IDS 2020
22nd International Drying Symposium

IDS'2020 – 22nd International Drying Symposium
Worcester, Massachusetts, USA, June 28 – July 1, 2020
DOI: http://dx.doi.org/thisisa/placeholder/

Encapsulation of asparagus aromas by spray drying with different carrier formulations

Siccama J.W. [a*]; **Pegiou, E.** [b]; **Zhang, L.** [a]; **Schutyser M.A.I.** [a]

[a] Laboratory of Food Process Engineering, Wageningen University, Wageningen, the Netherlands

[b] Laboratory of Plant Physiology, Wageningen University, Wageningen, the Netherlands

*E-mail of the corresponding author: joanne.siccama@wur.nl

Abstract

Asparagus juice concentrate was spray-dried using different carrier formulations. Maltodextrin was partially replaced by cellulose-based carriers, i.e. asparagus fibre, citrus fibre or microcrystalline cellulose. The maximum level of replacement was found to be 3 % w/w for asparagus/citrus fibre and 10 % w/w for microcrystalline cellulose, due to fibre insolubility. The powders obtained after drying 40 % w/w initial solids retained asparagus key volatile 2-Methoxy-3-isopropyl pyrazine better than 30 % w/w. The partial replacement resulted in powder with similar physical properties but slightly reduced retention of the pyrazine. We concluded that asparagus fibre could potentially be used as a carrier agent.

Keywords: *asparagus fibres; spray drying; volatiles retention; GC-MS; metabolomics*

1. Introduction

White asparagus (*Asparagus officinalis*) is a vegetable rich in volatile compounds with a characteristic flavour profile [1]. Traditionally asparagus powders are of poor quality due to aroma loss during hot air drying. Alternatively, spray drying is a method commonly used in the food industry to preserve fruit and vegetable juices in powder form [2, 3]. Spray drying allows for the encapsulation of volatile compounds thanks to the formation of a semi-permeable skin during the dehydration process [4]. However, similar to other vegetable and fruit juices, spray drying of asparagus juice is challenging because of the high levels of small sugars, which result in stickiness problems due to their low glass transition temperature (T_g). Therefore, carrier agents such as maltodextrin with low dextrose equivalents (DE) are often added since they have a high T_g and enable skin formation during drying [5]. Previous work demonstrated that spray drying of asparagus juice concentrate with the addition of maltodextrin improved the physical properties and volatile profile of the spray-dried asparagus powder [6].

Nowadays, the transition towards producing “clean-label” foods is gaining greater attention [7, 8]. Spray-dried powder containing maltodextrin as a carrier agent does not meet the requirement of “clean-label” production. Therefore, we suggest partly replacing maltodextrin with fibres that are considered to be more authentic and are perceived as being more natural, such as asparagus fibre. Specifically, asparagus fibre can be obtained by drying after the juice has been pressed out. Asparagus fibre might be used to (partially) replace maltodextrin as the carrier agent during the spray drying of asparagus juice, which enables complete use of the asparagus. However, replacing maltodextrin with asparagus fibre might be challenging due to its high cellulose content. Cellulose and maltodextrin have different physicochemical properties. For example, the addition of insoluble cellulose-based material may increase the viscosity of the feed, which may complicate the spray drying process.

Therefore, the objective of this research was to investigate the influence of partially replacing maltodextrin with cellulose-based carriers on the physical and aroma properties of obtained spray-dried asparagus powder. Various cellulose-based fibres were tested for their suitability to replace maltodextrin (MD), i.e. asparagus fibre (AF), citrus fibre (CF) and microcrystalline cellulose (MCC). The residual moisture content of the spray-dried powders was analysed The retention of asparagus key volatile 2-Methoxy-3-isopropyl pyrazine, which contributes to an earthy aroma of the asparagus [1], was studied in depth by headspace solid-phase microextraction (HS-SPME), followed by gas chromatography-mass spectrometry (GC–MS).

2. Materials and Methods

2.1. Sample preparation

Raw, fresh asparagus cut-offs (*Asparagus officinalis*) were kindly provided by Teboza BV (Helden, The Netherlands). Concentrated asparagus juice was prepared from asparagus cut-offs by Wageningen Food & Biobased Research (Wageningen, the Netherlands). The juice was concentrated using reverse osmosis by a factor of 5.6 to produce an asparagus concentrate with a final dry matter content of 21.7 % w/w. This concentrated juice was aliquoted into 40 ml samples and stored in the freezer at -20 °C before experimentation. After pressing out the juice, the remaining asparagus fibre was dried in a hot air oven (Heraeus, Hanau, Germany) at 60 °C for 24 hours. Subsequently, the fibre was milled into fine powder in the multi mill with the ZPS configuration (Hosokawa Alpine AG, Augsburg, Germany). Other carrier materials used were maltodextrin DE12 (Roquette Frères), citrus fibre (Herbacel® AQ® Plus Citrus) and microcrystalline cellulose (Sigma-Aldrich).

For every experiment, a new tube of the concentrated asparagus juice was thawed in the fridge at 4 °C for 18-20 hours. Feed solutions with 30 and 40 % w/w initial solids were prepared with only MD as a carrier or where MD was partially replaced with AF (1 or 3 % w/w), CF (1 or 3 % w/w) or MCC (3 or 10 % w/w). The carrier materials were added to the asparagus juice concentrate and then stirred at room temperature at 400 rpm for 1 hour. All samples were prepared in duplicate.

2.2. Spray drying experiment

Spray drying was performed using a Büchi Mini Spray Dryer B-290 (Büchi Labortechnik AG, Flawil, Switzerland). The aspirator rate applied was 90 %, which corresponded to approximately 35 m^3 air/h. The speed of the peristaltic pump was adjusted to 3 – 10.5 ml feed/min to reach the desired outlet air temperature. The inlet temperature (T_{in}) was set to 180 °C. In all cases, it was aimed to reach an outlet temperature (T_{out}) of 90 °C by adjusting the peristaltic pump.

2.3. Moisture content

Spray-dried powder (~ 0.5 g) was placed in a hot air oven (Heraeus, Hanau, Germany) to determine its moisture content. The powders were weighed before and after drying at 105 °C overnight, and the moisture content of the powder was calculated on a total weight basis (w/w). All measurements were carried out in triplicate.

2.4. Volatile analysis

We made a selection of spray-dried powders for the volatile compound analysis, i.e. MD, MD+3% AF, MD+3% AF and MD+3% MCC all with 30 % and 40 % initial solids content. The corresponding feed solutions were analysed as well. Samples were weighed based on 30 mg of asparagus solids and were transferred to 10-ml GC vials, which were subsequently stored at -80 °C until further analysis.

Volatile compounds in the headspace were measured using GC-MS and solid-phase microextraction (SPME) and were adsorbed onto a PDMS/DVB/CAR fibre (Supelco, PA, USA). Volatiles were thermally desorbed from the fibre and transferred onto the GC column (Zebron ZB-5MSplus, Phenomenex, the Netherlands). An Agilent GC7890A coupled to a 5975C quadrupole mass spectrometer was used. For calculating retention indices (RIs), a series of n-alkanes (C7-C21) was injected and analysed using the same method as for the samples and was part of the sample series.

GC-MS raw data were processed using an untargeted metabolomics workflow. MetAlign software was used for baseline correction (S/N >3) and alignment of the mass signals. The aligned mass signals were reconstructed to potential clusters (metabolites) using the MSClust tool. 2-Methoxy-3-isopropyl pyrazine was putatively identified (level 1) by matching the obtained mass spectra and experimental RIs with those in the commercial library (e.g. NIST17) and a reference analytical standard. The given level of identification follows the Metabolomics Standards Initiative [9].

3. Results and discussion

We analysed the physical properties and volatile profiles of the spray-dried powders. While performing the spray drying experiments, we found that partial replacement of MD could occur up to a maximum of 3 % in the case of AF and CF or 10 % in the case of MCC. Higher replacement levels led to clogging of the spray drying nozzle due to insolubility of the fibres and high viscosity of the feed solutions. The MCC particles in the feed solutions acted as inert particles and did not show swelling/water absorption compared to AF and CF, this may explain why more MCC could be added.

3.1. Moisture content

The residual moisture content of the powders was largely influenced by initial solids content (Table 1), where 40% resulted in lower moisture content compared to 30%. This difference could be explained with the higher concentration of asparagus solids, i.e. small sugars, on a dry weight basis since less carrier agent was added to the 30 % w/w samples. These small sugars increase the hygroscopicity of the powders and sugar-rich solutions may remain wet even after prolonged drying [10].

Table 1. Effects of carrier formulations and initial solids content on the residual moisture content of spray-dried asparagus powders

	Residual moisture content % (w/w)	
Sample name	**Initial solids 30 % (w/w)**	**Initial solids 40 % (w/w)**
MD	13.8 ± 0.0[ab]	6.7 ± 0.0[a]
MD+1% AF	13.9 ± 0.1[b]	8.5 ± 1.6[ab]
MD+3% AF	15.8 ± 0.8[ab]	8.7 ± 1.4[ab]
MD+1% CF	15.4 ± 1.7[ab]	7.4 ± 0.4[ab]
MD+3% CF	16.9 ± 1.6[ab]	9.3 ± 1.8[ab]
MD+3% MCC	14.8 ± 1.4[ab]	7.4 ± 0.6[ab]
MD+10% MCC	11.8 ± 0.2[ab*]	8.3 ± 0.1[b]

Carrier agents: maltodextrin (MD), asparagus fibre (AF), citrus fibre (CF) and microcrystalline cellulose (MCC). Moisture content is expressed as average with standard deviations (n=2). The values followed by different lowercase letters (a–b) within a column are significantly different at $p \leq 0.05$. *The MD+10% MCC with initial solids 30 % w/w powders were collected from the drying chamber instead of the collection vessel.

The residual moisture content of asparagus powders slightly increased as the replacement level of MD by other cellulose-based carriers increased at similar initial solids content. The powder MD+10% MCC with initial solids 30 % was collected from the drying chamber instead of the collection vessel, which explains the lower moisture content of this sample after being exposed to hot air for extended time.

3.2. Volatile analysis

The influence of carrier formulation on the retention of the asparagus aroma 2-Methoxy-3-isopropyl pyrazine was evaluated based on the measured relative abundances (Fig. 1A). We calculated the retention of 2-Methoxy-3-isopropyl pyrazine based on the ratio of the relative abundances in the feed solution (mix) and the corresponding spray-dried powder (Fig. 1B).

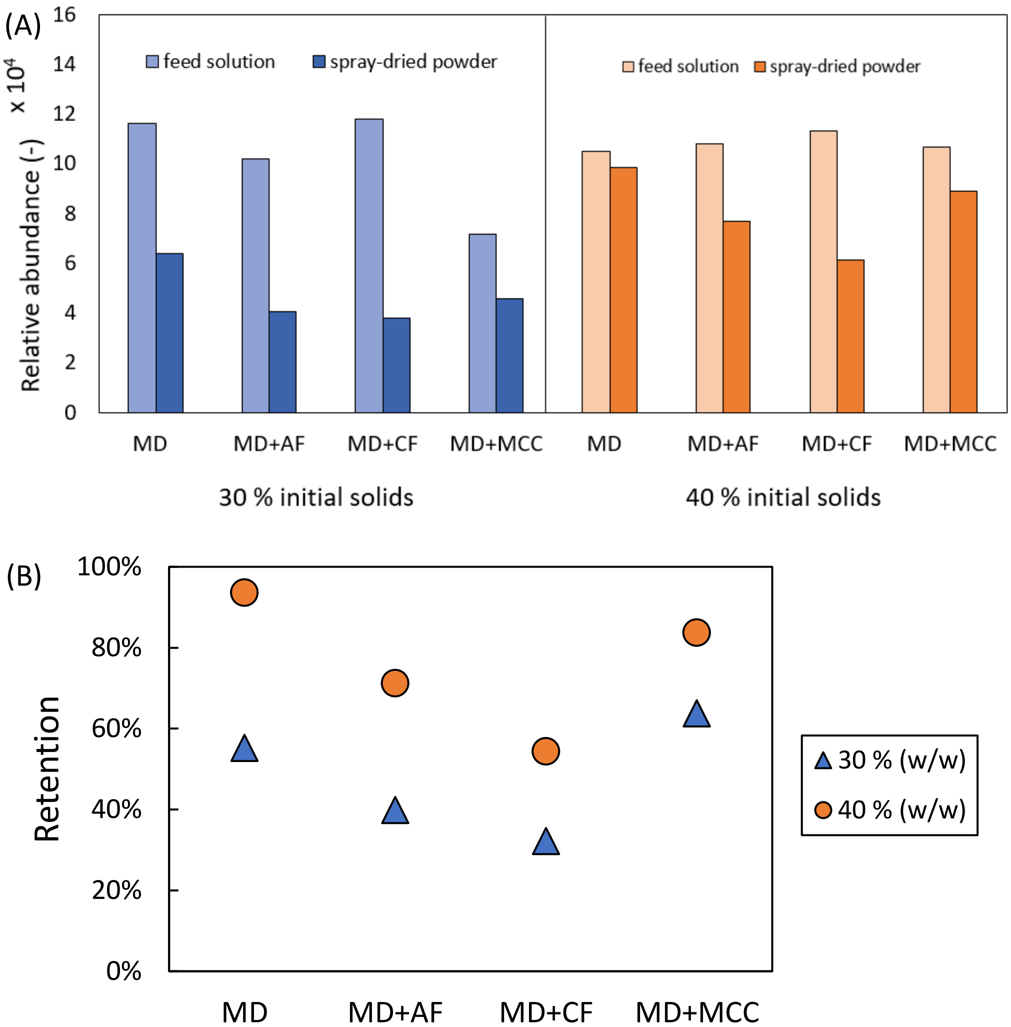

Fig. 1 (A) Relative abundances of 2-Methoxy-3-isopropyl pyrazine in the feed solution and spray-dried powder of asparagus juice concentrate with different carriers (i.e. maltodextrin or maltodextrin + 3 % w/w cellulose-based carrier) and initial solids content (30 or 40 % w/w). (B) Retention of 2-Methoxy-3-isopropyl pyrazine after spray drying. The retention was calculated based on the peak intensities of the mix before drying and the spray-dried powder. Carrier agents: maltodextrin (MD), asparagus fibre (AF), citrus fibre (CF) and microcrystalline cellulose (MCC).

We found that the retention of the pyrazine was higher in all samples with an initial solids content of 40 % w/w compared to 30 %. This might be explained by a shortened constant rate period and a fast formation of the semipermeable skin during drying of the 40 % w/w samples, which had a positive influence on the aroma encapsulation. Moreover, the partial replacement of MD with the cellulose-based carriers led to somewhat lower retention of the pyrazine with the addition of AF and MCC, as compared to the reference power with only MD. However, retention did not reduce more than 25 % compared to the reference powder and therefore was still considered acceptable. Nevertheless, the powders prepared with CF reduced the pyrazine retention significantly compared to only MD, i.e. 55 % to 32 % (30 % initial solids) and 94 % to 54 % (40 % initial solids). We conclude that the initial solids content has more impact on the volatile retention compared to the partial replacements tested in the study. In addition, CF may not be suitable as a carrier agent for the encapsulation of asparagus aromas.

4. Conclusions

In this study, we demonstrated that partial replacement of MD by cellulose-based carriers is possible for spray drying asparagus juice concentrate. The addition of cellulose-based carrier could be done up to a maximum of 3 % (w/w) in the case of AF and CF, thereby replacing 29 % of maltodextrin for 30 % w/w initial solids and 13 % of maltodextrin for 40 % w/w initial solids. For MCC, up to 10 % (w/w) was added, thereby replacing 94 % of maltodextrin for 30 % w/w initial solids and 42 % of maltodextrin for 40 % w/w initial solids. The residual moisture content of the spray-dried powders was influenced by the initial solids content of the feed, whereas the partial replacement did not significantly influence the moisture content.

The retention of the asparagus aroma 2-Methoxy-3-isopropyl pyrazine was largely determined by the initial solids content, where higher solids content improved the volatile retention. The replacement of MD with AF and MCC (all 3 % w/w in feed) resulted in slightly lower retention but was considered acceptable. We conclude that AF could potentially be used as a carrier agent to encapsulate aroma compounds in spray-dried asparagus powder, which enables complete usage of the asparagus.

Acknowledgements

This project is co-funded by TKI-E&I with the supplementary grant 'TKI- Toeslag' for Topconsortia for Knowledge and Innovation (TKI's) of the Ministry of Economic Affairs and Climate Policy.

5. References

[1] Pegiou, E.; Mumm, R.; Acharya, P.; de Vos, R. C. H.; Hall, R. D. Green and White Asparagus (*Asparagus Officinalis*): A Source of Developmental, Chemical and Urinary Intrigue. *Metabolites*, **2019**, *10* (1), 17. https://doi.org/10.3390/metabo10010017.

[2] Verma, A.; Vir Singh, S. Spray Drying of Fruit and Vegetable Juices-A Review. *Food Sci. Nutr.*, **2015**. https://doi.org/10.1080/10408398.2012.672939.

[3] Shishir, M. R. I.; Chen, W. Trends of Spray Drying: A Critical Review on Drying of Fruit and Vegetable Juices. *Trends Food Sci. Technol.*, **2017**, *65*, 49–67.

https://doi.org/10.1016/J.TIFS.2017.05.006.

[4] Coumans, W. J.; Kerkhof, P. J. A. M.; Ruin, S. Theoretical and Practical Aspects of Aroma Retention in Spray Drying and Freeze Drying. *Dry. Technol.*, **1994**, *12* (1–2), 99–149. https://doi.org/10.1080/07373939408959951.

[5] Madene, A.; Jacquot, M.; Scher, J.; Desobry, S. Flavour Encapsulation and Controlled Release - a Review. *Int. J. Food Sci. Technol.*, **2006**, *41* (1), 1–21. https://doi.org/10.1111/j.1365-2621.2005.00980.x.

[6] Siccama, J. W.; Pegiou, E.; Zhang, L.; Mumm, R.; Hall, R. D.; Boom, R. M. M.; Schutyser, M. A. I. Maltodextrin Improves Physical Properties and Volatile Compound Retention of Spray-Dried Asparagus Concentrate. *LWT - Food Sci. Technol.*, **2021**, *142* (February), 111058. https://doi.org/10.1016/j.lwt.2021.111058.

[7] Asioli, D.; Aschemann-Witzel, J.; Caputo, V.; Vecchio, R.; Annunziata, A.; Næs, T.; Varela, P. Making Sense of the “Clean Label” Trends: A Review of Consumer Food Choice Behavior and Discussion of Industry Implications. *Food Res. Int.*, **2017**, *99*, 58–71. https://doi.org/10.1016/j.foodres.2017.07.022.

[8] Ingredion. The Clean Label Guide to Europe https://emea.ingredion.com/Campaign/Clean-Label-Guide.html (accessed May 12, 2020).

[9] Sumner, L. W.; Amberg, A.; Barrett, D.; Beale, M. H.; Beger, R.; Daykin, C. A.; Fan, T. W. M.; Fiehn, O.; Goodacre, R.; Griffin, J. L.; et al. Proposed Minimum Reporting Standards for Chemical Analysis: Chemical Analysis Working Group (CAWG) Metabolomics Standards Initiative (MSI). *Metabolomics*, **2007**, *3* (3), 211–221. https://doi.org/10.1007/s11306-007-0082-2.

[10] Matsuno, R.; Adachi, S. Lipid Encapsulation Technology - Techniques and Applications to Food. *Trends Food Sci. Technol.*, **1993**, *4* (8), 256–261. https://doi.org/10.1016/0924-2244(93)90141-V.