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Effect of Foam-mat Drying on Bioactive, Powder and Thermal Properties of Carrot Juice Powders

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Abstract: Foam-mat drying is a promising method due to involvement of proteins as foaming agent which acts like a wall material wrapping around bioactives and retards their degradation. In this study, powder properties of foam-mat dried carrot juice powders including 15% egg albumen (EA) and 15% egg albumen+ 10% whey protein isolate (WPI) during 90 days of storage at room conditions were evaluated by means of physical, chemical, thermal (DSC and TGA) and microstructural (SEM) analyses. The powder flow properties (wettability, hygroscopicity, degree of caking, Carr index and Hausner ratio) have been significantly affected from the moisture uptake during storage; however, powder flow properties of 15% EA+ 10% WPI powders were found to be better than 15% EA powders as it was also confirmed by particle diameter distributions that remained the same before and after the storage period. Besides, the total antioxidant, phenolic and carotenoid contents of 15% EA+ 10% WPI carrot powder was found comparably higher than 15% EA powders due to possible encapsulating mechanism of whey proteins. In addition to delaying of physical and chemical deteriorations in powders with WPI incorporation, thermal stability of the foam-mat dried carrot juice powders was also improved in 15% EA+ 10% WPI powder.

Key words: Carrot, foam, whey protein, powder, carotenoid, microstructure.

INTRODUCTION

Fruit and vegetables have limited shelf life and exposed to microbial spoilage due to their low acidity and high moisture content (above 85%) (Demir et al. 2007). Also, vegetables are susceptible to enzymatic activity that further results in an unavoidable deterioration of product quality (Ma et al. 2013, Franco et al. 2016). Root vegetables especially carrot has moderate to high respiration rates; therefore, mild or conventional food processing methods like modified atmosphere packaging, vegetable juice production including thermal treatment, freezing, canning, pickling or drying are employed to extend the shelf life (Sharma et al. 2009, Sinha et al. 2011, Yilmaz et al. 2019). Drying of carrots with different methods is extensively studied; however, foam-mat drying of carrot juice and characterization of the carrot juice powder quality during storage has not yet been studied.

Foam-mat drying is a two-step drying process; firstly, the food material to be dried (in liquid, semi-liquid or paste form) is rigorously mixed and foamed with the addition of foaming and stabilizing agents such as; egg albumin, whey protein isolate, faba bean protein concentrate, soy bean protein isolate, methyl cellulose, carboxymethyl cellulose or commercial stabilizers (Kudra & Ratti 2006, Asokapandian et al. 2016, Chaux-Gutiérrez et al. 2017, Hardy & Jideani 2017, Darniadi et al. 2018, Cakmak 2020, Cakmak & Ozyurt 2021a, de Cól et al. 2021, Kanha et al. 2022, Nunes et al. 2022). In the second step, foamed material is gently transferred to a surface of a tray or platform and is

dried in a tray dryer, freeze dryer or microwave dryer. The advantages of foam-mat drying method are homogeneous heating of the food material, accelerating the moisture transfer by increased surface area with air incorporation into the foam and increasing energy efficiency of drying (Kudra & Ratti 2006, Asokapandian et al. 2016, Hardy & Jideani 2017). With foam-mat drying method, the product quality is improved compared to other conventional drying methods or is preserved better by foam-mat drying method in terms of product yield, powder flowability, solubility, rehydration behavior, antioxidant retention, color and thus final product quality (Chaux-Gutiérrez et al. 2017, Seerangurayar et al. 2017, Darniadi et al. 2018, Cakmak 2020, de Cól et al. 2021). In addition, fruit juices or pulps with appreciable sugar content like bacaba, blueberry, cantaloupe, crab apple, date, mango or muskmelon were successfully dried by foam-mat drying (Wilson et al. 2014, Asokapandian et al. 2016, Hardy & Jideani 2017, Salahi et al. 2017, Seerangurayar et al. 2017, Darniadi et al. 2018, Cakmak 2020, de Cól et al. 2021).

Foam-mat drying is generally carried out between 50 to 70°C for direct air drying methods or from -40 to -30°C for freeze drying applications. Therefore, this method helps to retain the bioactive compounds compared to the alternative drying applications like spray drying because preserving phenolic compounds of the fruit or vegetable powders are quite challenging due to high drying temperatures. Drying of sugar rich fruit or vegetables is especially problematic; however, low temperature employment in foam-mat drying improves not only rehydration ability of the powder but also produce powders with a desirable flowability, density, and solubility (Kudra & Ratti 2006, Wilson et al. 2014, Seerangurayar et al. 2017). In the study of Wilson et al. (2014), the carotenoids of foam-mat dried mango powder was preserved during six-month storage period, while the HMF and non-enzymatic browning in powders were minimized. There are several reports on use of foam-mat for drying of plant-based foods. Reis et al. (2021) reviewed in their study that different drying periods, temperature, foaming agents, foaming conditions, foam thickness were applied for extending shelf life of plant based foods and retention of bioactive compounds naturally found in them were directly related with the aforesaid parameters. To best of our knowledge, the effects of usage of different foaming agents namely egg albumen alone and together with whey proteins in the foam-mat drying on the phenolic compound, carotenoids and the powder properties of carrot powders during their have not been studied before.

Thus, the aim of this study is to produce carrot juice powders by foam-mat drying method with the addition of 15% EA alone and 15% EA with 10% WPI and to evaluate the storage stability and powder properties by means of physical, chemical, thermal and microstructural analyses.

Abbreviations

CI	Carr index
DPPH	2,2-diphenyl-1-picrylhydrazyl
EA	Egg albumen
HMF	5-hydroxymethyl furfural
HR	Hausner ratio
WPI	Whey protein isolate powder

MATERIALS AND METHODS

Materials

Fresh carrots (*Daucus carota* var. Nantes) and whole eggs were purchased from a local supermarket in Corum, Turkey and the samples were kept in refrigerator at 4°C prior to the analysis. Whey protein isolate with 96% protein was purchased from local distributor (Hipro Iso whey, Bionet Tic. A.S., Istanbul).

Synthetic β-carotene standard (C4582, Sigma-Aldrich, Germany), Folin-Ciocalteau phenol reactive (E9252, Sigma-Aldrich, Germany), DPPH radical (2,2-diphenyl-1-picrylhydrazyl) (D9132, Sigma-Aldrich, Germany), Na_2CO_3 (2024, J.T. Baker, USA), acetone (100983, Merck, Germany), methanol (34885, Sigma-Aldrich, Germany), petroleum ether (24587, Sigma-Aldrich, Germany), sodium chloride (S9888, Sigma-Aldrich, Germany) and sodium sulfate (13464, Sigma-Aldrich, Germany) were purchased from the local distributors. Transparent polypropylene-polyamid-polyethylene composite films with a thickness of 175 µm used for packaging of the carrot powders were kindly donated by Superfilm (Medguard NB 3011 EMH, Turkey).

Production of carrot juice

Fresh carrots were cleaned and juice was extracted according to the previous study of Cakmak & Ozyurt (2021a). Extracted juice was filled into glass bottles and treated at 95°C for 5 min (Demir et al. 2007) in a water bath (Wise Bath, WB22, Daihan Scientific, South Korea), and cooled to 4°C. Carrot juices were kept in PET bottles in a freezer (Regal, 4542 A NF, Turkey) at -18°C until all analyses.

Production of foam-mat dried carrot juice powders

In the previous study about foam stability of carrot juice (Cakmak & Ozyurt 2021a), the most stable foam structure was found to be the 15% EA+ 10% WPI foam formulation which was mixed at the highest speed with a hand-blender (Arzum Pasto AR-183, Turkey) for 8 min whipping time. In addition to this formulation, 15% EA including foams were prepared similar to previously given foaming conditions.

The foams were spread evenly on Petri dishes (OD: 90 mm) with 6 mm thickness and dried at 60°C preheated built-in oven (Model no: NV60K7140BB, Samsung, Turkey) with upper-lower heating function at 0.9 m/s steady air velocity until constant weight was observed. Drying was performed in duplicate and dried foams were removed from petri dishes manually and grinded into fine powder by a grinder (Fakir Aromatic, Germany). The carrot powders were then placed in transparent 175 µm polypropylene-polyamid-polyethylene composite film (Superfilm, Medguard NB 3011 EMH, Turkey) sachets that were prepared with a heat sealer (Impulse Sealer PFS-400, Bonjay Technology Co., China). In order to determine the possible variations of powder properties, these sachets were stored at room temperature in the dark for 3 months, and the storage analyses were performed on the initial day (Day 0), 15th, 30th, 60th and 90th day of storage period.

Powder properties

Moisture

The moisture content of the carrot juice, egg albumen and dried carrot powders were analysed using drying oven (Memmert, UN55, Germany) according to the standard methods of AOAC (2000).

Wettability

Wettability of carrot powders was determined according to the method of Gong et al. (2007) with some modifications. 0.1 g powder was carefully placed in a beaker containing 100 mL distilled water and duration of the powder becoming completely soaked was recorded in seconds.

Hygroscopicity

%Hygroscopicity of foam-mat dried powders was determined according to the method of Goula & Adamopoulos (2008). 1 g of powder was spread evenly on Petri dishes, and the dishes were placed in a dessicator conditioned to 23°C and 75.36±0.3% RH using saturated sodium chloride solution. The dishes were weighed every 10 min of 90 min storage period. The final hygroscopicity values of the powder samples were compared.

Degree of caking

The samples after hygroscopicity analysis performed were used for the degree of caking analysis (Goula & Adamopoulos 2008). The moisture absorbed samples were dried at 70°C in an oven for 3h (Memmert, UN55, Germany). Following drying, the Petri dishes were weighed and screened through 500 µm sieve. The degree of caking was calculated with respect to the following equation:

$$\textit{degree of caking} = \frac{b \times 100}{a} \quad (1)$$

where a is the powder used in g, and b is the powder retained on the sieve in g.

Bulk density, tapped density, Carr index, and Hausner ratio

Bulk and tapped densities of the powders were determined according to Jinapong et al. (2008). In order to classify the powders according to their flowability, cohesiveness and stickiness, Carr index (CI) and Hausner ratio (HR) were calculated by the following equations:

$$CI\left(\%\right) = \frac{\left(\rho_{tapped} - \rho_{bulk}\right)}{\rho_{tapped}} \times 100 \quad (2)$$

$$HR = \frac{\rho_{tapped}}{\rho_{bulk}} \quad (3)$$

where ρ_{bulk} represents the bulk density of powder in g/mL, and ρ_{tapped} represents tapped density of the powder in g/mL.

Flowability of carrot powders was compared based on CI values, and powders are classified accordingly: CI< 15%: very good flowability, 15-20%: good, 20-35%: fair, 35-45%: bad and CI> 45%: very bad flowability (Jinapong et al. 2008). Cohesiveness of the powders was characterized by Hausner ratio: 1.0 < HR < 1.1: considered as free flowing powder (low cohesiveness), 1.1 < HR < 1.25: medium flowing powder, 1.25 < HR < 1.4: intermediate cohesiveness (hard to flow) and HR > 1.4: high cohesiveness (problem in fluidization) (Barbosa-Canovas et al. 2005, Jinapong et al. 2008, Seerangurayar et al. 2017). The powder property analyses were performed at least in triplicate and the average of them was reported.

Particle diameter distribution

The particle size of foam-mat dried powders before and after the storage period was determined gravimetrically by using ASTM-E11 standard set of sieves (Jeotest, JG035/1, Turkey). 5 g of powder was placed on the top sieve and shaken for 10 min. The mass retained on each sieve was recorded and the diameter distribution was calculated. The average of triplicate measurements was given.

Color analysis

Color values of the samples were measured with a spectrophotometer (Konica Minolta, CM3600D, Japan) using a 8 mm (measurement diameter) target mask attached with illuminant D65 and 10° observer according to CIE L*a*b* color space. In this space, the lightness is expressed by L* ranging from L*=0 being black, and L*=100 being white, and -a* being green, +a* being red, -b* values represents blue and +b* represents yellow. Hue angle (h*), chroma (C*) and total color difference (ΔE*) were calculated using the following equations:

$$h^* = \arctan\left(\frac{b^*}{a^*}\right) \tag{4}$$

$$C^* = \sqrt{(a^*)^2 + (b^*)^2} \tag{5}$$

$$\Delta E^* = \sqrt{\left(L^*_{sample} - L^*_0\right)^2 + \left(a^*_{sample} - a^*_0\right)^2 + \left(b^*_{sample} - b^*_0\right)^2} \tag{6}$$

Here subscript “0” refers to the carrot juice, and sample subscript refers to the carrot powder at any storage time. In order to evaluate the possible differences in color values depending on the foam thickness and drying temperature elevation, the aforementioned foam formulations were dried at 50, 60 and 70°C with 5 mm and 6 mm thicknesses. The average of at least 6 measurements was reported in the study.

Total carotenoid content

The method of Rodriguez-Amaya (2001) has been modified for measuring the carotenoid content of the powders. Firstly, 1 g of sample was homogenized with 5 mL of cold acetone. The mixture was filtered using coarse filter paper and washed with 10 mL of cold acetone until the filter paper became colorless. Then the resulting mixture was transferred to a separatory funnel with 8 mL of previously added petroleum ether. To trigger saponification, 60 mL of distilled water was added to the mixture. The lower phase was removed and distilled water was added to the separation funnel again. This process was repeated thrice. Using glass wool and sodium sulfate, the remaining mixture was filtered in the separation funnel to remove the rest of the distilled water. The absorbance of the obtained liquid was read at 450 nm wavelength and calculated using the following formula:

$$\textit{Total carotenoid content} = \frac{\textit{Absorbance value} \times \textit{Sample volume (ml)} \times 10^4}{2592 \times \textit{Sample amount (g)}} \tag{7}$$

Here the constant 2592 represents the absorption coefficient of β-carotene in petroleum ether, which is commonly found in carrot and the results were expressed as μg β-carotene/g DM.

Antioxidant activity

Antioxidant activity of the powder product was determined according to DPPH radical scavenging activity method developed by Brand-Williams et al. (1995). The samples were dissolved into 10 mL of methanol. 1 mL of the methanol extracts of the samples and 0.5 mL of the DPPH solution were incubated for 20 min at room temperature in a dark environment. At the end of the period, absorbance was read in a UV-Vis spectrophotometer (Varian Cary 50 Bio, Australia) at the wavelength of 520 nm. Antioxidant activity is expressed as Inhibition %, and is calculated using the following equation:

$$Inhibition\ (\%) = \frac{A_0 - A_1}{A_0} \times 100 \quad (8)$$

Here A_0 is the absorbance of the control, and A_1 is the absorbance of the extractives or standard.

Total phenolics

The amount of total phenolics was analyzed using the Folin-Ciocalteau (FC) method (Singleton & Rossi 1965). According to the method, 50 µL of sample extracts were dissolved in methanol and 250 µL Folin-Ciocalteau reagent were kept at room temperature for 5 min. Then, 750 µL of saturated Na_2CO_3 solution was added and the mixture was made up to 5 mL with distilled water. The mixture was kept at room temperature in the dark for 120 min and then absorbances were measured at 765 nm in a spectrometer (Varian Cary 50 Bio, Australia). The results were expressed as gallic acid equivalent (mg GAE/g DM). The total carotenoid content, antioxidant activity and total phenolics experiments were performed in triplicate.

Thermal properties of powders

The glass transition temperature (Tg) of the powders were analysed by a differential scanning calorimeter (DSC-1/700, Mettler Toledo, Switzerland) according to the study of Janiszewska-Turak et al. (2017) with some modifications. An empty aluminium pan was used as a reference pan. The measurements were conducted with 100% N2 purge gas at a flow rate of 50 mL/min. 10 mg of powder was weighed in an aluminium pan and tightly closed with a lid. The samples were equilibrated at -25°C for 5 min, and heated to 130°C at the rate of 10°C/ min, and then they were cooled from 130°C to 25°C.

Thermogravimetric analysis (TGA) was performed using a Mettler Toledo (TGA/DSC 1HT, Switzerland) instrument with similar purge gas and sample amount given for DSC analysis. The samples were heated from 25°C to 800°C with the heating rate of 10°C/ min.

Particle morphology

Microstructure of carrot powders before (0th day) and after storage (90th day) were evaluated with scanning electron microscopy (SEM) (FEI, Quanta 450 FEG, USA) operating at an accelerating voltage of 10kV and at low vacuum conditions.

Statistical analysis

Statistical analyses were conducted using SPSS vers. 16.0 (SPSS Inc., USA) software. The results were given as the mean value ± standard deviation. One-way analysis of variance (ANOVA) test with Duncan's multiple comparison were performed at 95% confidence interval.

RESULTS AND DISCUSSION

Powder properties

Since the properties of food powders change with moisture transfer, the powders are considered as dynamic systems (Peleg 2009). Also, the moisture content of powdered food materials depend on drying temperature, foam thickness and their composition (initial moisture, protein, sugar content etc.), so it affects the flowability and thermal properties of the powders (Quek et al. 2007, Jinapong et al. 2008, Balasubramanian et al. 2016, Salahi et al. 2017, Dehghannya et al. 2018). Powder properties of foam-mat dried 15% EA and 15% EA+ 10% WPI carrot juice powders were given in Table I. Moisture content of the 15% EA+ 10% WPI powder was significantly increased with storage time ($p< 0.05$); however, the moisture of 15% EA powder did not change significantly after the first month ($p> 0.05$). At the very beginning of storage period, the moisture content of the 15% EA+ 10% WPI powder were found comparably lower than 15% EA powder. As it was reported by Yang & Foegeding (2010), the adsorption mechanism of egg albumen proteins and whey proteins are rather diffent at the air water interface which is possibly affecting the moisture transfer rate thus the final moisture of dried foams. The moisture variations in foam-mat dried powders during storage have not been found in the literature; but the initial moisture content of foam-mat dried lime juice powder was between 2.7-2.8% (Dehghannya et al. 2018), foam-mat freeze-dried blueberry juice powder was 3-4% (Darniadi et al. 2018), for foam-mat dried yacon juice powders it was between 3.5-6.6% (Franco et al. 2016), and 1.8-6.9% for black rice bran anthocyanin extracts (Kanha et al. 2022). Moisture of food powders varies between 0.2-12% in general, but 2-10% moisture level is recommended in order to achieve physical and chemical stability of powders (Barbosa-Canovas et al. 2005).

Reconstitution properties of food powders for example wettability, dispersibility, solubility and sinkability are related to the water absorption capacity of powders, hence easily/rapidly wettable powders are preferred (Barbosa-Canovas et al. 2005, Gong et al. 2007, Quek et al. 2007, Salahi et al. 2017, Dehghannya et al. 2018). Wettability of the carrot juice powders of 15% EA+ 10% WPI formulation was found significantly lower than the 15% EA formulation throughout the storage period ($p< 0.05$); however, wettability of both of these powders gradually increased depending on the period of storage ($p< 0.05$). Overall, the foam-mat dried carrot juice powders were comparable to literature; for example, wettability value of spray dried bayberry powder was found to be between 15-120 s, whereas it was between 451-2258 s for foam-mat freeze-dried date powders (Seerangurayar et al. 2018), and above 1000 s for spray dried skim milk powder (Barbosa-Canovas et al. 2005).

%Hygroscopicity values at 90th day was shown in Table I, and 15% EA+ 10% WPI powders had significantly lower hygroscopicity compared to the 15% EA powders during the storage period ($p< 0.05$). On the other hand, their %hygroscopicity had increased significantly throughout the storage. Since the food powders have high affinity to water, they are highly hygroscopic and have tendency for caking (Barbosa-Canovas et al. 2005). Spray-dried tomato powders with various amounts of maltodextrin had similar hygroscopicity values to the present study (Goula & Adamopoulos 2008), although Salahi et al. (2017) reported quite higher values (16.7-20.8%) for foam-mat dried cantaloupe pulp powders.

Caking is a complex phenomenon as it is a result of temperature and moisture that adversely affects the flow properties of powder products (Barbosa-Canovas et al. 2005). Carbohydrates of

the food powders that is initially in amorphous states transformed into crystalline by formation of liquid bridges on the powder surface (Barbosa-Canovas et al. 2005, Schuck et al. 2012). Besides, glass transition temperature and water activity of powders directly affect the caking behavior (Schuck et al. 2012, Darniadi et al. 2018). In literature, degree of caking for spray-dried tomato powder was between 8.9-22% (Goula & Adamopoulos 2008), while for spray-dried orange juice powder it was between 5.9-24.8% (Goula & Adamopoulos 2010). In this study, degree of caking for 15% EA+ 10% WPI powders were comparably lower than the spray dried powders in the aforementioned studies, although the results of 15% EA powder formulation exceeded previously mentioned values. This behavior could be related with the possible encapsulation effect of whey proteins, since the sugars may prevent getting into contact with the absorbed moisture and therefore it increases the resistance to caking (Barbosa-Canovas et al. 2005). Similar to 15% EA powder, Seerangurayar et al. (2018) have stated that the degree of caking for foam-mat freeze-dried date powders were found between 75-91% depending on the maturity stage.

Flowability and cohesiveness of food powders are evaluated by Carr index and Hausner ratios (Jinapong et al. 2008). In Table 1, it can be seen that 15%EA powder flowability was classified in "Good-Very Good" group, while 15% EA+ 10% WPI powders fell within the range of "Fair" to "Good" (Jinapong et al. 2008). Besides, 15% EA powder during whole storage period had "low" cohesivess (easily fluidized), and "intermediate" to "low" cohesiveness was observed for the 15% EA+ 10% WPI powders, respectively (Barbosa-Canovas et al. 2005, Jinapong et al. 2008). The Hausner ratio is known to be sensitive to particle size and shape (Barbosa-Canovas et al. 2005), and coarse particles have

Table I. Powder properties of the samples during storage period.

Storage period (days)	Moisture (%)		Wettability (s)		Hygroscopicity %	
	15% EA	15% EA+ 10% WPI	15% EA	15% EA+ 10% WPI	15% EA	15% EA+ 10% WPI
0	$4.44\pm0.37^{a;B}$	$3.09\pm0.62^{a;A}$	$24.5\pm4.1^{a;B}$	$14.3\pm3.3^{a;A}$	$4.30^{a;B}$	$3.02^{b;A}$
15	$4.47\pm0.75^{a;B}$	$2.98\pm0.31^{a;A}$	$41.8\pm3.5^{b;B}$	$17.8\pm1.7^{a;A}$	$4.39^{b;B}$	$2.93^{a;A}$
30	$5.99\pm0.71^{b;B}$	$3.91\pm0.15^{b;A}$	$45.8\pm2.5^{b;B}$	$27.5\pm3.1^{b;A}$	$5.84^{d;B}$	$3.84^{d;A}$
60	$6.32\pm0.24^{b;B}$	$3.57\pm0.38^{a,b;A}$	$47.8\pm1.7^{b;B}$	$33.3\pm2.9^{c;A}$	$6.13^{e;B}$	$3.48^{c;A}$
90	$5.82\pm0.58^{b;A}$	$5.15\pm0.45^{c;A}$	$145.3\pm9.3^{c;B}$	$32.8\pm2.6^{c;A}$	$5.70^{c;B}$	$5.06^{e;A}$

Storage period (days)	Degree of caking		CI (%)		HR	
	15% EA	15% EA+ 10% WPI	15% EA	15% EA+ 10% WPI	15% EA	15% EA+ 10% WPI
0	$72.58^{a;B}$	$0.36^{a;A}$	$15.15^{c;A}$	$17.05^{a;A}$	$1.18^{c;A}$	$1.21^{a;A}$
15	$88.66^{b,c;B}$	$1.34^{c;A}$	$11.34^{b;A}$	$26.03^{b;B}$	$1.13^{b;A}$	$1.35^{b;B}$
30	$77.57^{a,b;B}$	$1.06^{b,c;A}$	$6.90^{a;A}$	$23.86^{b;B}$	$1.07^{a;A}$	$1.31^{b;B}$
60	$79.43^{a,b;B}$	$1.26^{c;A}$	$13.12^{b,c;A}$	$25.06^{b;B}$	$1.15^{b,c;A}$	$1.33^{b;B}$
90	$97.74^{c;B}$	$0.81^{b;A}$	$6.73^{a;A}$	$25.91^{b;B}$	$1.07^{a;A}$	$1.35^{b;B}$

[a-e] Different letters in the same column are statistically different ($p < 0.05$).
[A-B] Different letters in the same row are statistically different ($p < 0.05$).

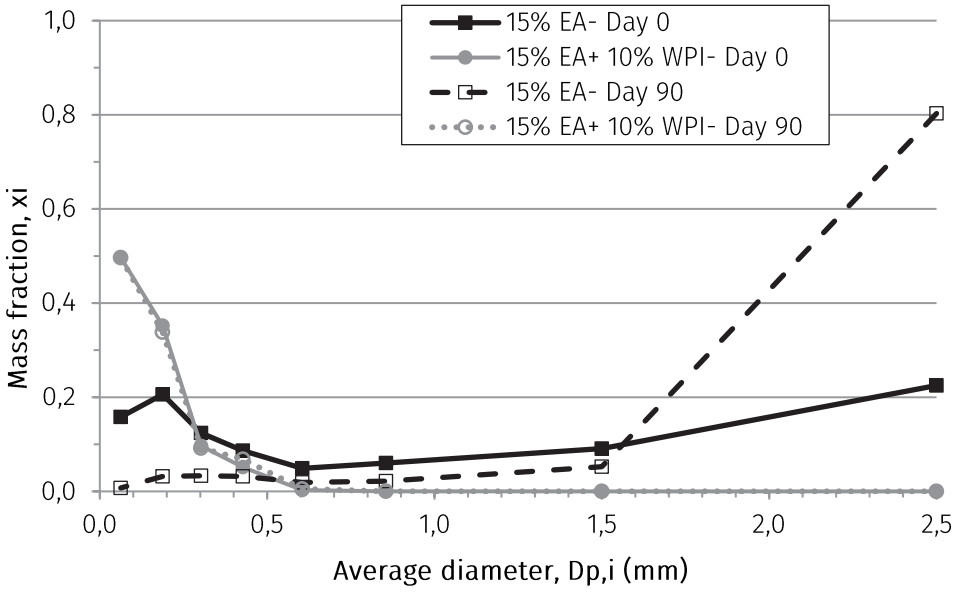

Figure 1. Particle diameter distribution of the 15% EA and 15% EA+10% WPI powders at the initial day (Day 0) and final day (Day 90) of storage period.

better flowability compared to the fine particles (Jinapong et al. 2008). This behavior is confirmed by the results of particle diameter analysis (Fig. 1), since the 15% EA powders have much bigger particles.

The particle diameter distribution of 15% EA and 15% EA+ 10% WPI powders are given in Fig.1. The diameters of 15% EA+ 10% WPI powders at the initial day and final day of storage were almost similar and overlapping, although the diameter distribution of 15% EA powders had significantly changed due to particle agglomeration. Especially the mass fraction of particles bigger than 1.5 mm had increased more than threefold. These results were in agreement with the degree of caking, since the food powders with high sugar content may rapidly absorb water and cause the powder caking by agglomeration of the particles (Jinapong et al. 2008, Cakmak 2020). Besides, high moisture content of powders is associated with the increased ability of particle adherence (de Cól et al. 2021). Although the moisture of carrot powders were statistically in the same group on the 90th day (p> 0.05), the presence of whey proteins in the foam formulation by its encapsulating mechanism probably improved the powder properties and increased the resistance to caking (Barbosa-Canovas et al. 2005).

Color values of powders

Color values were measured at the initial day of storage, and the effects of foam thickness on powder color were reported as well. As can be seen in Table II, foam formulation and the drying temperature had significant effect (p< 0.05) on the measured color values similar to the literature (Franco et al. 2016). 15% EA+ 10% WPI powders had significantly higher L*, but lower a* values compared to 15% EA powders independent of the drying temperature. This could be due to the initial color of the foams with 15% EA+ 10% WPI incorporation had resulted in relatively high lightness due to WPI addition, although color values of the foams with 15% EA were closer to the carrot juice (L*: 41.58±0.16, a*:12.56±0.24, b*: 20.25±0.24, h*: 1.02±0.00 and C*: 23.83±0.32). Besides, WPI incorporation might increase the volume of air entrapped in the foam structure and caused lighter color with low yellowness (b*) intensity compared to 15% EA powder (Cakmak & Ozyurt 2021a). This similarity was

also observable in ΔE values of 15% EA formulations being smaller than 15% EA+ 10% WPI powders (p< 0.05). In the study of de Cól et al. (2021), increasing the foam thickness of bacaba pulp increased the drying time hence resulted in darker color (lower lightness) and increased redness value. But this behavior was not observable for 15% EA powders, whereas there was a clear tendency between lightness reduction with respect to foam thickness for 15% EA+ 10% WPI powder.

The redness of 15% EA powders decreased with respect to increased drying temperatures. This behavior is associated with the carotenoid degradation due to thermal treatment (Sharma et al. 2009, Hardy & Jideani 2017, Yilmaz et al. 2019). However, the period of exposure to drying air possibly predominated the final color of the powders, since the drying period of the carrot juice powders was shortened between 62-75% upon the increment of drying temperature from 50°C to 70°C (Cakmak & Ozyurt 2021b).

C* value represents the paleness of color, and is stated to be negatively effected (decreased) from the carotenoid degradation (Yilmaz et al. 2019). The chroma values of 15% EA powders decreased with respect to increased drying temperatures independent of foam thickness (p< 0.05). In contrast, there was no significant difference in the C* of 15% EA+ 10% WPI powders with drying temperature for 5 mm thickness (p> 0.05). For 6 mm, the chroma value of 15% EA+ 10% WPI dried at 70°C was significantly higher than the powders dried at 50 and 60°C (p< 0.05). Preservation of original foam color particularly by means of lightness and chroma might be linked to the whey proteins which acted as a barrier around carrot juice carotenoids.

Table II. Color values of carrot juice powder dried at different foam thicknesses and temperatures.

Color values	15% EA -5 mm			15% EA+ 10% WPI -5 mm		
	50°C	60°C	70°C	50°C	60°C	70°C
L*	54.57±0.25$^{a;A}$	56.40±0.85$^{b;B}$	55.89±0.43$^{b;B}$	63.49±0.28$^{a;C}$	67.42±0.43$^{b;D}$	69.35±1.19$^{c;E}$
a*	29.94±0.24$^{b;D}$	29.78±0.52$^{b;D}$	27.60±0.19$^{a;C}$	25.67±0.48$^{b;B}$	27.33±0.40$^{c;C}$	24.06±0.88$^{a;A}$
b*	24.85±0.43$^{b;B,C}$	24.83±0.79$^{b;B,C}$	21.81±0.19$^{a;A}$	24.25±0.40^{a,B}	26.90±0.19$^{c;D}$	25.55±1.08^{b,C}
h*	0.69±0.01$^{b;B}$	0.69±0.01$^{b;B}$	0.67±0.00$^{a;A}$	0.76±0.01$^{a;C}$	0.78±0.01$^{b;D}$	0.82±0.01$^{c;E}$
C*	38.91±0.43$^{b;B}$	38.78±0.89$^{b;B}$	35.17±0.26$^{a;A}$	35.31±0.53$^{a;A}$	38.35±0.39$^{b;B}$	35.10±1.36$^{a;A}$
ΔE	22.18±0.34$^{b;B}$	23.19±0.99$^{c;C}$	20.82±0.35$^{a;A}$	25.84±0.19$^{a;D}$	30.49±0.16$^{b;E}$	30.57±0.63$^{b;E}$

Color values	15% EA -6 mm			15% EA+ 10% WPI -6 mm		
	50°C	60°C	70°C	50°C	60°C	70°C
L*	54.14±0.42$^{a;A}$	53.63±0.44$^{a;A}$	56.82±0.93$^{b;B}$	62.24±1.03$^{b;D}$	60.05±0.85$^{a;C}$	65.63±1.63$^{c;E}$
a*	30.27±0.36$^{c;F}$	26.99±0.50$^{a;D}$	28.41±0.35$^{b;E}$	22.15±0.38$^{b;B}$	20.65±0.80$^{a;A}$	23.94±0.27$^{c;C}$
b*	24.51±0.40$^{c;E}$	21.71±0.23$^{a;C}$	23.30±0.60$^{b;D}$	20.60±0.43$^{b;B}$	18.68±1.25$^{a;A}$	22.93±0.52$^{c;D}$
h*	0.68±0.00$^{a,b;A}$	0.68±0.01$^{a;A}$	0.69±0.01$^{b;A}$	0.75±0.01$^{b;C}$	0.73±0.02$^{a;B}$	0.76±0.01$^{b;D}$
C*	38.94±0.52$^{c;F}$	34.64±0.51$^{a;D}$	36.75±0.63$^{b;E}$	30.25±0.46$^{b;B}$	27.85±1.39$^{a;A}$	33.15±0.50$^{c;C}$
ΔE	22.12±0.58$^{b;C}$	18.86±0.54$^{a;A}$	22.21±0.94$^{b;C}$	22.79±0.93$^{b;C}$	20.27±0.98$^{a;B}$	26.75±1.48$^{c;D}$

$^{a-c}$ Different letters in the same column (for same sample) are statistically different (p < 0.05).
$^{A-E}$ Different letters in the same row are statistically different (p < 0.05).

Antioxidant activity, total phenolics and carotenoid contents of powders

Measurement of antioxidant activity using DPPH reactive is known as an easy and valid method to evaluate the radical scavenging ability of antioxidants. The change in inhibition values of carrot powder samples during storage was shown in Table III. Binding potential of antioxidants to free radicals gives information about radical scavenging activity of the sample and is expressed as inhibition percent. As a result, higher scavenging activity means higher antioxidant capacity present in the sample (Singh et al. 2011). No significant difference was observed in the inhibition values of the samples on the first day of storage ($p > 0.05$). It was determined that the inhibition values during storage decreased significantly over time, and the lowest inhibition value was found in the sample of 15% EA powder on the final day of storage ($p < 0.05$). The stability of bioactive compounds of carrot powders during storage is an important criterion especially to be used as functional ingredients in several food compositions (Irigoiti et al. 2021). Therefore, 15% EA+ 10% WPI powder even after 90 day storage with respect to the higher inhibition capacity had more potential for food enrichment purposes compared to 15% EA powder.

The total phenolic contents of carrot foam powders during the storage period were presented in Table III. In general, it was determined that the 15% EA+ 10% WPI sample had a higher total phenolic content compared to the 15% EA sample. A significant difference was found between the total phenolic contents in both carrot powders during the storage period ($p < 0.05$). A similar trend was observed in DPPH radical scavenging activity. Both results can be explained by the fact that phenolic acids and antioxidants act as scavengers of free radicals produced in oxidation reactions (Kim et al. 2006).

The number and position of substituted hydroxyl, methoxy, and methyl group in the phenolic compounds can influence the affinity between phenolic compounds and proteins (de Morais et al. 2020) and so, phenolic compounds may interact with protein leading to the formation of soluble and insoluble complexes. As a result of this interaction, conformational changes in protein occur (Yang et al. 2021). Moreover, this complexation may suppress the reducing and antioxidant capacity of phenolic compounds related to H-bonding formation (de Morais et al. 2020). Further studies should be carried out for the detailed explanation of the reason of these changes.

The total carotenoid contents of carrot powder samples during storage were presented in Table III. On the first day of storage, the total carotenoid content of the 15% EA sample was higher than that

Table III. Antioxidant capacity, total phenolics and total carotenoids of carrot juice powders during storage.

Storage period (days)	Inhibition %		Total phenolics (mg GAE/g DM)		Total carotenoids (µg β-carotene/g DM)	
	15% EA	15% EA+ 10% WPI	15% EA	15% EA+ 10% WPI	15% EA	15% EA+ 10% WPI
0	$62.84\pm2.28^{a,A}$	$59.45\pm1.20^{a,A}$	$41.70\pm0.18^{a,A}$	$41.93\pm0.05^{a,B}$	$73.63\pm2.67^{a,A}$	$70.39\pm0.83^{a,B}$
15	$61.31\pm1.37^{a,A}$	$59.45\pm1.05^{a,B}$	$36.50\pm0.22^{b,A}$	$38.47\pm0.09^{b,B}$	$67.22\pm2.90^{b,A}$	$68.68\pm0.10^{b,A}$
30	$44.89\pm2.14^{b,A}$	$58.89\pm0.67^{a,B}$	$29.46\pm0.04^{c,A}$	$29.84\pm0.46^{c,A}$	$51.05\pm0.15^{c,A}$	$60.06\pm0.62^{c,B}$
60	$15.55\pm2.81^{c,A}$	$44.61\pm1.66^{b,B}$	$18.14\pm0.48^{d,A}$	$23.31\pm0.13^{d,B}$	$44.84\pm0.06^{d,A}$	$54.70\pm0.98^{d,B}$
90	$11.97\pm1.51^{d,A}$	$33.15\pm0.47^{c,B}$	$16.27\pm0.24^{e,A}$	$22.47\pm0.11^{e,B}$	$36.49\pm0.05^{e,A}$	$48.56\pm1.06^{e,B}$

[a-e] Different letters in the same column are statistically different ($p < 0.05$).
[A-B] Different letters in the same row are statistically different ($p < 0.05$).

of the 15% EA+ 10% WPI sample. At the end of the storage period, the content at 15% EA+ 10% WPI sample was found to be higher than the 15% EA sample. It appears that the use of different foaming agents has a significant effect on the carotenoid content. Chaux-Gutiérrez et al. (2017) also stated in their study that the foaming agents were able to improve the carotenoid retention. A decrease in carotenoid content was observed with increasing storage time. As it is stated in the literature, total carotenoid losses are based on the epoxy forming structure and isomerization of carotenoids and this reduction may also be related with autoxidation (Wilson et al. 2014).

Thermal properties of powders

The thermal properties of powders were measured by DSC (Fig. 2) and TGA (Fig. 3) at the initial and final days of storage period. There was clear difference in the thermal properties of each powder type due to moisture absorption during storage period. Although the moisture of 15% EA and 15% EA+ 10% WPI powders were similar on Day 90, DSC thermograms of 15% EA powders had significantly changed during 90 days. The glass transition temperature (Tg) of 15% EA powder was 30.6°C on Day 0, and decreased to 24.1°C at Day 90, while Tg of 15% EA+ 10% WPI was 52.3°C at Day 0, and decreased to 31.2°C on Day 90. The glass transition of carrot powder dried with different wall materials was found between 34.9-45.9°C, whereas the lowest Tg was observed for the sample dried with whey protein isolate similar to this present study (Janiszewska-Turak et al. 2017). In the study of Katekhong & Charoenrein (2016), the Tg of freeze-dried egg albumen was found between 50.4-57.9°C, and the increment in moisture content resulted in lower glass transition temperatures. Similarly, the glass transition of spray dried or hydrolyzed egg albumen was found at 86-107°C and 11-104°C between 0.05-0.64 water activity values, respectively (Rao & Labuza 2012). This behavior pertains to the plasticizing effect of water and moisture absorption of dried material during storage period (Katekhong & Charoenrein 2016, Janiszewska-Turak et al. 2017).

TG curves of carrot powders (Fig. 3) were almost overlapping for 15% EA+ 10% WPI samples, while the thermograms of 15% EA were significantly different on the initial and final storage period. In literature, the weight loss around 100-200°C is associated with the removal of free and bound water (Lekshmi et al. 2019, Misra & Yadav 2020). So the mass loss from 8% to 10% between 25-200°C of carrot juice powders was related to evaporation of water. Besides, secondary and the most significant mass loss (~30%) in 15% EA powders were observed between 200-250°C. At the same temperature range, the mass loss was around 10% for 15% EA+ 10% WPI powders, since the peak area shown in derivative of TG curves was comparably smaller for these samples. The higher mass loss (around 30%) in 15% EA+ 10% WPI powders was detected between 240-350°C. Moreover, thermal stability of encapsulated squalene was improved and the mass loss related with the degradation was delayed to higher temperatures compared with the samples that were not encapsulated with whey proteins (Lekshmi et al. 2019). Therefore, WPI incorporation ameliorated the thermal stability of carrot juice powders by detaining the secondary decomposition into further temperature intervals similar to the literature. The mass loss until 400°C is stated as dehydration, degradation and depolymerization of the material (Lekshmi et al. 2019, Misra & Yadav 2020).

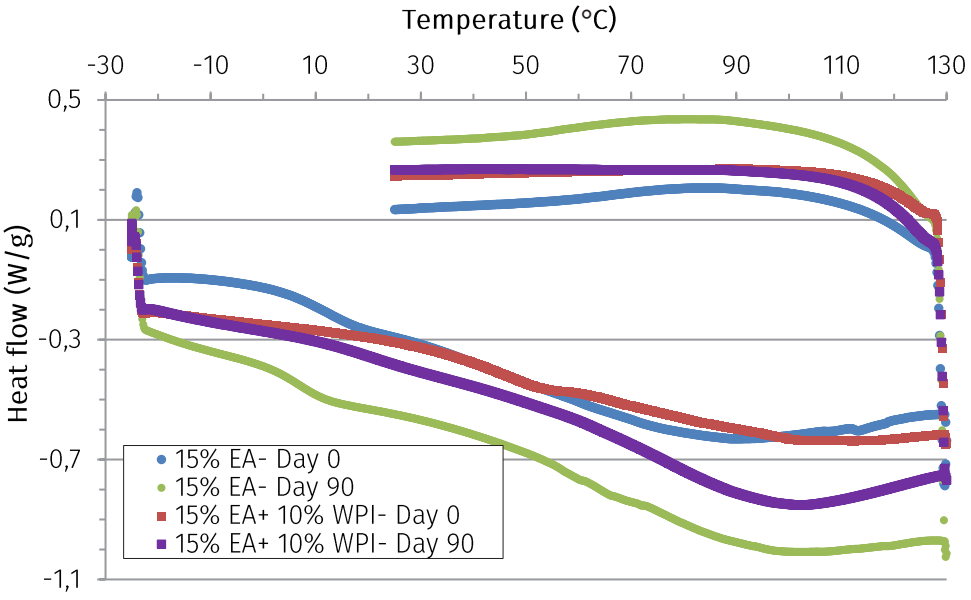

Figure 2. DSC profiles of the foam-mat dried carrot juice powders.

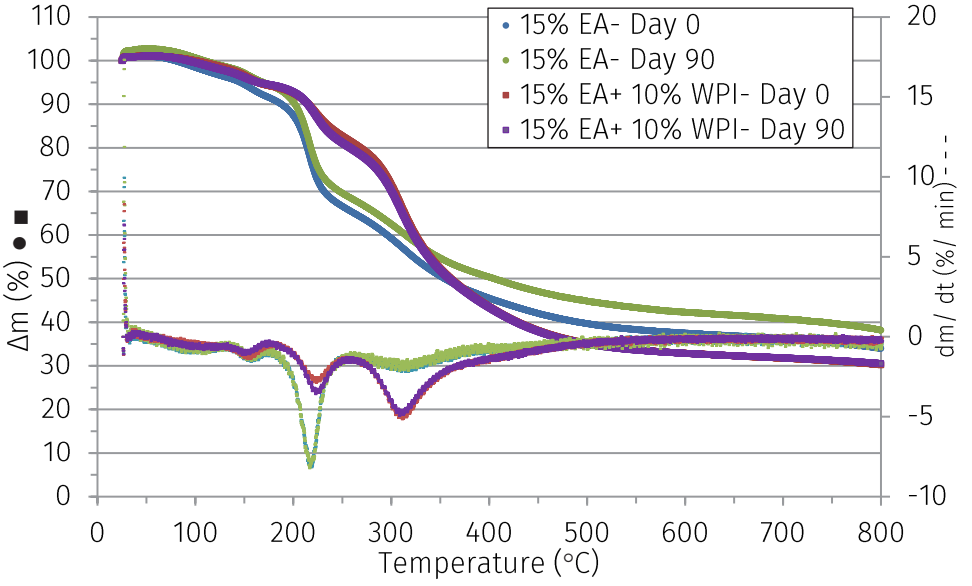

Figure 3. TG and DTG curves of foam-mat dried carrot juice powders.

Particle morphology of powders

Microstructure variations with respect to storage of carrot powders are shown in Fig. 4. The micrographs of both powders at the initial day of storage showed no signs of agglomeration; however, the particles had porous and irregular shape and had different sizes. Similar particle morphology was observed for the foam-mat dried date, yacon juice and black rice bran anthocyanin powders (Franco et al. 2016, Seerangurayar et al. 2017, Kanha et al. 2022). On the other hand, 15% EA powder on the final day of storage (Fig. 4c) had agglomerated and compact structure due to significant increment in the moisture and degree of caking values. This result was consistent with the particle diameter

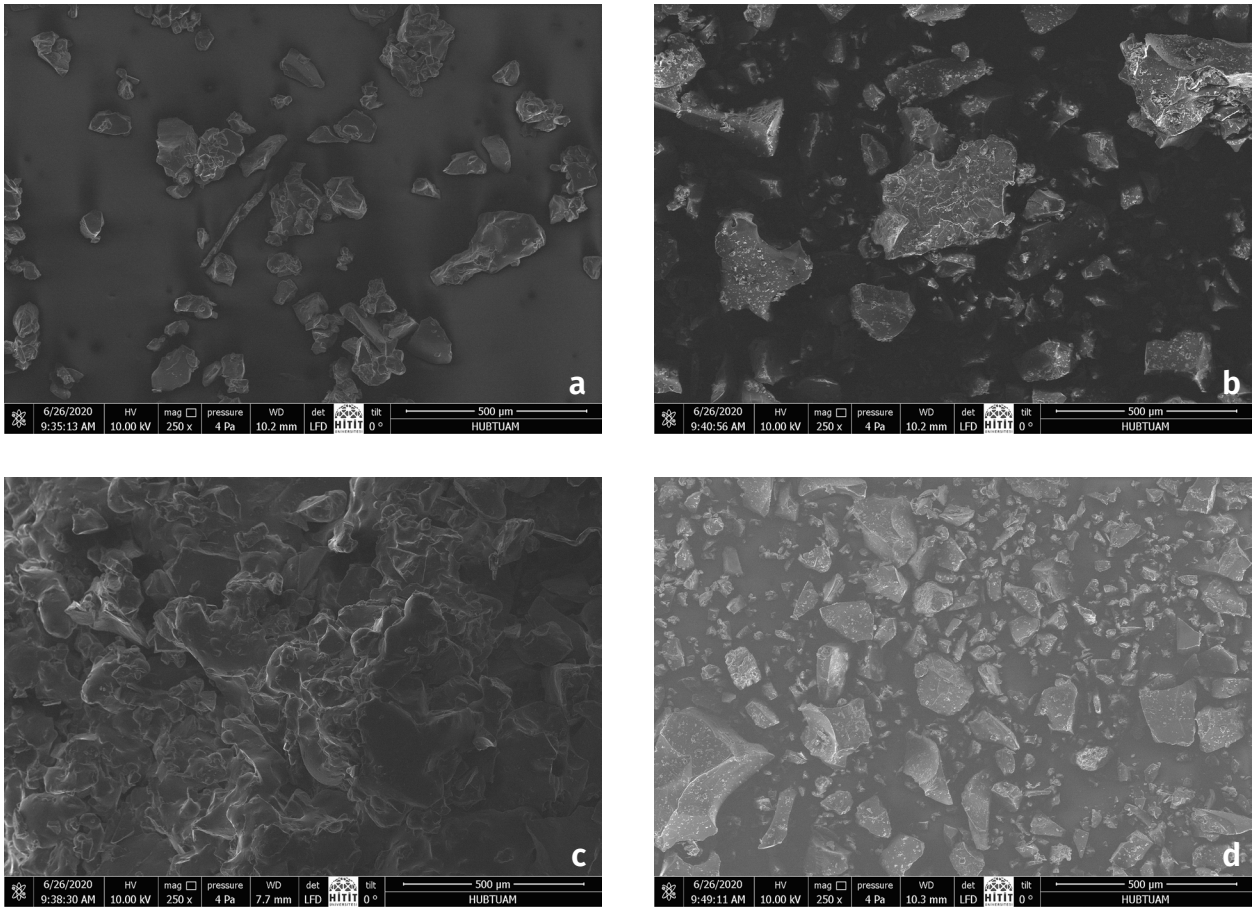

Figure 4. SEM micrographs of foam-mat dried carrot juice powders; a: 15% EA- Day 0, b: 15% EA+ 10% WPI- Day 0, c: 15% EA- Day 90, d: 15% EA+ 10% WPI- Day 90 (at 250× magnification).

distribution of the 15% EA powders (Fig. 1), since the particle size was significantly increased during storage period. In contrast, 15% EA+ 10% WPI powder on 90th day were analogous to the sample at the initial day and the porous structure without formation of aggregates was maintained. The amount of carrier or foaming agents may improve the powder flow properties and thus helps to maintain the porous structure (Franco et al. 2016, Seerangurayar et al. 2017).

CONCLUSIONS

Foam-mat drying method allows the preservation of functional, flow and thermal properties of the powders because of being a low drying temperature application, and improving moisture transfer by increasing the surface area of the food material by foaming. In this study, EA and EA-WPI mixtures were used as foaming agents for producing foam-mat air-dried carrot juice powders. The powder properties were significantly affected from the foaming formulations and the length of storage, though the flow properties of 15% EA+10% WPI powders were found better than 15% EA powders. Presence of WPI in foam formulation protected the carrot powders from physical, chemical and

thermal degradation. The color values of powders were changed with respect to drying temperatures, since the exposure to drying environment at 70°C was significantly shorter than at 50°C. According to the obtained results, the foam-mat dried carrot powder with EA+WPI incorporation has a potential to be used in various food formulations including bakeries, beverages or pasta as a functional ingredient for enrichment of nutritional composition of the added foods. This drying method allowed encapsulation of carrot bioactives at low drying temperatures, so the production of carrot powders are easily adaptable by small or industrial scale production with lower cost of equipment compared to freeze or spray dryers. Also, longer shelf life compared to fresh carrot increases the prospective uses by improved powder properties like its flowability. Moreover, further studies may focus on foam-mat drying of carrot juice by employing plant-based protein sources such as legume proteins for producing fully-plant based carrot juice powder.

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Hulya Cakmak conceived the idea; Hulya Cakmak and Vasfiye Hazal Ozyurt contributed to the experimental study, validation of results, wrote the manuscript draft and read and approved the final manuscript.