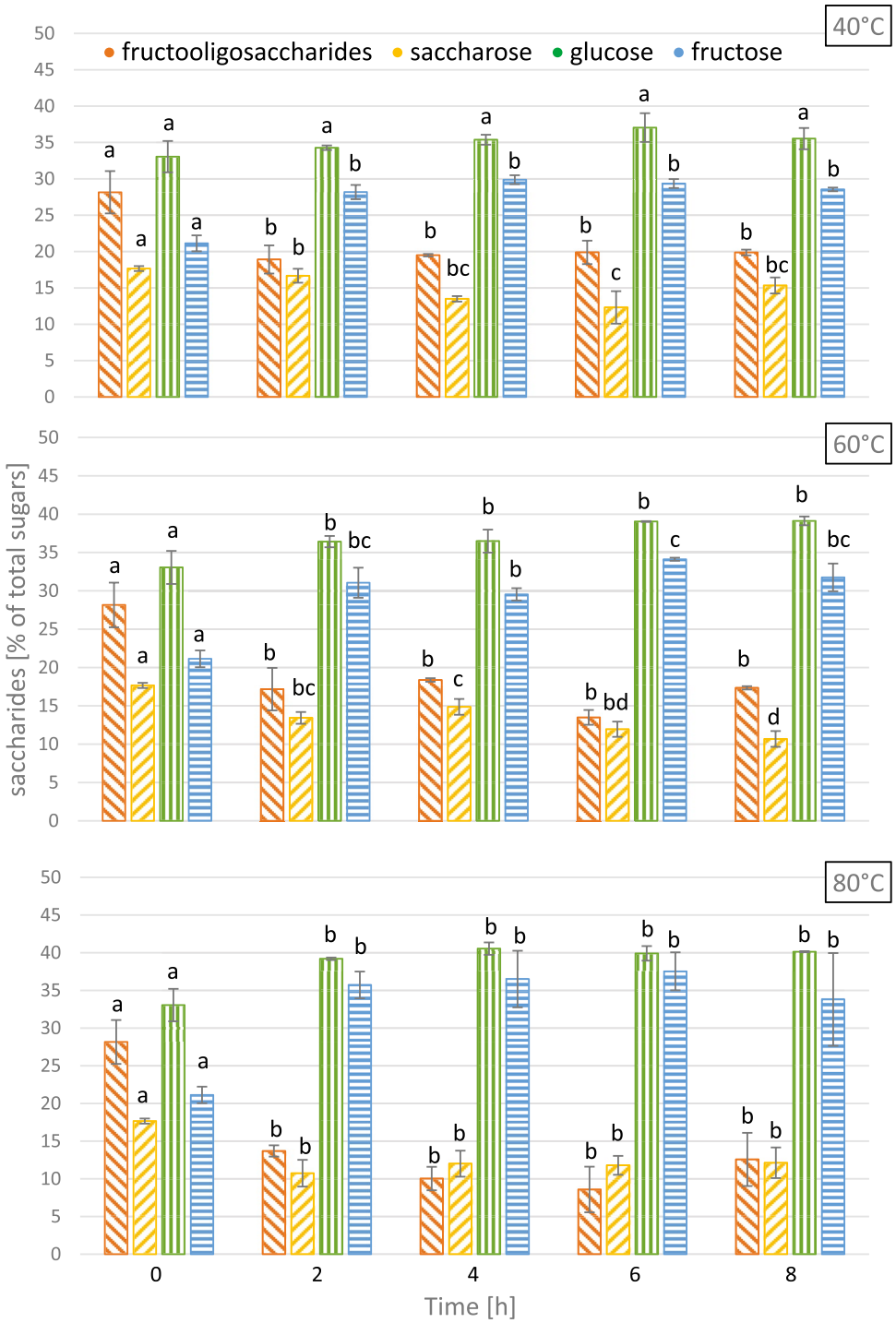

Fig. 3 Share of individual saccharides in total sugars in plums after osmoconcentration convectively dried at different temperatures (acidity of osmo-dehydrated material 0.77 g/100 g)

The above data therefore indicate that in order to reduce FOS losses, convective drying of plums (following initial osmotic dehydration) should be carried out in appropriately selected conditions, because obtaining the target dry substance content may entail various FOS losses depending on temperature and time. To obtain about 70% dry substance content, plums should be dried by convection for 8 h at 40 °C, between 4 and 6 h at 60 °C, or between 2 and 4 h at 80 °C (Table 2). Use of the lowest temperature enables the highest retention of FOS, which is about 70% after 8 h. At 60 °C, the retention ranges from 65.2% to 47.9%. At 80 °C, approximately 60% of FOS is lost after just 2 h.

The stability results obtained in this work can be compared with the results of research on other products, subjected to other techniques of processing, preservation and storage. For example, in apple puree, at low temperature (4 °C),

fructooligosaccharides exhibited high stability over a period of 30 days (Keenan et al., 2011). Similarly, in apple impregnated with fructooligosaccharides (pH approx. 4.8) FOS remained stable for four weeks at room temperature; after six weeks, the loss was about 11% (Mejía-Águila et al., 2021).

Significant losses of FOS should be taken into account in the case of long-term storage of fruit juices. In pineapple, mango and orange juices, the FOS content decreased from 3.5–3.8 g/100 ml to 2.0–2.4 g/100 ml after six months of storage at 4 °C, while at room temperature, the content fell to only 0.4–0.6 g/100 ml (Renuka et al., 2009). Even at sub-zero temperatures, significant losses of FOS can occur. Topolska et al. (2017) showed that in strawberry sorbets (with the addition of fructans of various origin) FOS retention was approximately 70% after 8 weeks of storage at –22 °C.

In addition to long storage time, high temperatures (associated with various technological operations) can also cause significant losses of FOS. L'homme et al. (2003) reported the loss of FOS (kestose) in products based on apples (apple dessert and stewed apples with bananas) heated at 80–110 °C for up to 30 min. The losses were significant. In the stewed apples with bananas, the kestose content was 0.10 g/100 g of freeze-dried product, while the levels of kestose in the raw ingredients – apple puree and banana puree (mixed in the ratio 72:16) – were respectively 0.69 and 0.68 g/100 g of lyophilisate.

Depletion of fructooligosaccharides also occurred during the production of guava jam (Mesquita et al., 2013). The initial proportion of FOS in the pulp was 16.87 g/100 g; after the jam had been made and then pasteurized in boiling water, the proportion of FOS decreased by 35%.

Pasteurization and sterilization also affected the level of FOS in the products obtained by Benitez et al. (2013) from onion waste. Those treatments caused a 35% decrease on average in the content in the juice during pasteurization (100 °C, 11–17 min depending on the onion variety) and a 56% decrease on average during sterilization (115 °C, 17–18 min depending on the onion variety).

Also, extrusion to obtain breakfast cereals caused the degradation of FOS (Duar et al., 2015). At temperatures of 120–170 °C, the percentage recovery of previously added fructooligosaccharides was below 50%, and in some cases even below 25%.

On the other hand, Cascales et al. (2021) reported 100% stability of FOS in pineapple nectar subjected to thermal preservation at 115 °C for 15 s (pH 3.2–3.5). Similarly, Courtin et al. (2009) noted a low level of FOS decomposition (up to 10%) in model solutions with pH 2–3, when heated at 100 °C for 5–60 min. Glibowski et al. (2020) confirmed the stability of FOS in apple juice (pH 3.68) subjected to the following treatment: heating to 100 °C, addition of FOS, then (after 1 min) cooling (for approx. 1 h) to room temperature. In a study by Cao et al. (2002) fructooligosaccharides were stable at pH 4–8; no more than 3% of FOS was lost after 0.4–15 min of heating at 70–100 °C. On the other hand, lowering the pH below 4 and increasing the temperature to 121 °C resulted in complete degradation of FOS after 20 min.

Conclusions

The stability of fructooligosaccharides in convectively dried fruits after initial osmoconcentration depends significantly on the raw material used. Out of the tested material, apple tissue (with an acidity of 0.2 g/100 g) provides the highest stability of FOS; no hydrolysis of oligosaccharides occurs at 40–80 °C to such an extent as to cause significant changes in the saccharide profile. Convective drying of osmotically dehydrated strawberries (acidity 0.4 g/100 g) causes losses of FOS, particularly at temperatures of 60 °C (60% retention after 8 h) and 80 °C (40% retention after 8 h). Among the tested fruits, plums (acidity 0.8 g/100 g) exhibits the lowest stability of fructooligosaccharides. This stability is very dependent on temperature and time: at 80 °C only about 40% of the initial amount of FOS remained after 2 h, while at 40 °C about 70% remained after 8 h. Therefore, in the case of some fruits, special attention should be paid to the parameters of drying (time, temperature) if the goal is to obtain not only the dry substance content desired by the manufacturer, but also a satisfactory level of fructooligosaccharides.

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Author Contribution Patrycja Łuczak: carrying out experiments and analyses, data analysis, writing (original draft); Robert Klewicki: conceptualization, methodology, data analysis, supervision, critical review; Elżbieta Klewicka: writing (final draft), editing.

Data Availability The data supporting the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Competing of Interest The authors declare that the research was carried out in the absence of any commercial or financial relationships that could be construed as a conflict of interest.

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