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The effect of partial replacement of maltodextrin with vegetable fibres in spray-dried white asparagus powder on its physical and aroma properties

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ABSTRACT

Asparagus concentrate was spray-dried in different carrier formulations in which maltodextrin was partially replaced by cellulose-based carriers, i.e. asparagus fibre, citrus fibre or microcrystalline cellulose. This replacement was limited to a maximum level of 3% w/w for asparagus and citrus fibres, and 10% w/w for microcrystalline cellulose, due to fibre insolubility and increased viscosity of the feed. Powders obtained from feed solutions with an initial solids content of 40% w/w showed better physical properties and aroma retention than 30% w/w. Partial replacement of maltodextrin by cellulose-based carriers resulted in powders with similar physical properties as the control and did not detrimentally influence the aroma profiles as analyzed by headspace solid-phase microextraction and gas chromatography–mass spectrometry. This research shows that fibre obtained from asparagus waste streams could potentially be used as a carrier to produce spray-dried asparagus powder with retained key asparagus volatiles such as 2-methoxy-3-isopropyl pyrazine.

1. Introduction

White asparagus (*Asparagus officinalis*) is a popular vegetable consumed worldwide (Pegiou et al., 2019). When directly consumed, the cooked spears are considered a speciality/luxury vegetable and are appreciated for their distinct flavour profile which is perceived as being more subtle (slightly more bitter and less sweet) than its green counterpart. Flavour perception of food is determined by both its taste and aroma. The aromas that the human nose can perceive are numerous and their sensorial attributes can often be specifically described, e.g. earthy, pungent, fruity, cooked potato, etc. Taste attributes are generally limited to five modalities, i.e. sweetness, sourness, saltiness, bitterness and umami. Vegetables, including asparagus, often have rich aroma profiles which are also influenced by their processing history (e.g. fresh, canned, cooked, etc.). The aroma profile of *A. officinalis* has partly been studied in the past (Hoberg et al., 2008; Tressl, Bahri et al., 1977; Ulrich et al., 2001) and these studies have indicated that cooked asparagus shows a complex aroma profile consisting of volatile compounds from diverse chemical classes, including aldehydes, pyrazines and sulphur compounds. A general overview of the biochemistry, health benefits and flavour profile of asparagus can be found in Pegiou et al. (2019).

White asparagus is strongly seasonal and is only harvested for a limited period between March until June in Western Europe. However, after harvest, around one-third of the total material has to be discarded (Zhang et al., 2014). This significant waste stream partially consists of sub-standard, crooked and broken spears, but the main waste comprises the stem bases which are cut off to produce spears with the same desired length for delivery to the supermarkets. This waste stream could become a valuable resource for food ingredients. Valorisation of these materials could reduce the amount of agricultural waste while generating aroma-rich natural food products. For example, asparagus waste could potentially be dried into powder for use as a natural flavour ingredient for soups, sauces etc.

Nevertheless, drying of asparagus waste into powders with retained characteristic aroma profile could be challenging because volatile compounds are known to be rendered or lost during the processing of vegetables (Bangs and Reineccius, 1982; Madene et al., 2006). For example, 2-Methoxy-3-isopropyl pyrazine is a key odorant of white

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asparagus (Hoberg et al., 2008; Ulrich et al., 2001), which appears to be lost during thermal processing. Therefore, it is important to retain this compound as much as possible in dried asparagus powder. On the other hand, some volatile compounds may be formed during drying through processes such as thermal fragmentation, oxidative degradation of unsaturated fatty acids and initiation of Maillard reactions (Nijhuis et al., 1998; Tressl, Bahri et al., 1977). These newly-formed compounds may have a positive or negative (off-flavour) effect on the dried product. Specifically, key odorants that can be formed upon thermal processing of asparagus are sulphur-containing compounds such as Dimethyl sulphide (DMS) and 3-Methylthiopropanal, whose indirect precursor is the sulphur-containing amino acid Methionine (Tressl, Holzer et al., 1977; Tressl, Bahri et al., 1977; Ulrich et al., 2001). The formation of such compounds should be suppressed during drying so that they are preferably formed/synthesized only upon food preparation just before consumption. Therefore, different properties of the aroma compounds and their impact on the typical asparagus aroma profile should be taken into consideration when optimizing any drying strategy to make asparagus powder.

Different drying strategies to retain important aroma compounds in asparagus powder have been described recently (Siccama et al., 2019). Spray drying is a method commonly used in the food industry to preserve fruit and vegetable juices in powder form (Shishir and Chen, 2017; Verma and Vir Singh, 2015). Spray drying allows for the encapsulation of volatile compounds thanks to the formation of a semipermeable skin during the dehydration process (Coumans et al., 1994). Additionally, during spray drying, the atomised droplets remain relatively low in temperature even though high inlet air temperatures are applied. This makes spray drying also suitable for the drying of materials containing heat-sensitive compounds (Madene et al., 2006). However, similar to other vegetable and fruit juices, spray drying of asparagus juice is challenging because of the presence of high levels of small sugars. This leads to stickiness issues resulting in fouling during spray drying and as the glass transition temperature (T_g) of the product is below ambient temperature, this makes it problematic to obtain a glassy powder. Therefore, carrier agents are often added to increase the T_g of the feed and to avoid fouling (Verma and Vir Singh, 2015). In particular, maltodextrins with low dextrose equivalents (DE) are of interest since they have a high T_g and form an elastic skin upon drying. Siccama et al. (2021) demonstrated that the addition of maltodextrin DE12 improved the physical properties and volatile profile of spray-dried asparagus powder.

The addition of carrier agents to a food matrix entails that such ingredients must be added to the label of the final product. Nowadays, the transition towards producing "clean-label" foods that contain only natural ingredients is gaining ever greater attention among consumers (Asioli et al., 2017; Ingredion, 2014). For example, potato fibre is a more acceptable ingredient than maltodextrin (Ingredion, 2014). Spray-dried powder containing maltodextrin as a carrier agent does not meet the requirement of "clean-label" production. Therefore, it would be interesting to be able to at least partly replace maltodextrin with what are considered to be more authentic fibres, which are perceived as being more natural. Specifically, asparagus fibre might be used to (partially) replace maltodextrin as the carrier agent during the spray drying of asparagus juice, and so-doing, provide an extra opportunity to exploit the waste stream.

Here, various cellulose-based fibres have been tested for their suitability to replace maltodextrin. These were asparagus fibre (AF), citrus fibre (CF) and microcrystalline cellulose (MCC). Replacing maltodextrin by cellulose may be challenging due to their different physicochemical properties. Both maltodextrin and cellulose are carbohydrate polymers built up of glucose monomers, however, their conformation is different. The glucose-units in maltodextrin are coupled primarily through α-(1,4)-glucosidic linkages (Kennedy et al., 1995), whereas in cellulose through β-(1,4)-glucosidic linkages (BeMiller, 2019a). In addition, their degree of polymerization (DP) is significantly different. Maltodextrins are mixtures of polysaccharides derived from starch with DP values in the range of 2–20 and on average, have a DP $>$ 5 (BeMiller, 2019b) and are soluble in water. Cellulose is a large polysaccharide with an average DP $>$ 500 (BeMiller, 2019a), making it insoluble in water. The addition of insoluble cellulose-based material to the feed can lead to complications (e.g. increasing viscosity of the feed) for spray drying.

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. The hypothesis was that partial replacement would still provide a spray-dried asparagus powder of good quality. Specifically, it was expected that the addition of asparagus fibre may enrich the volatiles profile of the generated powders. For comparison, citrus and microcrystalline fibres were used as well to partly substitute maltodextrin. The quality of the spray-dried powders generated was evaluated on particle size distribution and particle morphology, as well as on the composition of the aroma compounds. The aroma profiles of the differently dried powders were compared by headspace solid-phase microextraction (HS-SPME), followed by gas chromatography-mass spectrometry (GC–MS).

2. Materials & methods

2.1. Materials

2.1.1. Asparagus material and vegetable fibres

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). Specifically, asparagus juice was pressed out of the cut-offs and was then centrifuged to remove any solids. 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 prior to 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 h. Subsequently, the fibre was milled into fine powder in the multi mill with the ZPS configuration (Hosokawa Alpine AG, Augsburg, Germany), with an impact mill speed of 8000 rpm, the air flow at 45 m^3/h and the classifier wheel speed at 3000 rpm. The particle size distribution of the milled asparagus fibre is reported in the Supplementary data (Fig. S1) where the average particle size (D_{50}) was 86 μm. The asparagus fibre had a moisture content of 4.4 ± 0.4% w/w and contained 74.8% total dietary fibre (i.e. 37.6% cellulose, 18.2% hemicellulose and 4.2% lignin, all on dry basis). Citrus fibre (Herbacel® AQ® Plus Citrus) was kindly provided by Unilever (Unilever BV, Wageningen, The Netherlands). The moisture content of citrus fibre was 6.2 ± 0.2% w/w and contained 85.6% total dietary fibre (i.e. 63.9% cellulose, 10.0% hemicellulose and 1.5% lignin, all on dry basis). The total dietary fibre was determined with the K-TDFR assay from Megazyme (Megazyme International Ireland, Wicklow, Ireland). The cellulose, hemicellulose and lignin contents were determined with the Neutral Detergent Fibre (NDF) method performed by Eurofins Agro (Eurofins Agro, Wageningen, The Netherlands). Microcrystalline cellulose (cellulose powder, 20 μm, 310697) was purchased from Sigma–Aldrich (St. Louis, MO, USA). Maltodextrin DE 12 was purchased from Roquette (Roquette Frères, Lestrem, France).

2.1.2. Analytical standards and chemicals

A series of *n*-Alkanes (C7-C21) was prepared from a set of stock solutions of the specific alkanes which had been purchased from Sigma-Aldrich (Milwaukee, WI, USA). The analytical standards (96–99% purity) that were used for the metabolite identification were Dimethyl sulphide, 3-Methylthiopropanal, Hexanal, 1-Hexanol, 2,3-Pentanedione, 2-Methylbutanol, 1-Octen-3-ol, 2-Pentylfuran, and 2-Methoxy-3-isopropyl pyrazine. All were purchased from Sigma-Aldrich. Methanol

(Biosolve BV, The Netherlands) and was used for the preparation of the standard solutions. Calcium chloride ($CaCl_2$) and Ethylenediaminetetraacetic acid (EDTA) were also purchased from Sigma-Aldrich. Ultrapure water (Milli-Q™ Reference Ultrapure Water Purification System) was used for the preparation of the EDTA solution.

2.2. Sample preparation for spray drying

For every experiment, a new tube of the concentrated asparagus juice was first taken from the freezer and the concentrate was thawed in the fridge at 4 °C for 18–20 h. Feed solutions for spray drying experiments were prepared according to Table 1. Specifically, feed solutions with an initial solids content of 30% or 40% w/w were prepared. In some samples, maltodextrin was partially replaced by cellulose-based materials, i.e. asparagus fibre (1 or 3% w/w), citrus fibre (1 or 3%) and MCC (3 or 10% w/w). The replacement level was determined based on measured viscosity of the feed solutions (Supplementary data, Fig. S2). The carrier materials were added to the asparagus concentrate and then stirred at room temperature at 400 rpm for 1 h. All samples were prepared in duplicate.

2.3. 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 an air flow of 35 m^3/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, which is within the range recommended by Reineccius (2004). In all cases, it was aimed to adjust the pump to reach an outlet temperature (T_{out}) of 90 °C to provide sufficient drying capacity and to avoid fouling in the drying chamber. However, in some cases even at the maximal pump speed, the outlet temperature exceeded 90 °C (Supplementary data, Table S1). Moreover, for the samples with MD + MCC 30–10%, we were not able to collect sufficient material from the collection vessel, instead, we collected the powder from the drying chamber.

2.4. 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.

2.5. Particle size distribution and morphology

The particle size distribution of the spray-dried samples was measured using a Mastersizer 3000 analyser (Malvern Inc, Worcestershire, UK). The laser diffraction measurement was performed using the dry powder disperser Aero S. The morphology of six powder samples, i.e. pure MCC, MD 40%, MD + AF 40–3%, MD + MCC 40–3%, MD + MCC 40–10% and CF + MD 40–3%, was investigated with an FEI Magellan 400 FESEM (FEI, Thermo Fisher Scientific, Hillsboro, United States of America) at the Wageningen Electron Microscopy Centre (Wageningen University and Research, Wageningen, the Netherlands). The MD + MCC 40–10% sample was first dried under vacuum at room temperature to remove the excess water present. All samples were fixed on the sample holder by carbon adhesive tabs and sputter-coated with 12 nm of tungsten. SEM images were taken at 2 kV.

2.6. Sample preparation for volatiles analysis

Volatile compound analysis was performed on the pure carriers, four samples of concentrated asparagus concentrate, all spray-dried powders and quality control samples consisting of a mix of the 28 spray-dried powders. Samples were weighed based on Eq. (1), so that all the samples contained 30 mg of asparagus solids. The 10-ml GC glass vials containing the samples were subsequently stored at −80 °C until further analysis.

$$mass = \frac{30}{X_s X_{a(db)}} \quad (1)$$

where X_s (kg solids/kg total) is the total solids content of the sample determined via moisture content analysis. $X_{a(db)}$ (kg asparagus solids/kg solids) is the fraction of asparagus solids in the solids content of the sample, which can be derived from the amount of asparagus solids from Table 1.

Prior to analysis, 0.73 g $CaCl_2$ was added to each sample. Next, a predefined volume of EDTA solution was added to all vials to obtain a total liquid volume of 1 ml per vial with a final concentration of 50 mM EDTA and 5 M $CaCl_2$ (saturated solution). The addition of $CaCl_2$ and EDTA was needed to deactivate any enzyme activity and to help drive the volatiles into the headspace (Verhoeven et al., 2011). All samples were thoroughly mixed using a vortex, followed by 15 min in an ultrasound bath and kept subsequently on ice until analysis.

2.7. Volatiles analysis

Volatile compounds in the headspace were measured using GC–MS and solid-phase microextraction (SPME) and were adsorbed onto a PDMS/DVB/CAR (Polydimethylsiloxane/Divinylbenzene/Carboxen) 50/30 μm diameter, 1 cm length (Supelco, PA, USA) fibre using an MPS2

Table 1
Preparation scheme of samples with maltodextrin (MD), asparagus fibre (AF), citrus fibre (CF) and microcrystalline cellulose (MCC) as carrier agents.

Sample name	Total solids (% w/w)	Asparagus solids in juice (% w/w)	Maltodextrin (% w/w)	Asparagus fibre (% w/w)	Citrus fibre (% w/w)	Microcrystalline cellulose (% w/w)
MD 30%	30	19	11	–	–	–
MD 40%	40	17	23	–	–	–
MD + AF 30–1%	30	19	10	1	–	–
MD + AF 30–3%	30	19	8	3	–	
MD + AF 40–1%	40	17	22	1	–	
MD + AF 40–3%	40	17	20	3	–	
MD + CF 30–1%	30	19	10	–	1	–
MD + CF 30–3%	30	19	8	–	3	–
MD + CF 40–1%	40	17	22	–	1	–
MD + CF 40–3%	40	17	20	–	3	–
MD + MCC 30–3%	30	19	8	–	–	3
MD + MCC 30–10%	30	19	1	–	–	10
MD + MCC 40–3%	40	17	20	–	–	3
MD + MCC 40–10%	40	17	13	–	–	10

sampling robot (Gerstel, Germany). Samples were pre-conditioned at 50 °C for 15 min under agitation (300 rpm). Volatiles were then trapped by exposing the fibre to the headspace of the vial for 15 min at 50 °C without agitation. Volatiles were thermally desorbed by inserting the fibre into the injector containing an empty glass liner (1 mm ID) (CIS4, Gerstel, Germany) at 250 °C for 2 min with a helium flow of 1 ml/min onto the GC column, in splitless mode. An Agilent GC7890A coupled to a 5975C quadrupole mass spectrometer was used. A Zebron ZB-5MSplus (5% phenyl and 95% dimethylarylene siloxane) column (Phenomenex, the Netherlands) with dimensions 30 m × 0.25 mm id × 1 μm film thickness was used for chromatographic separation. The GC oven temperature was programmed to start at 45 °C for 2 min, then increased at a rate of 8 °C/min to 250 °C, then at a rate of 15 °C/min to 280 °C and then held at 280 °C for 3 min. The carrier gas was helium, at a constant flow rate of 1 ml/min. The column effluent was ionised by electron impact at 70 eV, in the scan range *m/z* 33–330. The MS interface temperature was set to 280 °C. 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 as part of the same sample series. RIs were calculated using a third-degree equation.

2.7.1. Data (pre-)processing and analysis

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. Metabolites were putatively identified by matching the obtained mass spectra and experimental RIs with those in commercial (e.g. NIST17) and in-house libraries. The given level of identification follows the Metabolomics Standards Initiative (Sumner et al., 2007). Prior to statistical analysis, zero values in the processed data were substituted with randomised values of ca. 10% of the detection limit as this was determined in MetAlign during pre-processing.

2.8. Statistical analysis

All spray drying experiments were conducted at least in duplicate and results are presented as mean ± standard deviations. Significant differences in residual moisture contents of the powders were assessed by One-way analysis of variance (ANOVA) and Tukey's Honestly Significant Differences (HSD) post hoc test, with a $p \leq 0.05$ meaning the difference between groups was statistically significant. In the case of unequal variances, the Games-Howell post hoc test was used. The statistical analyses were performed using SPSS Statistics (SPSS 25; IBM, USA).

Processed GC–MS data were subjected to Principal Component Analysis (PCA) and Hierarchical Clustering Analysis (HCA), after log10 transformation and Pareto-scaling using SIMCA 15.0.2. software (Umetrics, Sartorius Stedim Data Analytics AB, Umeå, Sweden). Additional uni- and multi-variate statistical analyses were performed using RStudio with R version 4.0.3 (2020–10-10). Graphs were also produced using Microsoft Office Excel.

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. In addition, the replacement of MD by AF or CF was limited by the increased viscosity of the feed solutions (Supplementary data, Fig. S2) while partial replacement with MCC did not increase the viscosity of the feed solutions. The MCC particles 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. Physical properties

3.1.1. Moisture content

Residual moisture contents in spray-dried asparagus powders were measured (Table 2). It was found that the initial solids content largely influenced the residual moisture content in the powders. Solutions with an initial total solids content of 40% w/w resulted in powders with lower moisture contents as compared to powders obtained from the 30% w/w initial solids. This was, however, not a direct effect of the initial solids content, because we increased the feed flow rate of the 40% w/w feed solutions to obtain the same water evaporation rate and thus equal outlet temperature (i.e. 90 °C) as the 30% w/w solutions. We might explain the difference in moisture content by the difference in carrier formulations (Table 1). The powders prepared with 30% w/w initial solids contained more asparagus solids on a dry weight basis (63%) as compared to the powders prepared with 40% w/w solids (43%). Because asparagus solids contain a large amount of small sugars (±67% dry basis), the 30% w/w samples have a more hygroscopic nature compared to the 40% w/w samples. Drying of hygroscopic solutions, e.g. low molecular weight saccharides, is often hindered by a rapid decrease in drying rate at an intermediate moisture content. Consequently, these solutions remain wet even after prolonged drying (Matsuno and Adachi, 1993). Furthermore, the powders prepared using 30% w/w solids may also have absorbed moisture from the environment after they were taken from the collection vessel due to their high hygroscopicity (King et al., 1982; Li et al., 2018).

The residual moisture content of asparagus powders slightly increased as the replacement level of MD by other cellulose-based carriers increased, when the initial total solids content of the feed is kept the same (Table 2). The partial replacement of MD may have influenced the drying behaviour of the feed solutions. It is hypothesized that the (partially) insoluble fibres, i.e. AF, CF and MCC, are encapsulated by maltodextrin. Therefore, it is suggested that maltodextrin and asparagus solids dominated the drying behaviour. Do et al. (2018) found that after spray drying mulberry juice with MD, gum arabic and MCC, the powder contained the extract and MCC as separate particles. The moisture contents in their final products were significantly reduced from 4.54 to 3.20% after increasing the MCC concentrations from 0% to 2%. However, this positive effect of adding MCC to the feed solution on the residual moisture in mulberry powder was not observed in this study. This might be explained by the already high MCC concentration used in the current work. Moreover, the powder prepared from MD + MCC 30–10% solution 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.

Table 2
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	13.76 ± 0.01^{ab}	6.73 ± 0.03^{a}
MD + 1% AF	13.87 ± 0.13^{b}	8.51 ± 1.62^{ab}
MD + 3% AF	15.81 ± 0.79^{ab}	8.65 ± 1.38^{ab}
MD + 1% CF	15.41 ± 1.74^{ab}	7.39 ± 0.39^{ab}
MD + 3% CF	16.87 ± 1.61^{ab}	9.27 ± 1.78^{ab}
MD + 3% MCC	14.82 ± 1.40^{ab}	7.37 ± 0.64^{ab}
MD + 10% MCC	11.79 ± 0.18^{a}	8.32 ± 0.08^{b}

Note: 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 samples were collected from the drying chamber instead of the collection vessel.

3.1.2. Particle size distribution

The particle size distributions of the spray-dried powders prepared with only MD, MD + 3% AF, MD + 3% CF and MD + 10% MCC are plotted in Fig. 1. All powders prepared with 40% w/w initial solids content had one main peak located around 10 μm (Fig. 1A). For the powder obtained from MD + 10% MCC, this peak is shifted towards a larger particle size. The powders with 30% w/w initial solids content have a broad size distribution (Fig. 1B). The peaks located at sizes above 100 μm indicate undesired particle agglomeration. This agglomeration could be explained by increasing stickiness due to the higher residual moisture content and lower glass transition temperature (unpublished data) of these powders. The first peak of MD 30% has a smaller particle size compared to the peak of MD 40%, 8.7 μm and 14.5 μm respectively. This increase in particle size for the higher initial solids content is probably related to the increase in viscosity of the feed resulting in larger droplets upon atomization (Santos et al., 2018).

For the 30% w/w initial solids samples, we observe slightly different results for MD + AF 30–3% and MD + CF 30–3% (Fig. 1B). Less agglomeration and overall smaller particle size were observed for CF samples compared to AF. It is worth noting that the particle size distribution of the pure carrier materials AF and CF cannot help to explain the different particle size distributions (Supplementary data, Fig. S1) as the peaks do not overlap with the spray-dried powders.

For the 30% w/w initial solids, we observe a different size distribution for MD + MCC 30–3% and MD + MCC 30–10% (Fig. 1B). The distribution of MD + MCC 30–3% contains two peaks, and is similar to MD + AF 30–3%, whereas MD + MCC 30–10% shows one peak around 60 μm. The particle size distribution was most likely affected by the collection process, as the MD + MCC 30–10% powder was collected from the drying chamber instead of the collection vessel beneath the cyclone. Jedlińska et al. (2018) observed similar differences in particle size for powders collected from the drying chamber and the cyclone container. They suggested that the smaller particles are more easily blown out with the outlet air and thus could be mostly collected from the cyclone. The bigger particles, however, have larger inertia and therefore collide with the chamber wall and deposit there.

We can conclude that the physical properties of the powders prepared with 40% w/w initial solids are of better quality compared to 30% w/w. The 30% w/w powders have a higher residual moisture content, which makes the powder sticky and this is also related to the undesired agglomeration that is observed.

3.1.3. Particle morphology

Scanning electron microscopy pictures of the spray-dried powders prepared with 40% w/w initial solids are shown (Fig. 2) as these were not agglomerated compared to the 30% w/w initial solids powders. The MD 40% powder contained both dented (green box) and smooth (blue box) particles (Fig. 2A). The dented particles are characteristic for spray-dried maltodextrin DE12 (Both et al., 2018; Siemons, Politiek et al., 2020). The appearance of smooth particles may be linked to the feed

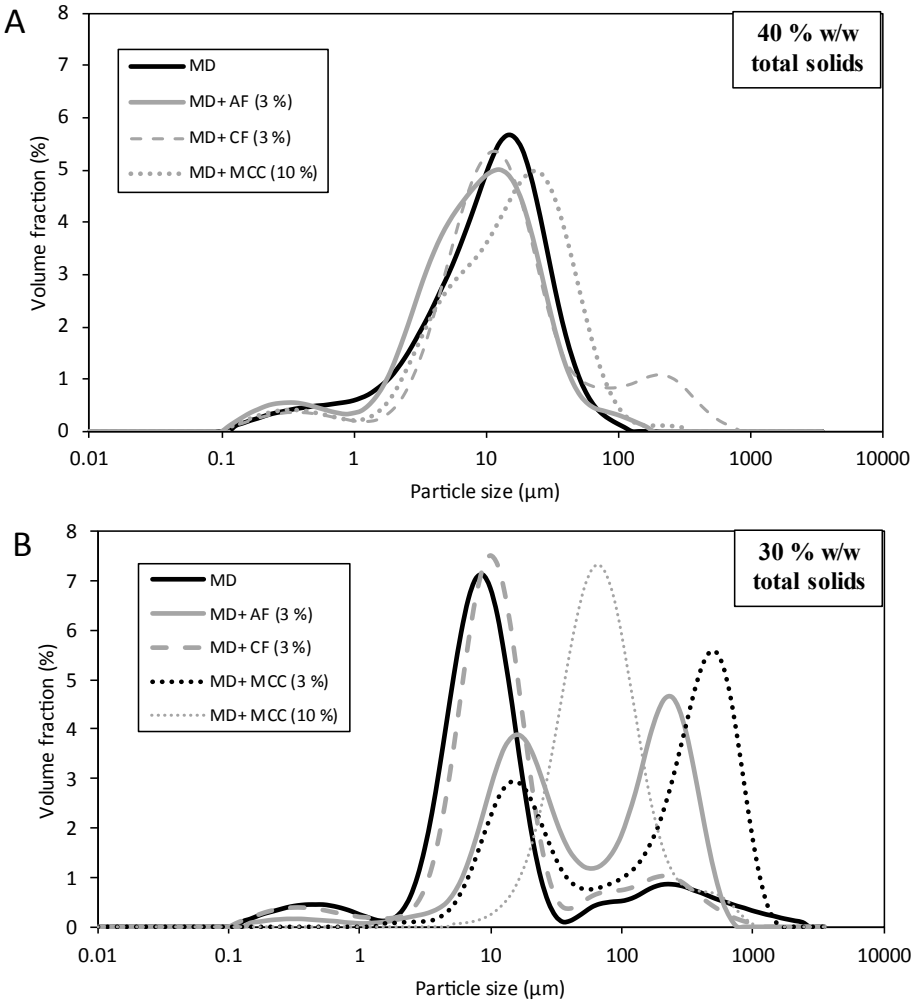

Fig. 1. (A) Particle size distribution of spray-dried asparagus powders prepared with different carrier agents and initial total solids content of 40% w/w. (B) Particle size distribution of spray-dried asparagus powders prepared with different carrier agents and initial total solids content of 30% w/w. Carrier agents: maltodextrin (MD), asparagus fibre (AF), citrus fibre (CF) and microcrystalline cellulose (MCC).

composition but could also be explained by the drying process. Smooth particles have previously been observed by Siemons, Vaessen et al. (2020) for drying of low molecular weight compounds xylose and trehalose. Besides, during spray drying, there is a broad droplet size distribution and each droplet experiences its own drying trajectory, which may influence the final particle morphology (Both et al., 2020). The morphologies of MD + AF 40–3% and MD + CF 40–3% powders were very similar to that of the MD 40% sample. For all three samples, individual particles with both smooth and wrinkled surfaces were observed. For MD + AF 40–3% few elongated particles were detected (Supplementary data, Fig. S3B). For the powders that were prepared with MCC, next to the round-shaped particles, elongated particles were observed (Fig. 2D,E). The elongated particles were probably MCC as they had similar size and shape as the pure MCC particles before drying (Fig. 2F) and more of these particles are observed with an increase in MCC concentration. We assume these particles are inert fibres and were thus not altered by the spray drying process. However, the surface of the spray-dried MCC particles was smooth whereas pure MCC particles have a rough surface. It is therefore hypothesized that maltodextrin encapsulated the microcrystalline cellulose particles and therefore formed this smooth surface. Next to the elongated partciles and some dented particles, perfect small spheres are observed for MD + MCC 40–10% (Fig. 2E). These small spheres may be related to the high concentration of small sugars in the soluble fraction. The addition of 10% microcrystalline cellulose reduced the maltodextrin concentration in the feed solution from 23 to 13% w/w (Table 1). Since microcrystalline cellulose is

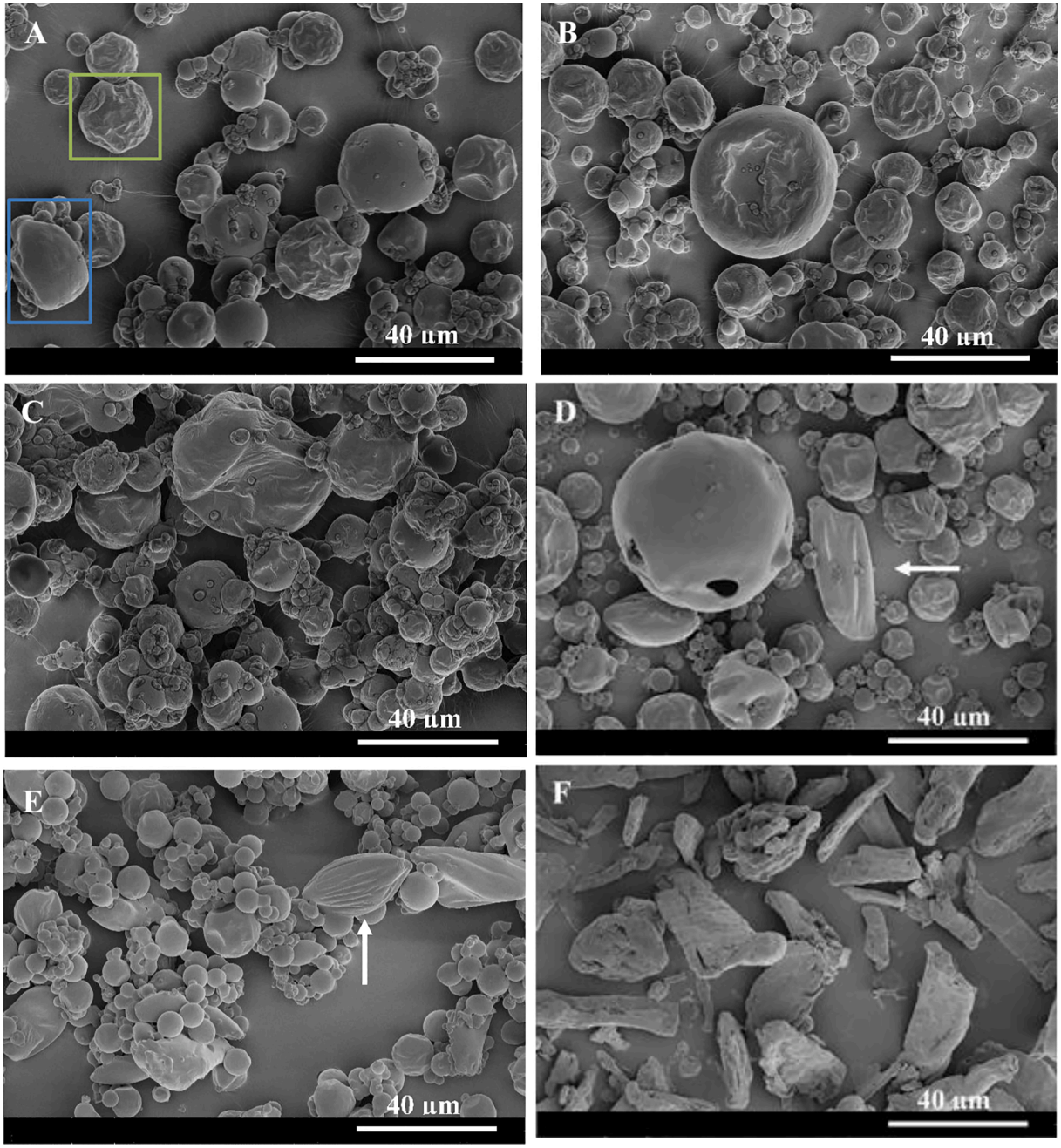

Fig. 2. Scanning electron microscopy images of spray-dried powders at a magnification of 2000x with (A): MD 40% (with the green box indicating a wrinkled particle, the blue box indicating a smooth particle), (B): MD + AF 40–3%, (C): MD + CF 40–3%, (D): MD + MCC 40–3%, (E): MD + MCC 40–10%, and (F): pure microcrystalline cellulose before spray drying. The arrows indicate the elongated particles. (For the list of abbreviations see Table 1.) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

completely insoluble, the soluble solids fraction in the feed solution therefore became richer in asparagus solids, i.e. small sugars, compared to the reference MD 40%. This large fraction of small sugars may have contributed to the formation of perfect small spheres and reduced the number of dented particles.

3.2. Volatiles analysis

3.2.1. Untargeted analysis of volatiles and comparison of spray-dried powders

We aimed to inspect the influence of the total solids content used for the spray drying of asparagus concentrate on the volatiles profile of the

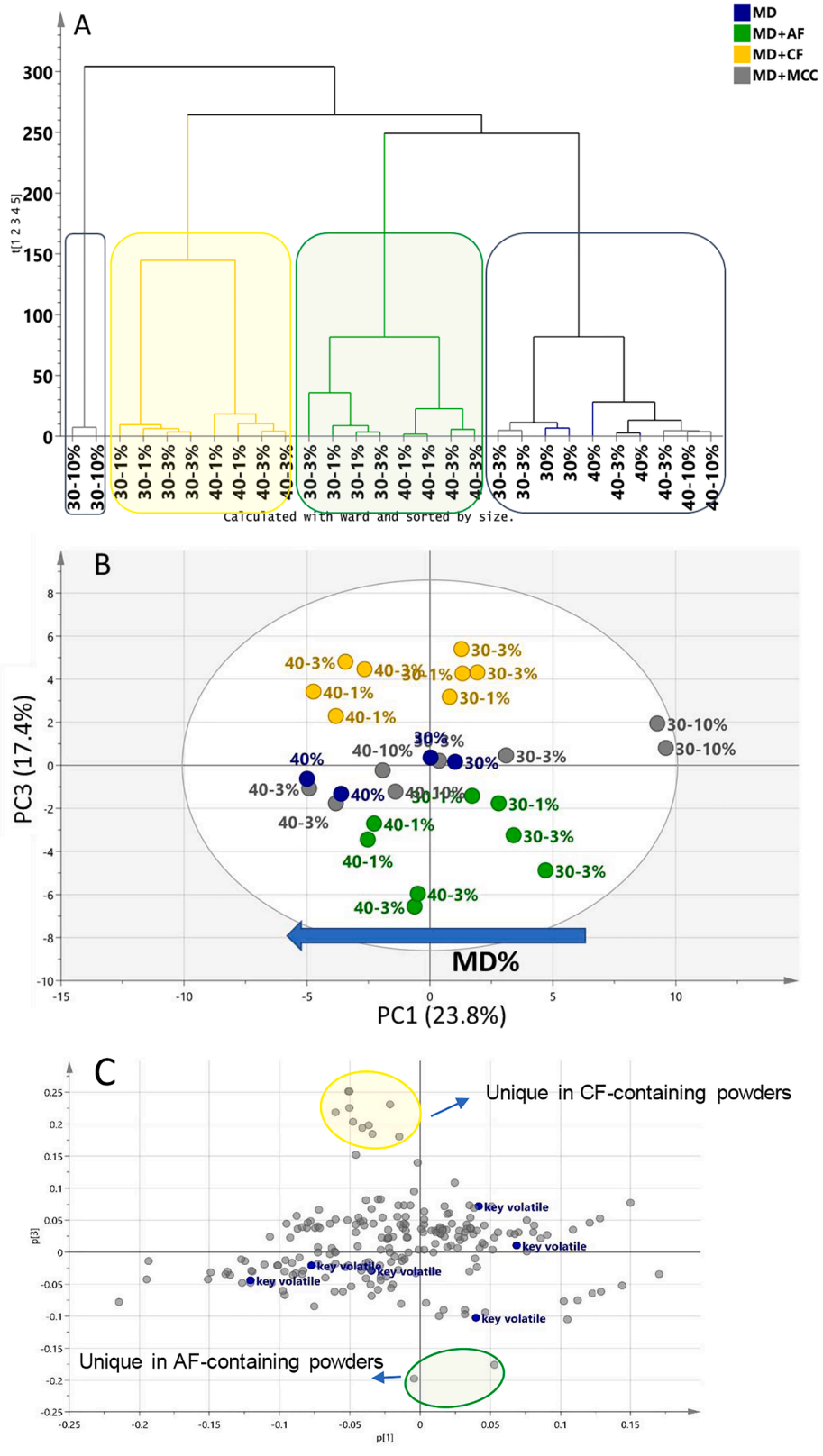

Fig. 3. Unsupervised multivariate statistics on the 221 volatiles detected in 28 spray-dried asparagus powders with different carrier agents (MD, MD + AF, MD + CF and MD + MCC) and carrier concentrations (total solids content 30% or 40% w/w, in case of MD + AF, MD + CF and MD + MCC the percentage of the MD that was replaced by AF/CF/MCC is indicated: 1/3/10%). Data have been log10-transformed and Pareto-scaled prior to analysis. (A): HCA Dendrogram showing the clustering of the spray-dried asparagus powders. HCA was calculated with Ward's method. Boxes indicate the formed clusters, based on the carrier type. (B): PCA score plot of the first and third PC with the % of explained variance given in the parenthesis. The ellipse represents the 95% confidence interval from the Hotelling T2 function. (C): Loading plot from the PCA plot of (B). Asparagus key volatiles based on (Hoberg et al., 2008; Ulrich et al., 2001) that were detected are indicated in the plot. (For the list of abbreviations see Table 1.)

obtained spray-dried powders. Moreover, the effect of partially replacing MD by cellulose-based carriers has also been investigated. HS–SPME was used for the extraction of the volatiles prior to GC–MS analysis as with a headspace extraction method, the volatiles profile should closely resemble the aroma profile that the potential consumer of the final product should experience. An untargeted metabolomics approach was followed to obtain a comprehensive overview of the similarities and variations between the volatile profiles of the different spray-dried asparagus powders. All spray-dried powder samples contained the same amount of asparagus solids to enable the direct comparison of the samples. The pure carriers were also analysed separately to verify potential unique compounds originating from the carriers themselves during spray drying.

A total of 244 compounds was detected. After filtering out all system artefacts, the remaining 221 volatile compounds were further studied as described in section 2.7.1 for untargeted data analysis. Identification of volatiles was restricted to those compounds that have been reported earlier in cooked asparagus materials and which have been highlighted as being key asparagus volatiles with significant sensory impact (Hoberg et al., 2008; Ulrich et al., 2001). Such compounds included e.g. hexanal, 3-methylthiopropanal, 2-methoxy-3-isopropyl pyrazine and 1-octen-3-ol. Moreover, some other compounds (e.g. the benzene derivative 1,2-dimethoxybenzene and the ester 2-butanol, 3-methyl, acetate) were further investigated as they could also be of particular relevance to the final flavour quality of the asparagus powder as proposed by Tressl, Bahri et al. (1977).

Initially, to investigate the influence of the various carriers on the overall volatiles profile, the structure of the dataset of the 221 volatiles in the 28 spray-dried powders was explored and analysed using unsupervised multivariate statistics. After performing hierarchical clustering using Ward's method (Ward and Joe, 1963), the effect of both the total solids content and the type of the cellulose-based carrier replacing MD was evident. In the dendrogram in Fig. 3A, the clustering of the analysed spray-dried powders is based on the carrier composition. Powders with CF form one cluster (yellow box), the AF-containing powders form a second cluster (green box) and the powders with MD and MD + MCC form a third cluster (black box). This implies that there is only a minor effect of partially substituting MD with MCC, as well as the potential contribution of unique compounds from CF and AF matrix to the volatile profile. In the same dendrogram, within each cluster, the powders with the same total solids content also cluster close together, implying that their profiles are similar. The two powders with 30% w/w total solids content, and 10% MCC, are clustered separately and distant from all other samples. This highlights the effect of the higher concentrations of MD on the volatiles profile, as these two samples contain the least MD% (Table 1). This deviation of the volatile profiles between the powders with 10% MCC and the rest can also be linked to the fact that the MD + MCC 30–10% powders had to be collected from the drying chamber instead of the collection vessel. This might influence the volatiles profile, due to the extra heat load, but this requires further investigation.

As most information can be extracted by looking at variation, a PCA was also performed on the spray-dried powder samples (Fig. 3B). The first and third principal components (PCs) are plotted, as these summarized the most valuable information in the dataset. In this PCA score plot, there is a clear effect of the total solids content as all powders with a total solids content of 30% w/w are located on the right side (positive PC1 values) and the powders with 40% w/w total solids content are located on the left side of the plot (negative PC1 values). Furthermore, the variation summarized in the first PC can be also explained more specifically by the concentration of maltodextrin (MD%). The blue arrow in the plot indicates that the samples are distributed across PC1 (x-axis) based on the MD%. The samples on the right had lower MD content, while those on the left of the plot had the higher MD%. In the same score plot, there is a trend along PC3 (y-axis), based on the type of the carrier replacing MD. Here, the samples containing AF locate in the lower part of the plot (negative PC3 values) while those with CF locate at the top of the PCA plot (positive PC3 values). This separation of the samples based on the type of the carrier, with the powders containing only MD or MD + MCC forming a group in the middle of the plot, implies that the asparagus and citrus fibres themselves may have contributed to the volatiles profile through their own aroma compounds. The pure carriers used for the spray drying of the asparagus concentrate were therefore also analysed. A subset of the raw total ion current (TIC) chromatograms is shown in Supplementary Fig. S4 and compared to the TIC chromatogram of the asparagus concentrate. Unique peaks correspond to compounds that originate from the AF and CF and were detected only in the AF- or CF- containing spray-dried powders, respectively. In the loading plot in Fig. 3C, the CF- and AF- specific compounds are indicated. The CF-specific compounds were seen to include a few monoterpenoids (e.g. p-mentha-1,3,8-triene) which could potentially impact the typical asparagus flavour profile. The AF-specific compounds were undecanenitrile and 5-methyl-2-phenyl-2-hexenal. None of these compounds have been previously reported in asparagus materials but they might have been formed here during the preparation process of the AF. The known key asparagus odorants (Ulrich et al., 2001) which are indicated in blue in the same plot (Fig. 3C: key volatile), were present in all spray-dried powders, but at different levels. In the future, sensory and olfactometry studies shall be used to evaluate whether these compounds have any impact on the sensory attributes of the powders. Both positive as well as negative (off flavour) attributes need to be evaluated. However, we anticipate that the latter is unlikely to be a major issue as such compounds are usually formed as a result of microbial deterioration, lipid oxidation, Maillard reactions and improper storage, all of which are unlikely in our process.

One- and two- way ANOVA showed that for most of the volatiles, the differences between the spray-dried powders were significant (p value < 0.05) both depending on the different type of carrier (Supplementary Fig. S5A) and different carrier concentrations (Supplementary Fig. S5B). The volcano plots in Supplementary Fig. S5C and S5D show the volatiles that were present at significantly different levels in the powders where MD was partially replaced by AF or CF (Supplementary Fig. S5C) and AF or MCC (Supplementary Fig. S5D) with a fold-change cut-off of 1.5. In both cases, volatiles that were significantly more abundant in the powders containing AF comprised of a number of alcohols (e.g. 1-pentanol and 3-methyl-1-butanol), some branched alkanes and compounds that could not be identified. Important asparagus odorants according to Hoberg et al. (2008) and Ulrich et al. (2001), such as 2-methoxy-3-isopropyl pyrazine, 3-methylthiopropanal and DMS were significantly more abundant in the powders containing AF compared to the ones containing CF (indicated with arrows in Supplementary Fig. S5C). Powders containing CF were characterized by especially higher amounts of some aldehydes and ketones (e.g. hexanal, heptanal, 2-butanone and 2-heptanone). In the case of powders containing MCC as carrier we found two aldehydes, one of which was pentanal, a few non-identified compounds and 2-methoxy-3-isopropyl pyrazine being more abundant compared to the AF – containing powders. Given the importance that the known asparagus odorants and the positive aroma attributes that the mentioned alcohols can have for the aroma profile of asparagus products, we suggest that using AF as a carrier for the spray drying of asparagus concentrate may have a positive impact on the aroma profile of the obtained powder.

The relative abundances of a number of identified volatiles are presented in Supplementary Fig. S6, and in most cases these are higher in the spray-dried powders with highest total solids content. In the case of compounds derived from asparagus but which are formed during processing due to applied high temperatures (e.g. 3-methylthiopropanal), peak intensities were significantly higher in the samples containing AF in the carrier, and specifically with the higher level of AF (3%). This implies an extra contribution of AF as a carrier in the volatiles profile of the generated spray-dried powders, and eventually the aroma profile of the final product. Moreover, this verifies that indeed high temperatures can lead to the formation of these sulphur-containing asparagus key

volatiles, given the drying method applied to the AF. Potential optimization of the processing of the raw asparagus fibre can lead to even richer aroma profile of the spray-dried powders with AF in the carrier.

3.2.2. Analysis and retention of selected volatiles in spray-dried powders

The influence of the spray drying on the volatiles and their retention in the powder is dependent on several variables including the drying conditions, the solids content and the relative volatility of the molecules (Bangs and Reineccius, 1982; Coumans et al., 1994; Jafari et al., 2008;

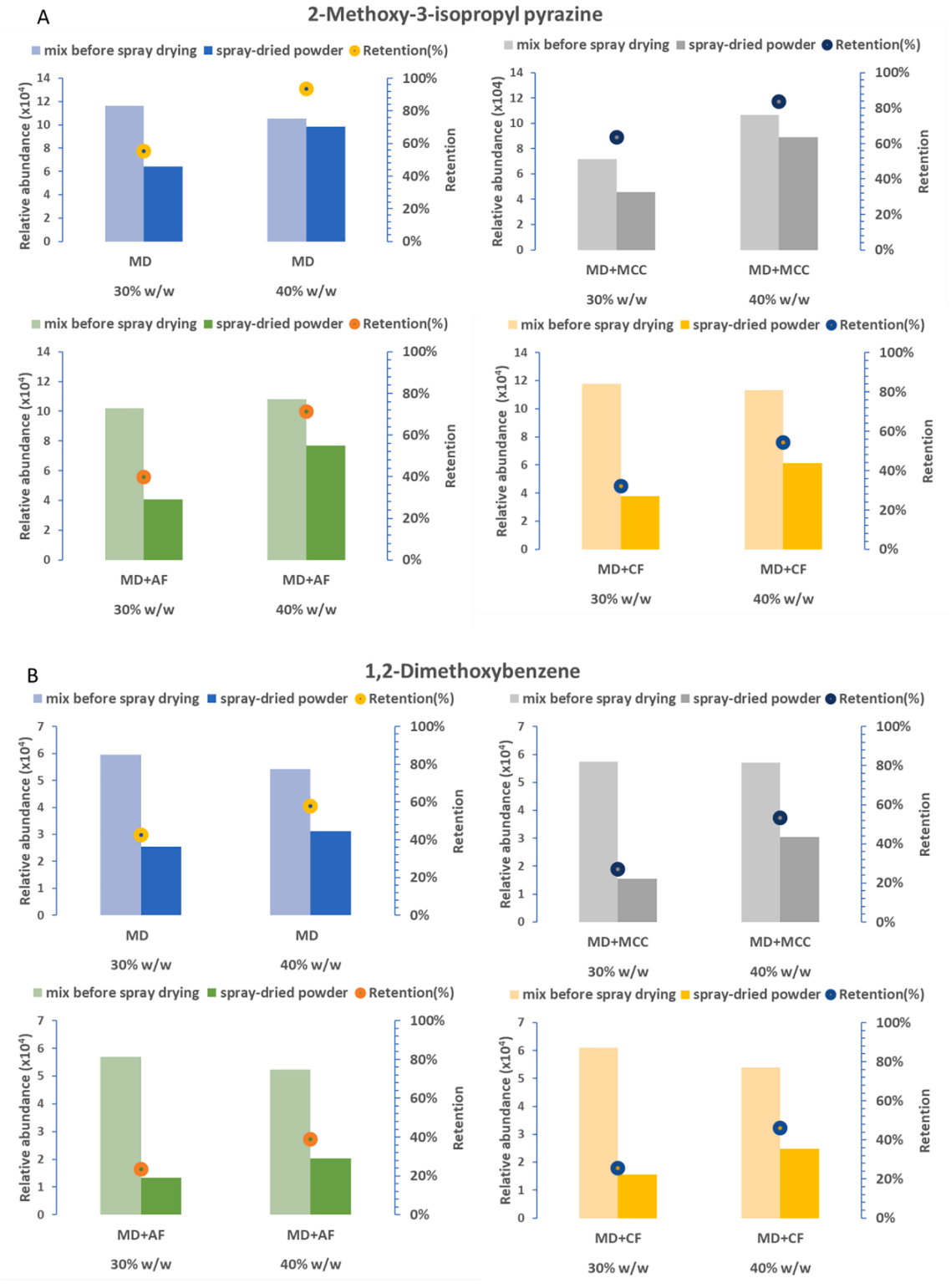

Fig. 4. Relative abundance in the mix before and after spray drying, and the retention% in the samples with 30% and 40% w/w total solids content and MD (blue graphs), MD + MCC (grey graphs), MD + AF (green graphs), or MD + CF (yellow graphs) of selected volatiles (A): 2-Methoxy-3-isopropyl pyrazine, (B): 1,2-Dimethoxybenzene and (C): 2-Butanol, 3-methyl, acetate. (For the list of abbreviations see Table 1.) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

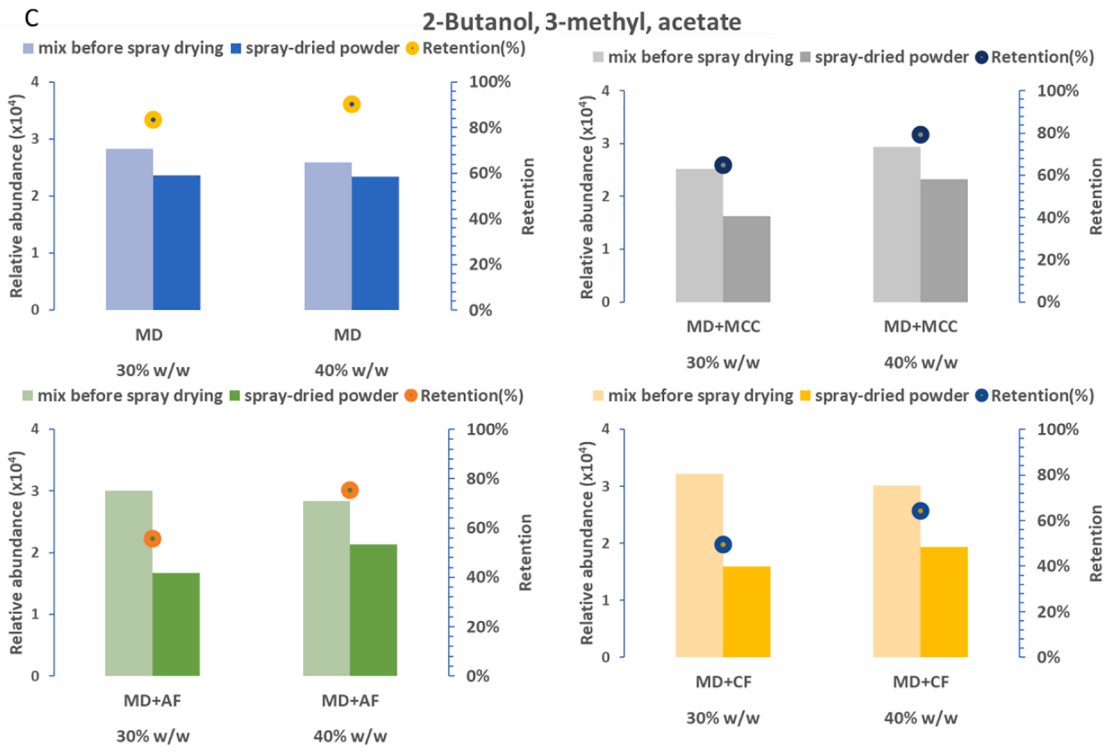

Fig. 4. (*continued*).

Rosenberg et al., 1990). The drying conditions in the presented spray drying experiments here were similar. We calculated the retention of a selection of specific key volatiles under different spray drying conditions (solids content and carrier types). The asparagus concentrate mixed with the carriers were analysed both before and after spray drying using the same HS-SPME GC–MS method. The samples with the same level of fibre supplement and those containing only MD were included in this analysis (Fig. 4). To estimate the retention of a detected volatile, the ratio of the peak intensity in the spray-dried powder to that in the mixture before spray drying was calculated. The level of retention was not the same for all volatiles analyzed in the spray-dried powders but there is an apparent trend (Fig. 4). Retention of volatiles was higher in the samples with a total solids content of 40% w/w, regardless of carrier type. A partial replacement of MD with the cellulose-based carriers led to lower retention of the volatiles, as compared to the powders containing only MD. However, the retention of important volatiles e.g. 2-methoxy-3-isopropyl pyrazine in the AF-containing powders was ca. 80% and thus, acceptable (Fig. 4A).

Moreover, when comparing the retention and the abundance of important asparagus volatiles (e.g. 2-butanol, 3-methyl- acetate and 2-methoxy-3-isopropyl pyrazine) between the samples where MD was partially replaced with a vegetable fibre (CF or AF), those powders with AF performed slightly better (Fig. 4 and Supplementary Fig. S6). Few studies have addressed the role of cellulose-based carrier agents in the encapsulation of bioactive compounds. Chiou and Langrish (2007) evaluated the potential of CF as a carrier agent to fully replace MD. The obtained spray-dried powders had good physical properties and the bioactive compound (*Hibiscus sabdariffa* L.) has been detected within the fibre carriers, thus demonstrating the potential of CF to replace MD. On the other hand, Yousefi et al. (2015) found that high MCC concentration detrimentally affected the encapsulation of bioactive compounds in spray-dried black raspberry juice due to the structural changes of the powder particles. Do et al. (2018) also concluded that the bioactive compounds in spray-dried mulberry powder were less protected when MCC was added and explained this by the observed separation of the mulberry extract and MCC particles in the powder. We conclude that the partial replacements tested in this study led to acceptable retention of asparagus volatile compounds.

For several compounds, including 3-Methylthiopropanal, the retention was higher than 100% (data not shown). This implies that the concentrations of these volatiles are higher in the spray-dried powder, implying that more of these volatiles have been formed during the spray drying process. This is likely due to e.g. thermal or oxidative degradation of the sulphur-containing amino acids or polyunsaturated fatty acids, or the initiation of temperature-induced Maillard reactions.

4. Conclusions

In this study, we demonstrated that partial replacement of MD by cellulose-based carriers is possible for successfully spray drying asparagus concentrate while retaining important asparagus volatile flavour compounds. Partial replacement of MD could be done up to a maximum of 3% in the case of AF and CF or 10% in the case of MCC. This limitation was due to the poor solubility of the vegetable fibres. Introducing an extra pre-treatment step for solubilising the asparagus fibre could overcome this disadvantage. Particle morphology and the levels of the detected volatile compounds differ in the prepared powders depending on the total solids content, as well as the type of the fibre used to replace MD. Spray-dried powders with a total solids content of 40% w/w led to the formation of better-quality powders, compared to 30% w/w total solids in terms of moisture content and particle morphology, as well as better retention of many volatiles. Furthermore, powders where MD was replaced by AF, showed similar morphology to the powders with only MD but had a richer volatile profile because of a contribution of the AF carrier providing extra asparagus aroma compounds. These results suggest the further exploitation of vegetable fibres as a potential (partial) replacement for MD. Further optimization of the processing technique used can contribute to effective exploitation of the extensive asparagus waste stream to produce high-quality natural food ingredients. Such natural flavour ingredients can then be used in soups, sauces, etc. to replace currently used artificial flavourings.

CRediT authorship contribution statement

Joanne W. Siccama: Conceptualization, Methodology, Formal analysis, Visualization, Writing - original draft. **Eirini Pegiou:** Conceptualization, Methodology, Formal analysis, Visualization, Writing - original draft. **Nienke M. Eijkelboom:** Methodology, Investigation, Formal analysis. **Lu Zhang:** Supervision, Writing - review & editing. **Roland Mumm:** Supervision, Methodology, Writing - review & editing. **Robert D. Hall:** Conceptualization, Supervision, Writing - review & editing. **Maarten A.I. Schutyser:** Conceptualization, Supervision, Writing - review & editing, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2021.129567.

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