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ATTRIBUTES OF LING-HEATHER HONEY POWDER OBTAINED BY DIFFERENT METHODS WITH SEVERAL CARRIERS

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Abstract

In order to obtain the best physicochemical and the most appealing honey powder as possible, this research assessed three procedures (spray drying, vacuum drying and freeze drying) as well as three carrier agents (Arabic gum, whey protein isolate and maltodextrin) for dehydrating ling-heather honeys (*Calluna vulgaris* (L.) Hull). Using each carrier agent, both vacuum and freeze drying were the procedures that provided higher recoveries (76%-98%) and higher honey final concentrations in the powders (56%-73%). The most suitable carrier agent was maltodextrin, because with it, lower moisture (1.90%-4.20%), higher solubility (21 s-123 s) and lower hygroscopicity (6.32%-13.68%) was achieved. Honey powders obtained with maltodextrin by vacuum and freeze drying exhibited higher recoveries (88%-98%) and the best sensory characteristics, with stronger floral odours and flavours, stronger sweetness, lower viscosity and lower waxy perceptions.

Keywords: honey powder; freeze drying; vacuum drying; spray drying; carrier agents.

Abbreviations:

SP: spray drying

VC: vacuum drying

FZ: freeze drying

MD: maltodextrin

AG: Arabic gum

WH: whey protein isolate

LH: Ling-heather honey

LHP: Ling-heather honey powder

Highlights

Spray, vacuum, freeze drying and three carriers were assessed to make honey powders.

At lab scale vacuum and freeze powders with maltodextrin showed the highest recoveries.

Honey powders with maltodextrin showed lowest moisture and highest solubility.

Powders with maltodextrin showed highest tapped density and lower hygroscopicity.

Vacuum and freeze powders with maltodextrin were the most appealing.

1. Introduction

Honey is the natural food made by bees, being very appreciated for its pleasant sensory attributes, excellent culinary properties, as well as health, cosmetic and technological advantages, among other benefits. The major components of honey are fructose and glucose that together with other sugars, provide high viscosity and stickiness, which can make honey difficult to handle for some culinary specialties and pharmaceutical applications. Honey powder is a good alternative that can be directly added into dry mixtures, seasonings or dry coatings, maintaining flavours, and providing ease of handling and weighing as well as better sanitary aspects (Samborska, 2019).

Making honey powder is difficult, because of the high sugar content with low glass transition temperature (T_g), which provokes a highly sticky product, extremely difficult to deal with (Jedlińska et al., 2019; Samborska et al., 2019; Shi, Fang, & Bhandari, 2013; Umesh Hebbar, Rastogi, & Subramanian, 2008). Therefore, the addition of carrier agents that provide high molecular weights is essential to make honey powder, because they increase T_{g}. Among them, Arabic gum, maltodextrin, starch or glucose syrup stand out (Fan, & Roos 2019; Ram, 2011; Nurhadi, Andoyo, Mahani, & Indiarto 2012; Sramek, Woerz, Horn, Weiss, & Kohlus, 2016). Likewise, the addition of low quantities of surface-active substances, such as whey protein isolate or sodium caseinate, is common for obtaining honey powder because the proteins help encapsulate honey (Samborska, Langa, Kamińska-Dwórznicka, & Witrowa-Rajchert, 2014a; Shi et al., 2013; Suhag & Nanda, 2016).

Spray drying is currently the most studied technique to obtain honey powder, having been described as more cost-effective, faster and easier to scale up option than other procedures (Samborska et al., 2014a; Shi et al., 2013; Suhag & Nanda, 2015). Some researchers improved powder recoveries (up to 90%) or/and obtained higher honey final concentrations (up to 80%) using proteins as carriers, diafiltration before spray drying or dehumidified air as a drying medium during spray drying (Jedlińska et al., 2019; Samborska, Sokolowska & Szulc, 2017; Shi et al., 2013). Vacuum drying (Nurhadi et al., 2012; Sahu, 2008), freeze drying (Sramek et al., 2016) vacuum puffing (Sahu & Devi, 2013), microwave-vacuum (Cui, Sun, Chen, & Sun, 2008) and foam-drying (Sramek et al., 2016) have been less-investigated methods. Vacuum and freeze drying could be promising procedures to obtain honey powders. So far literature has shown no data about the highest honey concentration that could be obtained using vacuum or freeze drying methods to make honey powder.

Most studies on powdered honeys were done with multifloral, rape, buckwheat and sunflower samples. Jedlińska et al. (2019) obtained honey powder using honeydew honeys, which were more easily dried than blossom honeys, due to the higher content of oligosaccharides of honeydew honeys. Studies of the properties of honey powders obtained with other honeys are of utmost interest nowadays, because various honeys can differently react to drying procedures and drying carriers, providing different features to the powders. Ling-heather (*Calluna vulgaris* (L.) Hull) and other heather honeys are very appreciated by the consumers due to their special flavours and potential biological properties. Ling heather are non-Newtonian honeys with thixotropic behaviour, so that their apparent viscosity decreases while shear rate increases due to high molecular weight colloidal matters such as proteins or dextrans (Osés et al., 2017). Furthermore, heather honeys have higher monosaccharides (around 71%) and lower disaccharides (around 4.9%) contents than other honeys (Pascual et al., 2018), which could be a disadvantage for obtaining honey powder, because glucose and fructose generate a higher stickiness than other sugars such as sucrose, which is related to the lower T*g* of the former sugars (31 ^{o}C for glucose and 5 ^{o}C for fructose), in comparison to the higher T*g* (62 ^{o}C) of sucrose (Jayasundera, Adhikari, Aldred, & Ghandi 2009).

In the last few years, several researchers studied the physicochemical properties of honey powders mainly obtained by spray drying, using Arabic gum, whey isolate proteins and maltodextrin as carrier agents (Nurhadi et al., 2012; Sahu & Nanda, 2016; Samborska, Gajek, & Kamińska-Dwórznicka, 2015a; Shi et al., 2013). Despite its paramount importance for consumers, sensory analysis of honey powders was little carried out, so far (Nurhadi et al., 2012). Samborska (2019) published a review summarizing the main methods and carriers used for drying honeys, as well as the problems and physicochemical properties of honey powders, concluding that *"the production of powdered honey is still a challenge both for industry as well as researchers"*. Agreeing with Samborska (2019), the aim of this work was to make a ling-heather honey powder with the highest honey content, the lowest carrier amount and the highest yield as possible, also achieving pleasant sensory characteristics and good physicochemical properties. Three different drying methods (spray, vacuum and freeze) and three different carrier agents (Arabic gum, whey protein isolate and maltodextrin) were researched.

2. Materials and Methods

2.1. Honey samples

This work was done with three ling-heather (*Calluna vulgaris* (L.) Hull) honeys (LH) from "Castilla-León" (Spain) supplied by beekeepers (LH1, LH2 and LH3). The botanical origins were determined by both melissopalinology (Louveaux, Maurizio, & Vorwohl, 1978; Terradillos, Muniategui, Sancho, Huidobro, & Simal-Lozano, 1994; Von der Ohe, Persano Oddo, Piana, Morlot & Martin, 2004) and sensory analyses

(Marcazzan, Mucignat-Caretta, Marchese & Piana, 2018; Persano Oddo & Piro, 2004; Piana et al., 2004). The corresponding powders (LHP) for each honey sample were named as: LHP1, LHP2 and LHP3.

2.1.1. Quality control parameters

Moisture, water activity (aw), fructose, glucose, sucrose, electrical conductivity, pH, acidity, formol number, diastase activity, hydroxymethylfurfural (HMF), proline and colour were determined by AOAC (2012), Bogdanov (2009), Codex, (2001), Commission Internationale de L'eclairage (2004), Hadorn & Zürcher (1963), Pascual et al. (2018) and Sancho, Muniategui, Huidrobo, & Simal (1991) procedures.

2.2. Carriers

Arabic gum (AG) (Sigma-Aldrich, 30888, spray dried, tested according to Ph Eur). Whey protein isolate (WH) with a protein content of 90% (Myprotein, Northwich, UK). Maltodextrin of dextrose equivalent 20 (MD) kindly provided by Calaf Nuances (Barcelona, Spain).

2.3. Drying methods

LHP (27 samples) were obtained from three honeys (LH1, LH2 and LH3) by three techniques: spray drying (SP), vacuum drying (VC) and freeze drying (FZ), using for each method three carriers: AG, WH and MD. In order to get the highest honey concentration for each technique with each carrier, different honey and carrier concentrations and different treatment conditions were tested for each technique before achieving the final procedure described in this work. To decide the final honey content, we chose the highest honey content, with which the final product could be properly pulverised. Table 1 shows the ratios of honey solids to carrier solids and feed solution concentration (%) obtained for each drying technique by each carrier.

SP: The feed solutions with different honey:carrier were prepared at 18% solid fraction (w/w). 300 ml of solution were spray dried for each run in a Büchi B-290 mini spray dryer (Büchi Labortechnik AG, Switzerland) in open-cycle system, at drying air inlet and outlet temperatures of 120 ± 1 ºC and 67.5 ± 2.5 ºC, respectively. The air flow rate was maintained at 50 mm with the aspirator rate at 70%, nozzle cleanness of 4 times/min and pump at 5%. The honey powders (SP-LHP) were collected from the cyclone after each spray-drying process and stored in glass jars in desiccator containing silica gel until analyses.

VC: honey and carriers were first mixed with water separately. Then, they were mixed together gently stirring with a magnetic stirrer at room temperature (24 ± 1.5 ºC), keeping a total solid concentration of 43-48% (w/w) and the ratios of honey solids (honey concentration) to carrier solids in feed solution as shown in Table 1. Each sample was poured into a silicon baking cup with 3-4 mm thickness and dried in a lab scale vacuum oven (Heraeus Instruments vacutherm, Thermo Scientific) at 60 ºC with absolute pressure maintained inside the chamber at 100 mbar (75 mmHg) for 24 h. Then, the samples were kept in desiccator until cooling and before analyses the samples were grounded with a mortar and the honey powders (VC-LHP) were stored in glass jars in desiccator containing silica gel.

FZ: The honey:carrier mixture was prepared in the same way as described for VC (Table 1) and was also poured in a silicon mold. The mix was frozen at -30 ºC for 5 h and later at -80 ºC for 24 h prior to be dehydrated by freeze drying in a Labconco Freezone 12L freeze dryer (Kansas, USA) throughout three days at 0.112 mbar pressure and immediately grounded. The honey powders (FZ-LHP) were stored in glass jars in desiccator containing silica gel until analyses.

2.4. Powder recovery

The powder recovery was calculated according to the following formula.

$$\%\ \text{powder recovery} = \frac{\text{Total solids in resulting powder}}{\text{Total solids content in feed}}\ \text{x}\ 100$$

2.5. Quality parameters and physical properties

Water activity (aw), proline and colour parameters L*, a*, b* of LHP were determined following the same procedures described for raw honeys.

2.5.1. Glass transition temperature

Thermal properties of the honey samples and powders obtained by LH1 (as representative for all the powders obtained) were measured using differential scanning calorimetry (DSC, 10-15 mg of the sample under a constant nitrogen flow of 50 mL·min–1, with a TA Instruments Q200 DSC analyser equipped with a cooling Intra-Cooler system, at 20 °C·min−1). The measuring method was based on a heating/cooling/heating sequence, which started at -80 °C and goes up to no more than 120 °C, thus avoiding degradation of the sample (Cordella et al., 2002; Cordella, Faucon, Cabrol-Bass, Sbirrazzuoli, 2003). The measurements were carried out in pierced aluminium pans, and values for T_g were calculated using "TA Universal Analysis" software.

2.5.2. Moisture content

The moisture content was determined gravimetrically by drying 1 g LHP at 105 ºC during 4 h (Samborska, Langa, Kamińska-Dwórznicka, & Witrowa-Rajchert, 2015b).

2.5.3. Hygroscopicity

For hygroscopicity measurement, 1 g LHP was placed in low-form glass weighing bottles and placed in desiccators at room temperature and equilibrated over a saturated solution of NaCl (75% relative humidity) for one week. Samples were weighed every hour during the first four hours to obtain the hygroscopic rate (g H_2O/min, the slope of weight change of honey powder) and the hygroscopic index that was expressed as g of absorbed moisture after one week per 100 g dry solids (g/100g) (Nurhadi et al., 2012; Shi et al., 2013; Suhag & Nanda, 2015).

2.5.4. Tapped density

Tapped density was measured by pouring 2 g LHP into an empty graduated cylinder and gently tapping 25 times and recording the volume (Suhag & Nanda, 2015, 2016), expressing the results as g/ml.

2.5.5. Solubility

The solubility of powders was measured by adding 1 g LHP to 25 ml of distilled water in a 100 ml glass beaker at room temperature. The mixture was agitated with a magnetic stirrer at 890 rpm (stirring bar 8 mm x 25 mm), recording the time required for each powder to dissolve completely (Samborska & Bieńkowska, 2013).

2.6. Sugars

Glucose, fructose and sucrose were determined following the same procedures for raw honeys by GC-chromatography after previous derivatization described by Pascual et al. (2018).

2.7.Sensory analysis

A descriptive quantitative sensory profile method was selected to obtain the sensory differences between 9 samples of LHP, performing the analysis with the powdered samples obtained with LH1. The method was developed following the ISO standards and international procedures (ISO 4121, 2003; ISO 13299, 2016; 6658, 2017; Marcazzan et al., 2018).

The panel consisted of seven selected assessors (4 women and 3 men, ranging between 31 and 57 years old) with previous experience in honey sensory analysis (one of them registered as expert in Sensory Analysis of Honey by the Italian Government).

In the first session the panel generated descriptors individually, in order to describe the differences between carriers and honey powders, tasting different ling heater honeys, carriers dissolved in water (AG, WH and MD) and three different powdered honeys obtained in previous experiments. In the same session nineteen attributes were chosen and defined in common (Table 2). Furthermore, the intensity of each descriptor was consensual.

The second session was carried out choosing as representative of the powders, the 9 samples of LHP1 (with the three carriers and obtained by the three drying procedures), in order to prevent palate saturation with 27 tastings if LHP2 and LHP3 were additionally tasted. LHP1 samples were dissolved in water (2.5:1 [honey:water]) and then assessed. All descriptors and the global perception were quantified for each sample in a 10 cm continuous unipolar scale (10 cm continuous scale, anchored in 1 cm (minimum) and 9 cm (maximum), marked from 1 to 9, where the left side of the scale corresponding to the absence of the characteristic and the right side to the maximum intensity. The intensity of the attributes was quantified by measuring from the left-hand side on the line scale to the assessor's mark, calculating the median value for each attribute.

The 9 LHP1 samples were presented at random and anonymous way (only the three-digit code) in a glass container with a lid to prevent the contamination and dispersion of the honey odours, maintaining the sample/volume ratio near 1/4. Plastic spoons were used to stir the honey during the olfactory assessment and to taste the product. Mineral water and low-salt bread were used to clean the palate between tastings.

2.7. Statistical analysis

All the assays were carried out in triplicate except sensory analysis. The results were expressed as averages and standard deviations. A normality test was done. One-way analysis of variance (ANOVA) followed by Tukey's honestly significant difference test ($p < 0.05$) were used for parametric values, while non-parametric values were analyzed using the Kruskal-Wallis test followed by box-and-whiskers graphic interpretations. For sensory analysis, principal components analysis (PCA) and cluster analysis using Ward's linkage algorithm and Euclidean distance were carried out. Analyses were done using software Statgraphics Centurion XVIII (Statgraphics Technologies, Inc., The Plains, VA, USA).

3. Results and Discussion

3.1. Honey samples

Table 3 shows the results of honeys' quality control parameters. With regard to the composition criteria that European honeys must meet, moisture (16.04-17.54%), sum of fructose and glucose (60.05-71.69%), sucrose (0.24-2.70%), electrical conductivity (0.33-1.01 mS/cm), free acid (41.02-49.77 meq/kg) diastase activity (3.93-23.55 Schade scale) and HMF content (0.00-18.01 mg/kg) complied with the European limits for these parameters (OJEC, 2002). Aw values (0.5400-0.6243) were within the usual range for honey samples, exhibiting LH3 the lowest aw result and the lowest moisture percentage. Aw values lower than 0.6 inhibit osmophilic yeasts that can be responsible for honey fermentation (Bogdanov, 2011). Samples' pH ranged from 3.87 to 4.14, being within the usual values for blossom honeys (Bogdanov, Ruoff, & Persano-Odoo, 2004). The three LH showed high free acidity (41.02-49.77 meq/kg) and very low lactone acidity (0.00-0.23 meq/kg). Formol number (0.61-0.93 meq/100 g), which is related to the free amino acid content (Hadorn & Zürcher, 1963), agreed with literature references (Andrade et al.,1999; Manuel Suisse des Denrées Alimentaires, 1974). Proline (73.06-107.24 mg/100 g) was higher than 18 mg/100 g, which is the minimum level proposed for authentic honeys (Bogdanov, 2016). Samples exhibited redness colour, which is typical for heather honeys. LH3 was lighter, less reddish and more yellowish compared with the other honeys. Some researchers pointed out that the darker the honeys were, the higher the phenolic contents and antioxidant activities were (Álvarez-Suárez et al., 2010; Meda, Lamien, Romito, Millogo, & Nacoulma, 2005).

3.2. Powdered honeys

3.2.1. Powder recovery

LHP showed recoveries between 59.88% and 97.90% (Figure 1). Regardless the honey sample, VC-LHP (80.55-97.90%) and FZ-LHP (73.62-96.41%) exhibited the highest recoveries, while SP-LHP showed the lowest (59.10-78.60%). The lower yield shown by SP in comparison with VC and FZ could be likely due to the greater temperature difference between the temperature at which the powder is obtained and its T_g (Jedlinska et al., 2019), because this fact increases the gumminess of the product, negatively affecting the yield. Anyway, our data for SP-LHP were higher than the 50% described as successful recoveries for spray drying by Shi et al. (2013). MD was the carrier, with which the highest recoveries were obtained by VC and FZ, whereas WH was the carrier agent with which the highest recovery was obtained by SP.

With regard to the carrier agents: 1) Using AG the recoveries varied between 60% and 69%, with 58% honey concentration (58:42 ratio) in SP-LHP. Similar values were described by Suhag, Nayik, & Nanda (2016) for the same carrier and the same procedure, achieving a honey powder with 65% of honey and 60% of recovery. Other researchers that also dehydrated honeys with AG by SP obtained powders with lower honey quantity (50%) and yields between 36.6% (Nurhadi et al., 2012) and 68%, increasing the recovery until 75% if caseinate (2%) was added (Samborska et al., 2014a). Samborska et al. (2017) obtained a powder with 75% honey, but with a yield of 25% by applying diafiltration previously to the SP. Dehydrating by VC, Nurhadi et al. (2012) obtained 73.8% recovery with 50% honey in the final powder. In our study, considerably higher yields (80%-89.4%) were obtained by VC with AG (VC-AG). FZ proved to be the best and most effective procedure to make powdered honeys, because using it, the highest recovery (87%-93%), and the highest concentration of honey in the powder (73%) were accomplished. 2) With WH, the same final honey concentration (72%) was obtained by the three techniques with the following recoveries: SP: 74.8-78.6%, VC: 85-95%, FZ: 75-94%. For SP, WH proved to be the most efficient carrier, because higher amounts of honey in the powder (72%) and higher recovery percentages (75%-79%) were achieved, agreeing with other studies (Shi et al, 2013; Suhag & Nanda, 2016). 3) Using MD, the yield varied from 59% to 72% and the final honey concentration in the powder was 46% in SP-LHP. Applying the same technique and employing the same carrier, other scientists showed recoveries between 9.7% (Nurhadi et al., 2012) and 74% (Samborska et al., 2020) with 50% honey in the final product. However, in order to get a higher honey concentration Shi et al. (2013) added 2.5% WH to the feed, obtaining a powder with 60% honey and a yield of 70%. Working with a rape honey, Samborska et al. (2019) obtained a powder with higher amount of honey

(60-80%) and with very high yield (75-90%) after using dehumidified air throughout the spray process, being likely that rape honeys are easier to dry than heather honeys. In our study, higher yields and higher honey concentrations were obtained using VC (90-98% yield, 56% honey) and FZ (88-96% yield, 72% honey), being our results considerably better than those obtained by Nurhadi et al. (2012), using VC and MD (73% recovery, 50% honey in the powder) or by Fan & Roos (2019), who obtained a powder with 50% of honey using FZ.

3.2.2. Physical properties

3.2.2.1. Glass transition temperature (T_g)

Table 4 shows a single T_g in LH (between -27.33 and -29.81 °C). Our values were higher than the results obtained for other honeys (Kántor, Pitsi & Thoen, 1999; Nurhadi et al., 2012; Shi et al., 2013; Sramek et al., 2016), where values between -40.2 and -51 °C were described. The contrasting values could be due to the different botanical origins, or to the different T_g measuring procedure. Nurhadi et al. (2012) explained that the glass transition temperature of liquid honey is strongly affected by moisture content, since water acts as plasticizer and sample´s T_g decreases considerably.

However, LHP1 showed two transitions at higher temperatures (Table 4). This fact can be explained by the dual role of water as compatibiliser and plasticiser. The observed two *Tg* in dried samples correspond to two different polymers. *Tg*1 was likely due to dry honey polymers. *Tg*2 (that was less noticeable than the first one, Figure 1-suplementary material) was likely due to the carriers. *Tg*2 seemed to be lower than *Tg* of pure carriers (205.5 ºC for MD; 194.5 ºC for AG; 132.12 ºC for WH, Nurhadi et al., 2012; Shi et al., 2013), which showed a partial compatibility between honey polymers and carriers, but not a total compatibility, since in the latter case, only a single *Tg* would be observed. Values for *Tg*2 of powders obtained with WP were lower than *Tg*2 for powders obtained with MD, confirming the influence of the chemical nature of carriers in the compatibility of mixtures. Sample SP-AG-LHP1 can be highlighted, because only a single T_g was observed at 49.86 °C, meaning that the carrier and honey polymers were totally compatible in this case.

The T_g1 for LHP1 ranged from 13.31 to 25.99 °C, except for SP-AG-LHP1. The results were lower than those obtained by other researchers who described T_g values between 42.6 and 49.7 °C in powders obtained by vacuum and T_g values between 47.5 and 82.14 °C in powders obtained by spray drying (Sahu et al., 2008; Samborska et al., 2020; Shi et al., 2013). However, we obtained similar T_g values than Jedlinska et al. (2019) and Samborska et al. (2019) for honey powders obtained by dehumidified air spray drying (T_g between 6.5 and 26.1°C). These researchers obtained similar honey concentration (60-80%) than us in final powders, confirming that the T_g values decrease as honey concentration increases. MD-LHP exhibited the higher T_g values for all the drying procedures, which is probably due to the lower honey:carrier ratio in these samples.

The low values of T_g for honey powders (close to room temperature), suggest that powdered honeys should be stored at refrigeration temperatures in high-barrier packaging because amorphous materials are unstable if stored at temperature close to or higher than T_g (Jedlinska et al., 2019).

3.2.2.2. Colour

LHP colour is a quality factor of paramount importance for consumers. Honey colour varies from colourless and light yellow to dark amber or nearly black, sometimes with green or reddish reflexes (Bogdanov, 2011), being related to the botanical origin. Heather honeys, together with chestnut and honeydew honeys are dark coloured with L* values lower than 50 (González-Miret, Terrab, Hernanz, Fernández-Recamales, & Heredia 2005). Figure 2 shows photos of the powders, where the colour of powders can be observed. Table 5 show that lightness (L*) of LHP was significantly higher ($p<0.05$) for SP-LHP and significantly lower ($p<0.05$) for VC-LHP. Higher L* values were described by Nurhadi et al. (2012) in SP and VC powders with AG and MD (94.3-98.1) and by Samborska et al. (2019) in SP powders with MD (93.9-94.3). Generally, AG-LHP showed higher lightness. As expected, LHP3 showed the highest values for lightness.

Regarding a* values, all the carriers and most SP-LHP showed negative values, which meant that they had a slightly green tonality. Conversely, VC-LHP and FZ-LHP tended to red, being the VC-LHP the reddest. LHP3 exhibited the lowest a* values, which agreed with the values obtained for the LH (Table 3). In general, AG-LHP showed lower a* values than WH-LHP and MD-LHP. VC-WH-LHP exhibited the highest a* values ($p<0.05$). Negative a* values were also described in other studies for SP powdered honeys (-0.1 to -0.5) (Nurhadi et al., 2012; Samborska et al., 2019). However, in VC powders Nurhadi et al. (2012) obtained negative values (-0.2). This contrasting difference between the results of Nurhadi et al. (2012) and the results of this study is likely due to the different time of drying. The honey powders made by Nurhadi et al. (2012) were obtained in 1 h, whereas the LHP of our research were obtained in 24 h.

With regard to b* values, all LHP tended to yellow. SP-LHP showed the lowest b* values, in contrast to VC-LHP, which exhibited the highest b* values ($p<0.05$). In comparison to LHP1 and LHP2, LHP3 showed the lowest b* values, conversely to the data obtained for the crude LH3 (Table 3). All AG-LHP showed the lowest b* values and VC-WH-LHP the highest values. Nurhadi et al. (2012) obtained lower b* values with AG and MD (2.1-10.5), also obtaining higher b* values in VC powders in comparison with SP powders.

Comparing the colour parameters between the crude honeys and their powders it can be observed that in LHP L* increased (tending to whitish), while a* and b* decreased, probably due to the use of carriers. Furthermore, the significant decrease of L* value and the increase in a* and b* values of VC-LHP, particularly for the VC-WH-LHP, could be attributed to Maillard reactions and fructose and glucose caramelization. VC-LHP were dehydrated at 60 ºC for a long time. Moreover, it is likely that there were deteriorative reactions of polyphenols with proteins using WH (Samborska et al., 2014a).

3.2.2.3. Moisture content and water activity

Table 5 shows the results for moisture and aw. LHP showed moisture results between 1.90% and 7.48%. In general, the lowest moisture percentages were obtained for honeys dehydrated with MD (MD-LHP) and the highest moisture percentages for honeys dehydrated with WH (WH-LHP). Similar values and the same tendency were shown for Shi et al. (2013) and Suhag & Nanda (2016) in SP honey powders, where the honeys dehydrated with WH exhibited the highest moisture (4.5-5.7%), with GA intermediate moisture (4.6%) and with MD the lowest moisture (3.1-3.6%). This behaviour could be expected since there are differences among the chemical structures of the carrier agents. Proteins showed a strong ability to bind water and coated the droplets with a thin film layer, preventing evaporation (Suhag & Nanda, 2016). However, this tendency was not observed in other studies in VC-LHP, where the final moisture percentages ranged between 1.40% and 1.92% using AG, WP and MD without significant differences among carriers (Mutlu et al., 2020). Lower moisture values (0.9-1.6%) were obtained when dehumidified air was used for SP (Samborska et al., 2019). There was no difference among honey samples, honey concentration or drying method, although in general the VC-LHP had lower moisture percentages. Also, Nurhadi et al. (2012) showed lower moisture in VC honey powders (1.1-2%) than in SP honey powders (2.3-4.4%). Other studies concluded that the water content of the powders depended on the ratio of carrier in the powder. The higher the content of carrier, the higher the final water content (Samborska et al., 2015a; 2017; 2019). In our study, we compared the three MD-LHP, using different ratio honey:carrier for each treatment. MD-LHP1 and MD-LHP2 followed the mentioned trend, unlike MD-LHP3. In other FZ powders, 3.10% moisture was obtained in the honey powder with 50% honey, using glucose syrups + 1% of WP as drying agent (Sramek et al., 2016).

WH showed the lowest value for aw and AG the highest. LHP exhibited a great variability with aw values ranging between 0.2106 and 0.3421. Similar values were obtained by Nurhadi et al. (2012), Nurhadi & Roos (2017) and Shi et al. (2013), for SP and VC, while Samborska et al. (2014a, 2017, 2020), Samborska, & Czelejewska (2014b) obtained lower water activity results (0.066-0.138) in SP powders with AG or MD. These differences could be attributed to the type of honey, the time until analysis and the storage conditions.

3.2.2.4. Solubility and tapped density

MD was the carrier with the highest solubility (10 s), while AG showed the lowest (311 s). LHP also exhibited the same tendency, being MD-LHP the most soluble (37-92 s) regardless the drying procedure. Honey dehydrated with AG (AG-LHP) were the least soluble ($p < 0.05$), taking between 83 and 286 s (Figure 3A). FZ-LHP had the best solubility values, followed by SP-LHP, while VC-LHP showed the lowest solubility (p

< 0.05). Samborska & Bieńkowska (2013) obtained similar values for SP powders with MD (28-148 s). These authors showed that the lower the moisture content of the powders was the worst the solubility was. Nevertheless, in our research that relationship changed, because powders with lower moisture, such as MD-LHP showed the best solubility, while WH-LHP that were the samples with higher moisture, showed an intermediate solubility.

Tapped density was higher in MD (0.64 g/ml) than in the rest of carriers (0.42-0.45 g/ml). LHP did not follow any clear tendency depending on the carrier, although MD-LHP showed the highest density in most samples (0.29-0.79 g/ml) (Figure 3B). Also, Samborska et al. (2015b) and Shi et al. (2013) concluded that tapped density was higher in powders with MD, which was related to their high degree of agglomeration and structural collapse, resulting in a decrease in volume of powder particles. MD-LHP, with different honey concentration for each drying treatment (Table 1), showed that the tapped density of SP-MD-LHP was lower than the tapped density of VC-MD-LHP and FZ-MD-LHP, being FZ-MD-LHP1 the exception. Therefore, there was an increase of tapped density when honey concentration increased (or carrier concentration decreased). Similar results were observed in other studies (Suhag et al., 2016). Lower tapped density was shown in SP-LHP (0.24-0.50 g/ml), for which it was necessary more space for storing the product in comparison with VC-LHP (0.61-0.79 g/ml) and FZ-LHP (0.37-0.78 g/ml).VC-LHP showed the highest density, except for VC-MD-LHP2 and VC-MD-LHP3. Similar values were obtained by Mutlu et al. (2020) for VC powders with three different carriers (AG, WH, MD) (0.74-0.80 g/ml). A slightly negative correlation was observed between tapped density and moisture ($r = -0.3814$, $p = 0.0020$), agreeing with other researches (Samborska et al., 2015b; Shi et al., 2013). The density of a product is relevant to the storage, processing, packaging and distribution.

Having higher density could be positive, because it implies a lower volume and therefore a more efficient storage, packaging and distribution. However, it could also be negative, because higher density could provoke higher cohesion between particles and form easier agglomerates.

3.2.2.5. Hygroscopicity

LHP is a product with high hygroscopicity due to its high sugar content. Figure 4 represents the hygroscopicity of the honey powders and the carriers. Fig. 4A shows the percentage of water gained in one week (hygroscopic index). Fig 4B exhibits the amount of water gained per minute (hygroscopic rate). The carrier WH showed the highest hygroscopicity for both measurements.

In general MD-LHP showed lower hygroscopic index (7.21-13.14%), unlike AG-LHP that showed higher percentages (10.18-16.26%), except for VC-AG-LHP3 and FZ-AG-LHP3. The hygroscopic values obtained in our study were lower than those described in other investigations of honey SP powders using the three carriers and the same analysis conditions (1 g/25 ºC/75% RH/7 days), with values between 20.13% and 27.8% (Samborska et al. 2020; Shi et al., 2013; Suhag & Nanda, 2015). The divergent results could be due to the type of honey. Our results were also contradictory to those of Suhag & Nanda (2016), who observed the lowest hygroscopicity (23.1%) for honey SP powders with WH and the highest (26.4%) for powders with MD. However, our values agreed with the results of Samborska et al. (2015b), whose MD honey powders showed the lowest hygroscopicity. These researchers attributed their values to the lower hygroscopicity of MD, as well as to a combination of factors, such as the conformation and topology of molecule and the hydrophilic/hydrophobic sites absorbed at the interface (Pérez-Alonso, Beristain, Lobato-Calleros, Rodriguez-Huezo, & Vernon

Carter, 2006). SP-LHP showed hygroscopicity percentages between 8.37% and 15.58%, VC-LHP exhibited values between 7.9% and 15.43%, and FZ-LHP between 5.56% and 16.41%. Similar results were described by Nurhadi et al. (2012) in VC honey powders with AG and MD (12-13.7%). Analysis of variance revealed no significant effect of the drying method on the hygroscopic index. However, the SP-LHP showed the highest hygroscopicity rate (Figure 4B), being lower and similar the VC-LHP and FZ-LHP. Therefore, during the first 4 hours SP-LHP gained water faster than the powders obtained by the other two treatments. However, after one week, the hygroscopicity index were similar for powders obtained by all procedures. Higher hygroscopic rates were obtained by Nurhadi et al. (2012), who described values between 0.00020 and 0.00033 g H_2O/min in SP and VC powders, with lower results in powders obtained with MD than with AG.

Although some authors described a relationship between the moisture of the powder and the hygroscopicity (Shi et al., 2013), in our research that correlation was not found. However, a very weak correlation was found between the amount of honey in the final powder and the hygroscopic level ($r = 0.3$, $p = 0.017$), which was also previously observed by Samborska et al. (2019).

3.2.3. Proline

This amino acid is being used as a possible indicator of honey quality. It would be interesting to check to what extent the incorporation of additives with proline-containing proteins (e.g. whey) can increase the proline content in powdered honeys with respect to the starting honeys in order to detect in the future powdered honeys made with proline-containing compounds.

Regarding the carriers, as expected, WH showed the highest value, followed by AG that is composed of a mixture of polysaccharides with 10% proteinaceous material, with 50% of hydroxyproline, serine and proline (Lopera, Guzmán, Cataño, & Gallardo 2009). LHP showed lower proline contents than the corresponding LH, showing WP-LHP the highest values, followed by AG-LHP. LHP2 showed higher proline contents, agreeing with the higher value of LH2. FZ-LHP showed the highest proline contents, probably because these powders contained the highest amount of honey and were not subjected to any thermal treatment. VC-LHP showed the lowest proline content, mainly due to the intense thermal treatments received (60 ºC/24 h), where Maillard reaction between sugar and amino acid could provoke a proline decrease.

3.2.3. Sugars

Sugars' content can be used to detect honey adulteration. Thus, determining sugar changes after drying process is useful to verify honey powders from adulterated honeys or possible addition of sugars to the honey powder.

Fructose and glucose were the major constituents of LHP. Table 7 shows that among the carriers, only AG had a little amount of both monosaccharides. In most LHP, fructose was higher than glucose, although there were some exceptions. Sucrose was not detected in any powder. In chromatographic methods, both gas chromatography and HPLC, %RSD of around 3% are considered acceptable. A very good chromatographic method may have values around 1% RSD, being common in literature papers with values of 6%, 7% or even 8%. Assuming a method with a 1% RSD, a value lower than 1% sucrose is not analytically significant, which may explain why in our study values of 0.00 were obtained. Actually, we verified that there were small sucrose peaks in the chromatograms, but after dividing this value by the considerably higher area of mannitol (used as internal standard), and after taking this value of the division to the calibration curve of sucrose, values of 0.00 were obtained in all cases for the powdered honeys of our study. What is clear, however, is that sucrose contents decreased after drying treatment. Therefore, if high sucrose contents were detected in a powdered honey, it is likely that sucrose was added to the powder.

We did not find a clear trend regarding sugar composition in relation with the drying procedure or with the carrier used, although LHP showed lower quantity of sugars than LH. Comparing our results with those of other studies, lower values were obtained by Nurhadi et al. (2012) in SP and VC honey powders with AG and MD, while higher values were shown by Kozłowicz et al. (2020) and Tomczyk, Zaguła, Tarapatskyy, Kačániová, & Dżugan (2020) in SP powders with MD (fructose: 30.6-39.5%, glucose: 27.2-51.6%).

3.2.4. Sensory analysis

The first part consisted of establishing a previous descriptive quantitative sensory profile of LHP. Nineteen attributes were assessed, from which six features (floral olfactory characteristic, floral and candy flavour descriptor, sweetness, viscosity and waxy), together with the general perception were chosen for a second step, after performing a statistical study of significant differences ($p<0.05$). Figure 5 shows the average for each attribute for each LHP. WH-LHP were described as very tasteless, with extremely weak odours and flavours, high viscosity and poor general perception. AG-LHP were described as having higher viscosity, being waxier and exhibiting floral odour and better flavour perception. MD-LHP were the most appreciated, obtaining the highest punctuation regarding general perception. VC-MD-LHP and FZ-MD-LHP showed the highest score for floral odour and flavour as well as for sweeteness, and the lowest for viscosity and waxy perception. VC-LHP had higher candy flavour and FZ-LHP had higher floral odour. SP-LHP were the worst scored by the panel, having been described as the most viscose. Literature references show that only Nurhadi et al. (2012) performed a sensory analysis where the honey powder made by VC was more appealing than honey powder made by SP taking into consideration aroma and taste. These authors also found that the use of MD increased the sensory acceptance. With the results of the sensory analysis, a PCA was performed. The first principal component described 43.48% variance, while the second component described 29.97% (Figure 6A, 6B). PC1 was mainly defined by general perception, viscosity and waxy perception, while PC2 was mainly defined by floral odour and candy flavour. The powders could be divided into three different groups: group I, where VC-MD-LHP and FZ-MD-LHP were included, being the most appealing powders in terms of general perception, higher floral sweet flavours; group II, where WH-LHP and VC-AG-LHP were included, being the less appealing powders characterized by weak floral odour and flavour; and group III, where SP-AG-LHP, SP-MD-LHP and FZ-AG-LHP, where included, being these powders characterized by lower sweetness and candy flavour. The same division of powders into clusters was observed in the dendrogram chart (Figure 6C). Thus, it can be concluded that the drying method, the type of carrier and the honey content highly influence the powder's sensory acceptance.

4. Conclusion

For first time, three different drying procedures (SP, VC and FZ) and three different carrier agents (AG, WH and MD) were researched to make powdered honeys. Unlike SP, both VC and FZ were the procedures that provided higher recoveries and higher honey final concentrations in the powders using each carrier agent. The most suitable carrier agent was MD, because with it, lower moisture, higher solubility and lower hygroscopicity were accomplished. VC-MD-LHP and FZ-MD-LHP achieved higher recoveries and the best sensory characteristics, with stronger floral odours and flavours, stronger sweetness, lower viscosity and lower waxy perceptions.

Authorship contribution statement

Sandra María Osés: Conceptualization, Data curation, Investigation, Methodology, Resources, Supervision, Writing - original draft. Leire Cantero: Formal analysis, Investigation, Validation. Guillermo Puertas: Formal analysis, Investigation, Validation. Miranda Crespo: Formal analysis, Investigation, Validation. Lara González-Ceballos: Investigation, Formal analysis. Saúl Vallejos: Investigation, Formal análisis, Methodology, Writing – orginal draft. Miguel Ángel Fernández-Muiño: Investigation, Methodology, Resources, Supervision, Visualization, Writing - original draft. María Teresa Sancho: Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Supervision, Visualization, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no conflicts of interest with respect to the work described in this manuscript.

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Table 1. Averages of samples LHP1, LHP2 and LHP3 corresponding to the ratios of honey solids to carrier solids and feed solution concentration (%) obtained for each drying technique with three different carriers (AG, WH and MD). Standard deviations were lower than 0.2% for all percentages.

	SP		VC		FZ	
	Honey solids to carrier solids ratio	Feed solution concentration (%)	Honey solids to carrier solids ratio	Feed solution concentration (%)	Honey solids to carrier solids ratio	Feed solution concentration (%)
AG	58:42	18.7%	73:27	43.2%	73:27	43.2%
WH	72:28	18.7%	72:28	43.2%	72:28	43.2%
MD	46:54	17.2%	56:44	48.8%	72:28	43.2%

AG: Arabic gum; MD: maltodextrin; WH: whey protein isolate; SP: spray; VC: vacuum; FZ: freeze

Table 2. Attributes selected for the panel in this study for the sensory profile evaluation on powder honeys

Olfactory characteristics	Flavour descriptors	Taste descriptors	Texture perceptions
Floral	floral	sweetness	viscosity
Candy	candy	sourness	crystallized
Dairy	dairy	saltiness	waxy, unctuous
gum/tire	chemical	bitterness	
dried fruit	soap		
Animal			
Chemical			

Table 3. Physicochemical properties of ling-heather honeys used to obtain honey powder (n=3, except sugars n=1)

	LH1	LH2	LH3
Moisture (%)	16.72 ± 0.12$_b$	17.54 ± 0.05$_c$	16.04 ± 0.23$_a$
Aw	0.624 ± 0.044$_b$	0.605 ± 0.004$_b$	0.540 ± 0.008$_a$
Fructose (%)	37.14	34.59	32.16
Glucose (%)	34.55	33.52	27.89
Sucrose (%)	2.70	0.24	2.63
Conductivity (mS)	0.647 ± 0.002$_b$	1.012 ± 0.006$_c$	0.332 ± 0.001$_a$
pH	4.05 ± 0.01$_b$	4.14 ± 0.02$_c$	3.87 ± 0.03$_a$
Free acidity (meq/kg)	49.77 ± 3.05$_a$	49.70 ±2.49$_a$	41.02 ± 6.56$_a$
Lactone acidity (meq/kg)	0.00 ± 0.00$_a$	0.23 ±0.05$_a$	0.00 ± 0.00$_a$
Formol index (meq/100 g)	0.61 ± 0.05$_a$	0.74 ± 0.01$_a$	0.93 ±0.13$_a$
Diastase	3.93 ± 0.07$_a$	13.55 ± 0.35$_b$	23.55 ± 1.49$_c$
HMF (mg/kg)	0.00 ± 0.00$_a$	0.00 ± 0.00$_a$	18.01 ± .43$_b$
Proline (mg/100 g H)	74.33 ± 3.72$_a$	107.24 ± 4.85$_b$	73.06 ± 0.75$_a$
L*	24.77 ± 0.03$_a$	25.66 ± 0.03$_b$	44.61 ± 0.01$_c$
a*	16.54 ± 0.02$_b$	16.61 ± 0.03$_c$	10.17 ± 0.01$_a$
b*	33.15 ± 0.07$_a$	34.49±0.11$_b$	40.42 ± 0.03$_c$

a-c: different letters showed significant differences ($p<0.05$) between honeys

Table 4. Glass transition temperature T_g (ºC) of raw honeys (LH1, LH2, LH3) and honey powders (LHP1) obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD).

Sample	**T_g1 ($T_{MIDPOINT}$°C)**	**T_g2 ($T_{MIDPOINT}$°C)**
LH1	-27.66	-
LH2	-29.81	-
LH3	-27.33	
SP-AG-LHP1	49.86	-
SP-WH-LHP1	13.31	64.54
SP-MD-LHP1	25.44	90.58
VC-AG-LHP1	13.62	80.09
VC-WH-LHP1	22.20	71.08
VC-MD-LHP1	25.99	91.34
FZ-AG-LHP1	19.34	81.26
FZ-WH-LHP1	14.32	66.52
FZ-MD-LHP1	18.47	88.82

-: Absent (there is no T_g2)

Table 5. Colour parameters (L*, a* and b*) of carriers and powder honeys obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) (n=3).

						L*				
			LHP1			LHP2			LHP3	
		SP	VC	FZ	SP	VC	FZ	SP	VC	FZ
AG	89.52±0.11	^{C}88.50±0.02$_{b}$	^{A}59.74±0.14$_{c}$	^{B}73.29±0.01$_{c}$	^{C}88.42±0.02$_{c}$	^{A}63.73±0.02$_{c}$	^{B}71.80±0.06$_{c}$	^{C}93.79±0.06$_{c}$	^{B}79.06±0.02$_{c}$	^{A}75.84±0.04$_{a}$
WH	58.59±0.07	^{C}88.31±0.03$_{b}$	^{A}51.94±0.35$_{a}$	^{B}67.22±0.02$_{a}$	^{C}87.70±0.05$_{b}$	^{A}50.48±0.13$_{a}$	^{B}66.89±0.12$_{a}$	^{C}93.14±0.03$_{b}$	^{A}57.88±0.02$_{a}$	^{B}78.31±0.04$_{b}$
MD	95.50±0.19	^{C}79.78±0.05$_{a}$	^{A}58.72±0.16$_{b}$	^{B}70.99±0.03$_{b}$	^{C}84.86±0.03$_{a}$	^{A}63.43±0.03$_{b}$	^{B}69.46±0.17$_{b}$	^{C}92.31±0.04$_{a}$	^{A}76.98±0.02$_{b}$	^{B}83.05±0.03$_{c}$
						a*				
			LHP1			LHP2			LHP3	
		SP	VC	FZ	SP	VC	FZ	SP	VC	FZ
AG	-0.68±0.01	^{A}-0.15±0.01$_{b}$	^{C}7.56±0.04$_{a}$	^{B}3.87±0.01$_{a}$	^{A}-0.47±0.02$_{a}$	^{C}6.11±0.01$_{a}$	^{B}4.55±0.05$_{a}$	^{A}-1.31±0.01$_{b}$	^{C}2.60±0.01$_{a}$	^{B}0.03±0.03$_{a}$
WH	-0.35±0.01	^{A}-0.28±0.01$_{a}$	^{C}13.20±0.14$_{c}$	^{B}5.50±0.01$_{b}$	^{A}-0.26±0.01$_{b}$	^{C}13.18±0.05$_{c}$	^{B}5.72±0.05$_{b}$	^{A}-1.19±0.01$_{c}$	^{C}12.55±0.01$_{c}$	^{B}2.40±0.01$_{c}$
MD	-1.64±0.01	^{A}2.43±0.01$_{c}$	^{C}8.46±0.10$_{b}$	^{B}5.74±0.02$_{c}$	^{A}0.95±0.01$_{c}$	^{C}7.84±0.01$_{b}$	^{B}6.04±0.09$_{c}$	^{A}-1.59±0.01$_{a}$	^{C}2.73±0.00$_{b}$	^{B}1.15±0.02$_{b}$
						b*				
			LHP1			LHP2			LHP3	
		SP	VC	FZ	SP	VC	FZ	SP	VC	FZ
AG	9.72±0.00	^{A}12.18±0.03$_{a}$	^{C}29.26±0.11$_{a}$	^{B}24.71±0.00$_{a}$	^{A}11.89±0.03$_{a}$	^{C}28.65±0.03$_{a}$	^{B}27.20±0.09$_{a}$	^{A}5.64±0.02$_{a}$	^{C}22.33±0.01$_{a}$	^{B}20.94±0.04$_{a}$
WH	17.34±0.03	^{A}13.49±0.01$_{b}$	^{C}34.70±0.07$_{c}$	^{B}26.22±0.05$_{b}$	^{A}14.13±0.03$_{b}$	^{C}32.96±0.22$_{c}$	^{B}27.73±0.13$_{b}$	^{A}7.81±0.01$_{c}$	^{C}35.56±0.01$_{c}$	^{B}22.49±0.07$_{b}$
MD	1.16±0.02	^{A}21.87±0.02$_{c}$	^{C}30.99±0.26$_{b}$	^{B}28.40±0.03$_{c}$	^{A}18.26±0.04$_{c}$	^{C}32.60±0.03$_{b}$	^{B}29.05±0.19$_{c}$	^{A}7.01±0.02$_{b}$	^{C}27.37±0.02$_{b}$	^{B}20.92±0.31$_{a}$

A-C: Different letters showed significant differences ($p<0.05$) between drying procedures for each honey and carrier.
a-c: different letters showed significant differences ($p<0.05$) between carries for each honey and drying procedure.

Table 6. Moisture (%) and water activity of carriers and powder honeys obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) (n=3).

Moisture										
		LHP1			**LHP2**			**LHP3**		
		SP	**VC**	**FZ**	**SP**	**VC**	**FZ**	**SP**	**VC**	**FZ**
AG	7.51±0.33	^{A}4.63±0.47$_{a}$	^{A}3.43±0.12$_{b}$	^{A}4.09±0.68$_{b}$	^{A}3.00±0.11$_{a}$	^{B}4.52±0.13$_{b}$	^{B}4.59±0.27$_{b}$	^{B}4.87±0.20$_{b}$	^{A}2.74±0.56$_{a}$	^{B}5.12±0.36$_{b}$
WH	1.33±0.20	^{B}7.27±0.23$_{b}$	^{A}5.74±0.28$_{b}$	^{B}7.40±0.61$_{c}$	^{B}7.02±0.16$_{c}$	^{A}5.35±0.66$_{b}$	^{A}5.99±0.19$_{c}$	^{C}7.48±0.36$_{c}$	^{A}3.91±0.46$_{b}$	^{B}5.67±0.13$_{b}$
MD	2.34±0.27	^{B}4.08±0.10$_{a}$	^{A}2.61±0.26$_{a}$	^{A}2.21±0.16$_{a}$	^{C}3.80±0.01$_{b}$	^{B}2.91±0.21$_{a}$	^{A}1.90±0.00$_{a}$	^{A}1.92±0.09$_{a}$	^{A}2.12±0.09$_{a}$	^{B}3.42±0.09$_{a}$
aw										
		LHP1			**LHP2**			**LHP3**		
		SP	**VC**	**FZ**	**SP**	**VC**	**FZ**	**SP**	**VC**	**FZ**
AG	0.324±0.003	^{A}0.263±0.001$_{b}$	^{B}0.317±0.021$_{ab}$	^{A}0.254±0.004$_{b}$	^{A}0.211±0.001$_{a}$	^{B}0.250±0.005$_{b}$	^{C}0.304±0.002$_{c}$	^{A}0.236±0.001$_{a}$	^{B}0.257±0.007$_{a}$	^{C}0.315±0.001$_{c}$
WH	0.120±0.001	^{A}0.256±0.002$_{a}$	^{B}0.297±0.001$_{a}$	^{A}0.253±0.007$_{b}$	^{B}0.257±0.001$_{b}$	^{A}0.240±0.000$_{a}$	^{C}0.263±0.002$_{b}$	^{B}0.260±0.002$_{b}$	^{B}0.260±0.003$_{a}$	^{A}0.250±0.002$_{a}$
MD	0.204±0.001	^{C}0.342±0.002$_{c}$	^{B}0.337±0.001$_{b}$	^{A}0.211±0.002$_{a}$	^{A}0.257±0.001$_{b}$	^{B}0.271±0.000$_{c}$	^{A}0.250±0.006$_{a}$	^{B}0.267±0.001$_{c}$	^{C}0.318±0.001$_{b}$	^{A}0.261±0.001$_{b}$

A-C: Different letters showed significant differences ($p<0.05$) between drying procedures for each honey and carrier.
a-c: different letters showed significant differences ($p<0.05$) between carries for each honey and drying procedure.

Table 7. Proline (mg/100g of powder in dry matter) of carriers and powder honeys obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) (n=3).

		LHP1			**LHP2**			**LHP3**		
		SP	**VC**	**FZ**	**SP**	**VC**	**FZ**	**SP**	**VC**	**FZ**
AG	25.62±2.23	^{A}31.48±1.75$_{b}$	^{A}29.45±0.97$_{b}$	^{B}42.44±0.56$_{b}$	^{B}50.73±1.37$_{b}$	^{A}37.05±1.05$_{b}$	^{C}65.68±1.75$_{a}$	^{A}25.81±2.37$_{a}$	^{B}31.88±1.78$_{b}$	^{C}43.53±1.02$_{b}$
WH	75.70±5.04	^{B}60.78±0.86$_{c}$	^{A}31.16±1.11$_{b}$	^{B}65.34±3.65$_{c}$	^{B}88.16±1.21$_{c}$	^{A}38.61±1.42$_{b}$	^{C}108.99±3.81$_{b}$	^{C}65.95±2.37$_{b}$	^{A}29.43±0.72$_{b}$	^{B}56.82±2.05$_{c}$
MD	8.21±0.23	^{B}25.40±0.11$_{a}$	^{A}21.96±0.88$_{a}$	^{C}36.03±1.05$_{a}$	^{B}38.16±1.80$_{a}$	^{A}24.75±0.50$_{a}$	^{C}67.70±0.17$_{a}$	^{A}21.39±1.44$_{a}$	^{A}19.56±1.01$_{a}$	^{B}35.73±1.51$_{a}$

A-C: Different letters showed significant differences ($p<0.05$) between drying procedures for each honey and carrier.
a-c: different letters showed significant differences ($p<0.05$) between carries for each honey and drying procedure.

Table 8. Fructose and glucose obtained by CG-MS. expressing in dry matter (g/100 g of dry powder) of carriers and powder honeys obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) (n=1).

Fructose										
		LHP1			LHP2			LHP3		
		SP	VC	FZ	SP	VC	FZ	SP	VC	FZ
AG	0.36	29.41	32.18	27.61	28.74	24.46	27.13	27.62	32.96	27.98
WH	0.00	28.99	31.82	31.15	27.97	28.76	30.21	29.71	25.57	31.68
MD	0.00	27.40	26.00	30.43	23.88	32.21	26.47	25.63	24.16	32.13
Glucose										
		LHP1			LHP2			LHP3		
		SP	VC	FZ	SP	VC	FZ	SP	VC	FZ
AG	0.25	24.68	27.52	21.53	25.46	29.74	21.38	23.57	29.43	21.91
WH	0.00	22.77	26.19	24.30	22.69	27.91	24.10	25.16	27.90	27.06
MD	0.00	25.38	21.83	26.50	23.26	32.93	27.15	26.58	25.13	28.36

Figure 1.

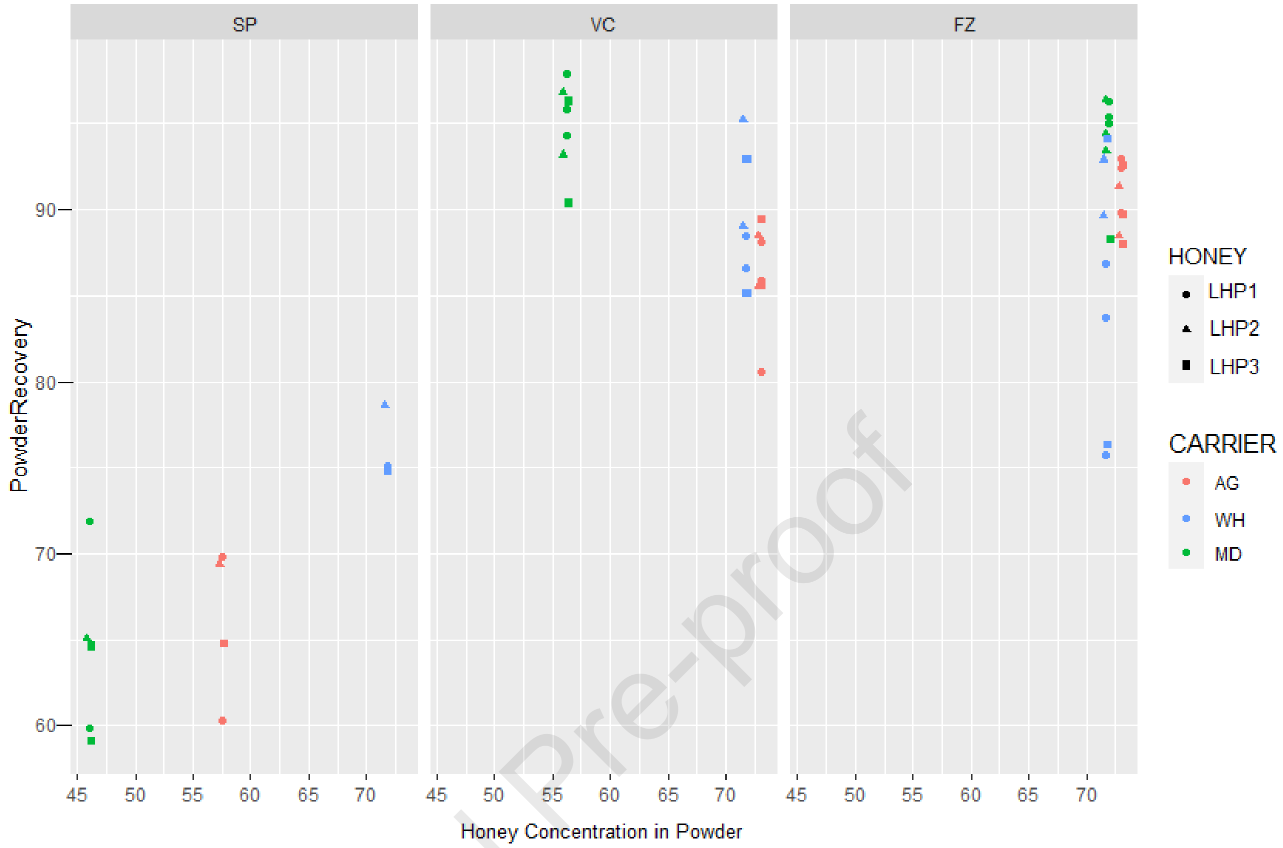
SP
VC
FZ
PowderRecovery
90
80
70
60
45
50
55
60
65
70
Honey Concentration in Powder
HONEY
LHP1
LHP2
LHP3
CARRIER
AG
WH
MD

Figure 2.

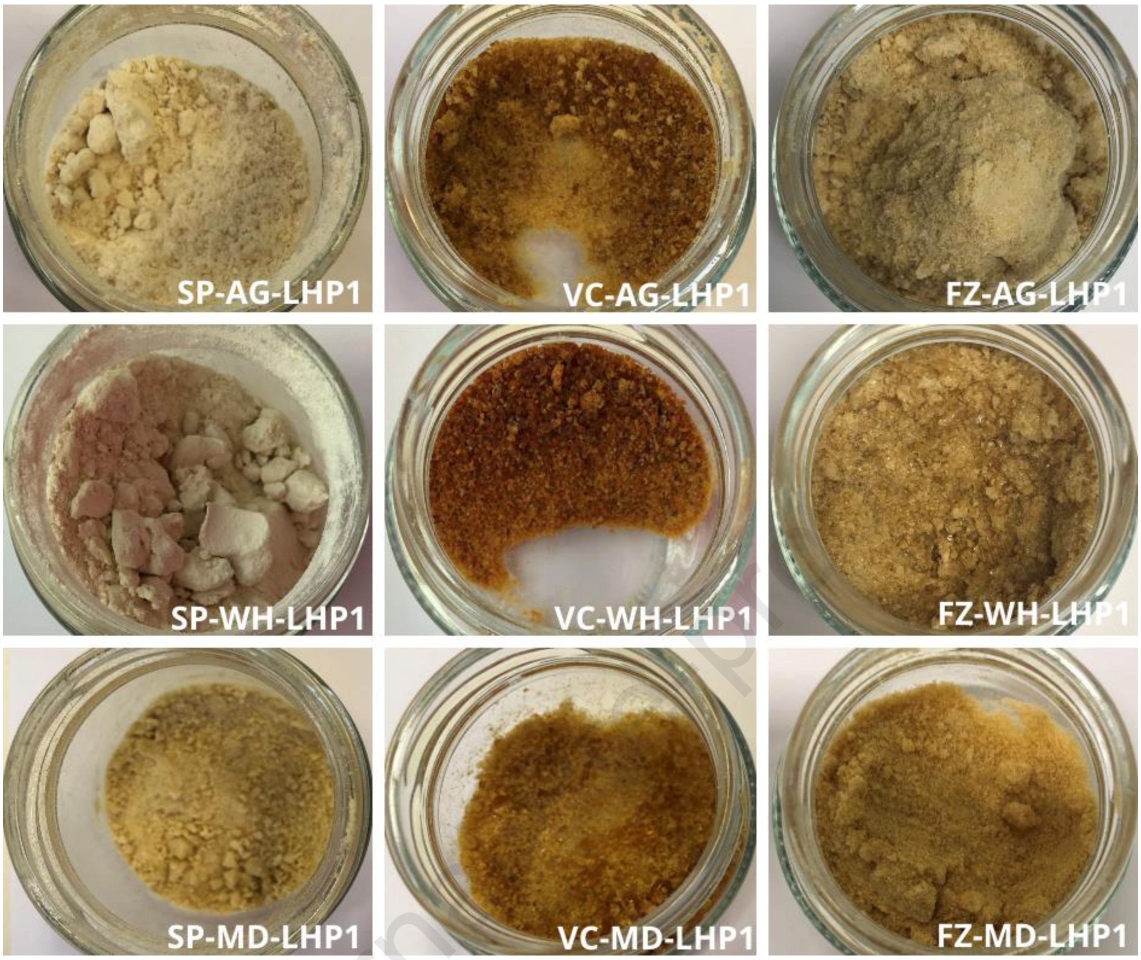
SP-AG-LHP1
VC-AG-LHP1
FZ-AG-LHP1
SP-WH-LHP1
VC-WH-LHP1
FZ-WH-LHP1
SP-MD-LHP1
VC-MD-LHP1
FZ-MD-LHP1

Figure 3.

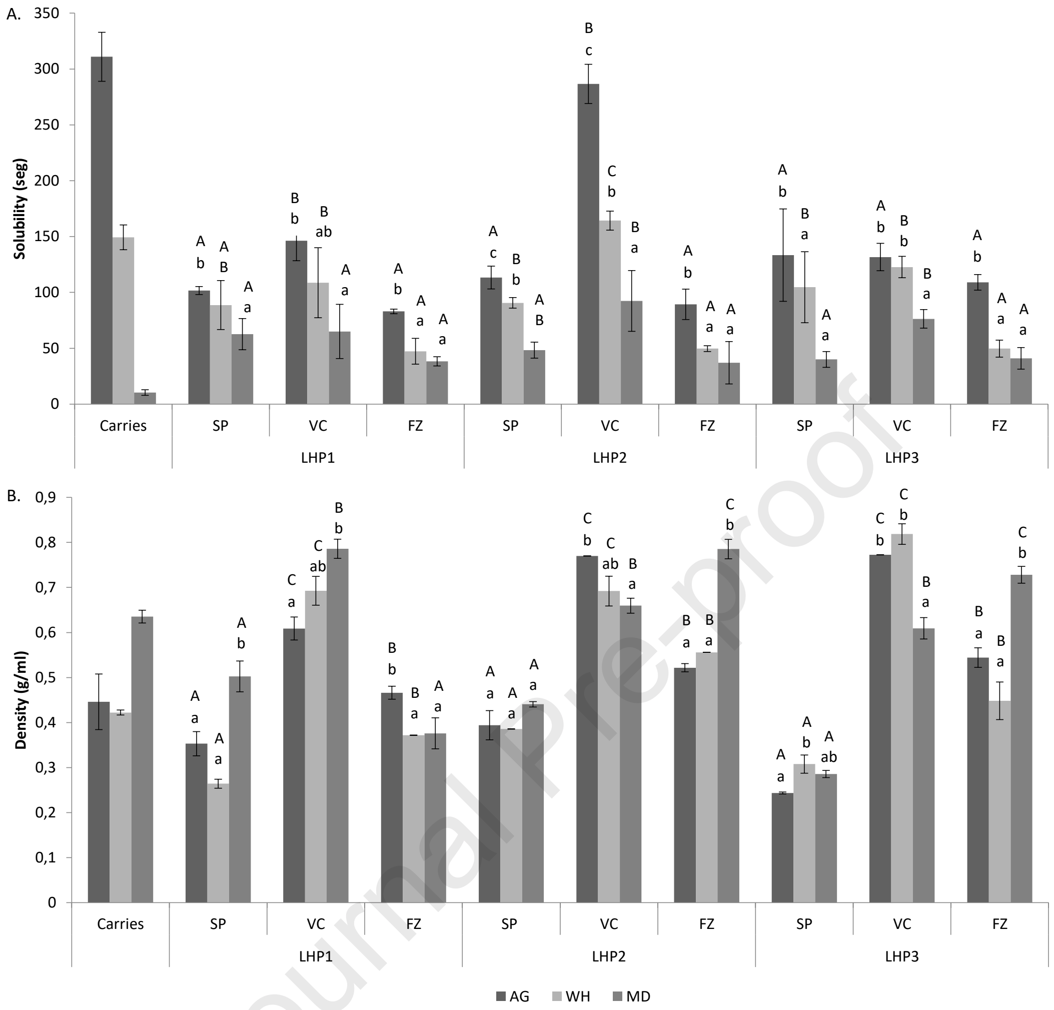
A.
Solubility (seg)
Carries
SP
VC
FZ
LHP1
LHP2
LHP3
B.
Density (g/ml)
AG
WH
MD

Figure 4.

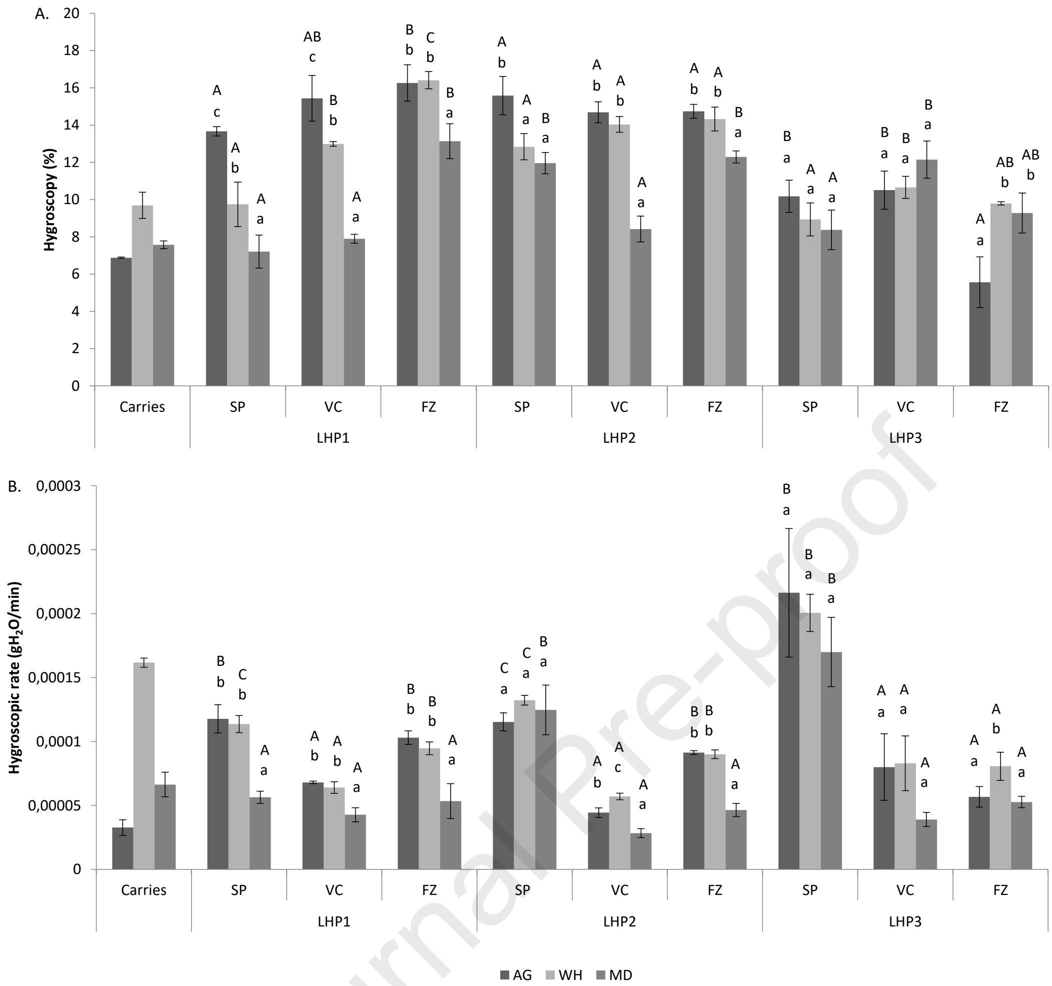
A.
Hygroscopy (%)
20
18
16
14
12
10
8
6
4
2
0
Carries
SP
VC
FZ
LHP1
LHP2
LHP3
B.
Hygroscopic rate (gH2O/min)
0,0003
0,00025
0,0002
0,00015
0,0001
0,00005
0
AG
WH
MD

Figure 5.

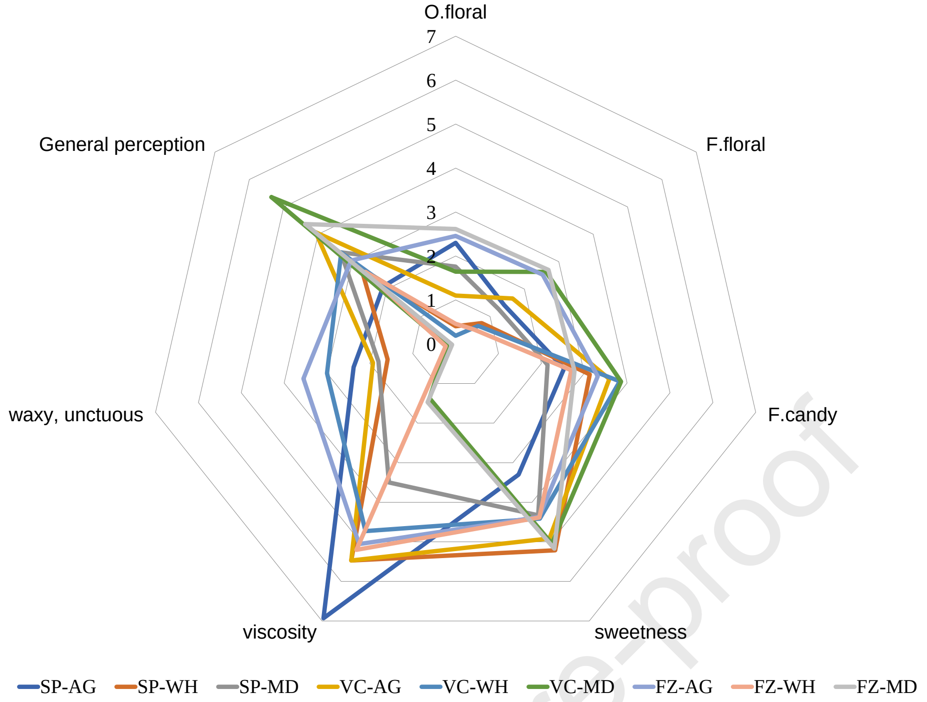
O.floral
7
6
5
4
3
2
1
0
F.floral
F.candy
sweetness
viscosity
waxy, unctuous
General perception
SP-AG
SP-WH
SP-MD
VC-AG
VC-WH
VC-MD
FZ-AG
FZ-WH
FZ-MD

Figure 6.

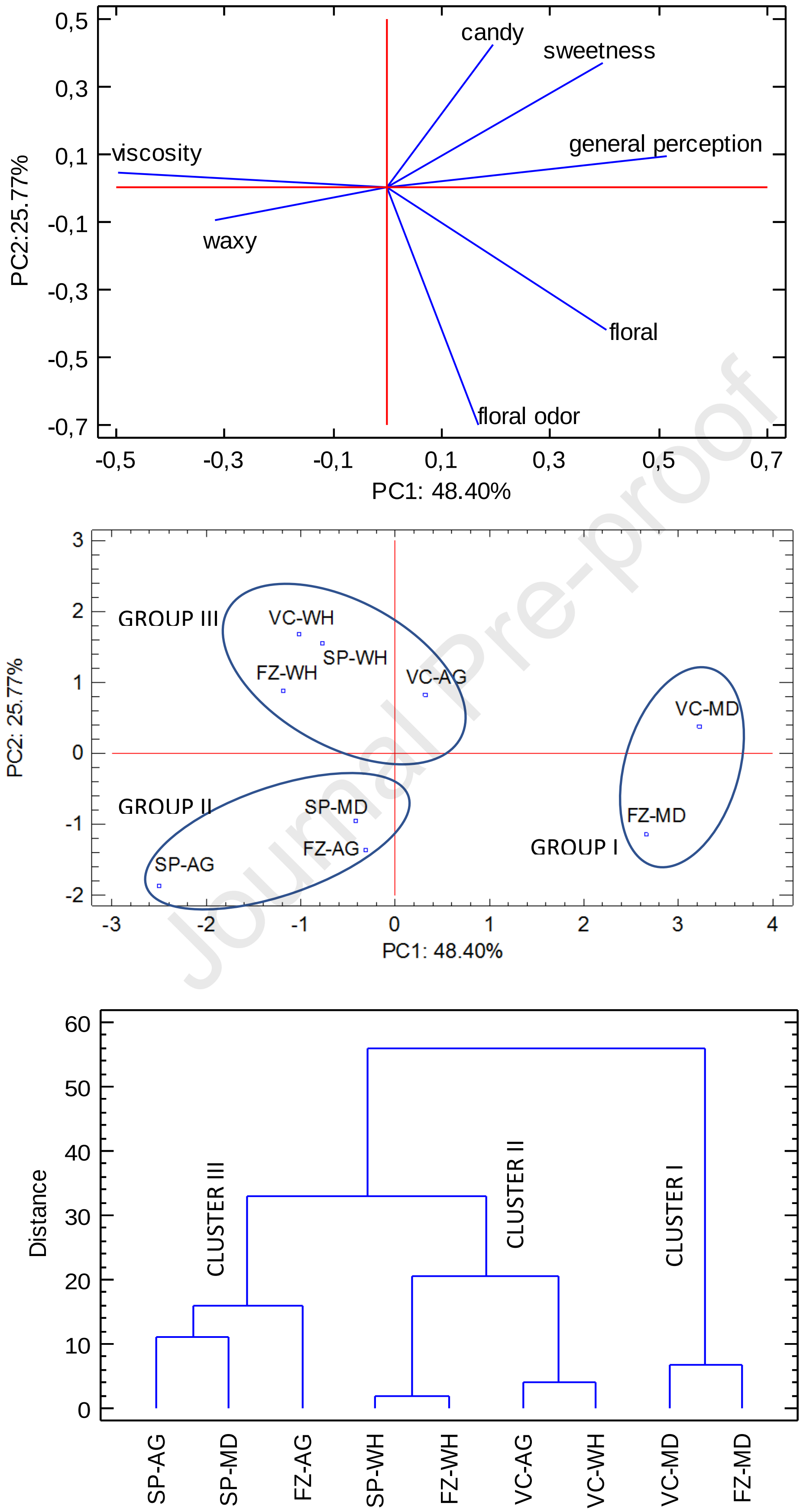
candy
sweetness
general perception
viscosity
waxy
floral
floral odor
PC2:25.77%
PC1: 48.40%
0,5
0,3
0,1
-0,1
-0,3
-0,5
-0,7
-0,5
-0,3
-0,1
0,1
0,3
0,5
0,7
GROUP III
VC-WH
SP-WH
FZ-WH
VC-AG
VC-MD
GROUP II
SP-MD
FZ-AG
SP-AG
FZ-MD
GROUP I
PC2: 25.77%
PC1: 48.40%
3
2
1
0
-1
-2
-3
-2
-1
0
1
2
3
4
Distance
60
50
40
30
20
10
0
CLUSTER III
CLUSTER II
CLUSTER I
SP-AG
SP-MD
FZ-AG
SP-WH
FZ-WH
VC-AG
VC-WH
VC-MD
FZ-MD

Figure captions

Figure 1. Diagrame of relation between powder recovery and honey quantity of powder honey samples obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD). Graphical done with R-Studio.

Figure 2. Pictures of ling-heater powders (LH1) obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD).

Figure 3. Solubility (s) and density (g/ml) of ling-heather powders (LHP) obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) as carries (n=3, density n=2). Error bars represent the standard deviation for each data point. A-C: Different letters showed significant differences ($p<0.05$) between drying procedures for each honey and carrier; a-c: different letters showed significant differences ($p<0.05$) between carries for each honey and drying procedure.

Figure 4: Hygroscopicity (%) and hygroscopic rate (gH_2O/min) of ling heather powders (LHP) obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) as carries (n=3). Error bars represent the standard deviation for each data point. A-C: Different letters showed significant differences ($p<0.05$) between drying procedures for each honey and carrier; a-c: different letters showed significant differences ($p<0.05$) between carries for each honey and drying procedure.

Figure 5. Diagram that represent the mean (n=7) of the sensorial descriptive analyses performed by experts, in LHP1 obtained by spray (SP), vacuum (VC) and freeze (FZ) drying, using Arabic gum (AG), whey (WH) and maltodextrin (MD) as carries.

Figure 6. PCA and dendrogram plot derived from hierarchical cluster analysis for sensorial attributes

Highlights

Spray, vacuum, freeze drying and three carriers were assessed to make honey powders.

At lab scale vacuum and freeze powders with maltodextrin showed the highest recoveries.

Honey powders with maltodextrin showed lowest moisture and highest solubility.

Powders with maltodextrin showed highest tapped density and lower hygroscopicity.

Vacuum and freeze powders with maltodextrin were the most appealing.

Declaration of interests

☒ 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.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: