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Application of Different Compositions of Apple Puree Gels and Drying Methods to Fabricate Snacks of Modified Structure, Storage Stability and Hygroscopicity

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Abstract: The aim of this study was to determine the effect of incorporation of apple puree and maltodextrin to agar sol on the sorption properties and structure of the dried gel. The effect of different drying methods on the sorption behaviour of aerated apple puree gels was also observed. The gels with the addition of 25% and 40% concentration of apple puree and with or without maltodextrin were prepared and dried. The foamed agar gel was subjected to freeze-drying, air-drying and vacuum-drying. The sorption properties of dried gels (adsorption isotherms, water uptake in time) were investigated. The relations between the glass transition temperature, water activity and water content were also obtained for some apple snacks. The increase in apple puree in freeze-dried gels increased the hygroscopicity and decreased the glass transition temperature (T_g). The water content at given activity and hygroscopicity were reduced by the addition of maltodextrin, which also caused the increase in T_g. The application of different drying methods enabled obtaining different structures of material. The open-pore, fragile materials were obtained by freeze-drying, the expanded matrix with big holes was characteristic for vacuum-dried gels, but the closed pores with thick walls were created during the air-drying.

Keywords: fruit gel; aeration; drying; sorption isotherms; glass transition; maltodextrin

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1. Introduction

Nowadays, the fast pace of life promotes the growth of snack consumption, which has become an important part of the daily diet [1]. Many consumers expect tasty and healthy food products based on fresh or processed fruits and vegetables as an alternative to the energy-dense, nutrient-poor snacks [2]. Additionally, these types of products should have an attractive appearance and texture. The application of a wide range of drying methods for fruits materials enables creating snacks with different structure and texture [3–7].

Drying is one of the oldest and common unit operations used to reduce the water moisture content to the level that guarantees the stability of food products during long-term storage. The application of this technology leads to slowing down enzymatic and non-enzymatic reactions as well as preventing microbial contamination of food. The quality of dehydrated products varies depending on the methods and conditions of drying [8,9]. A simple and relatively inexpensive dehydration technology, widely used in the food industry, is air-drying. During convective drying, the material is subjected to a continuous flow of hot air, which transports the evaporated water. The dehydration by the air-drying may cause undesirable changes in the quality of the material such as shrinkage, loss of volatile compounds, colour and nutrients [10,11].

Drying at low temperature and pressure (e.g., during freeze-drying or vacuum-drying) requires longer times of dehydration in comparison to other drying methods, but the obtained products are characterised by the high quality [8]. Porosity, colour, rehydration and retention of many nutrients (anthocyanins, phenolics, vitamin C) and flavours of fruit products obtained with the application of freeze-drying were more favourable than observed if other drying methods were used [12]. In the case of freeze-drying, the colour of apples, pears and oranges was preserved [13]. Freeze-drying contributed minimal volume reduction of different fruits (from 5 to 15%), while convective-drying caused excessive changes in volume (around 80%) [11]. The application of freeze-drying enabled 63% vitamin C retention in guava in comparison to only 25% when the other drying methods were used [14]. Freeze-drying of sea buckthorn pulp caused loss of vitamins E, C and phenolics reaching about 35%, 20% and 4%, respectively. However, it was possible, using freeze-drying, to retain more nutrients than by drying with a heat pump [15]. The preservation of lycopene, carotenoids, unsaturated fatty acids in oils and lipid-based oxidisable compounds in freeze-dried fruits can be limited due to the acceleration of oxidative reactions at low water activities [16]. The main disadvantage of freeze-drying is a high cost of the process [13,16]. The chemical, mechanical and thermal pre-treatments of fruits (immersion in the alkaline, peeling, blanching) were applied to enhance water transport during the freeze-drying and reduce the duration of the process as well as its costs. However, the use of some pre-treatment methods induced the loss of nutrients and vitamins. The application of infrared radiation, microwave energy and ultrasounds power in the freeze-drying of plant based foods was developed for intensification of the dehydration process. The limited examples of snack products with the addition of fruit materials (pulp and puree) were mainly fabricated with the application of the freeze-drying method or with a microwave-assisted freeze-drying technique [3,5,7]. Additionally, these products were obtained with the addition of foaming and stabilising agents (pectin, maltodextrin, potato protein) [17] as well as with carriers (maltodextrin, gum Arabic, bamboo fibre, whey protein isolate) [6,7,17]. Martínez-Navarrete et al. [6] stated that the freeze-drying method can provide different food textures such as crunchy fruit snacks with good acceptance of consumers. However, the drawback of the freeze-drying process can be a collapse of structure, which was observed for sugar-rich foods due to the glass transition of the amorphous matrix [18,19].

Fruit-based products on the market such as juices and purees are rich in low molecular weight soluble solids (sugars and organic acids). The rapid removal of water during drying of the material containing these kinds of compounds results in the formation of the amorphous state of the solid matrix. The amorphous solids show changes in structure, physical and chemical properties during the time that affect the stability of the material and, consequently, reduce the shelf life of dried products. The amorphous matrix may transform its structure from a stable solid glass to liquid-like rubber. This phenomenon occurs with an increase in water content in the amorphous food system at the temperature defined as the glass transition temperature T_g [20–23]. The significant physical defects, changes of physicochemical properties of dehydrated foods, and the worsening of their quality such as structure collapse, caking, stickiness, and change of colour can be observed at storage temperatures above T_g. To overcome this problem, high molecular weight biopolymers (maltodextrin, gums) with high T_g can be incorporated into fruit materials before drying [24,25].

As T_g depends on water content, knowledge of the relation between water activity and water content of the product described by the moisture sorption isotherms is necessary to predict the quality and stability of products during storage [22,26–28]. Many mathematical models can be used to describe the water sorption isotherms: these include models that refer to multilayer sorption (GAB model, Lewicki model), kinetics model with monolayer sorption (BET model) and semi-empirical models (Peleg model, Hasley model) [29].

There are many studies concerning the sorption behaviour and glass transition of dried fruit products [20,25,26,30,31]. However, the current literature about the effects of

fruit material addition on these properties in the case of the dried gel is still limited. Drying of gels enables obtaining novel structures and textures of products [3,32–34]. Novel gelled products can be created with different hydrocolloid agents and fruit materials. Among many hydrocolloids used in the production of gels, agar-agar is an effective gelling agent, which forms a thermo-reversible but stable gel over a wide range of temperatures [35]. Agar is a mixture of agarose and agaropectin fractions, but gelation is caused by the presence of agarose, which creates the physical gel. The structure of this gel is only formed by the polymer's molecules, which are linked by hydrogen bonds [36]. The formation of agar gels occurs after the cooling of sol solutions below 40 °C.

This study aimed to determine the impact of adding apple puree and maltodextrin to agar sol on the sorption properties and structure of the freeze-dried gel. The scope of research also involved the analysis of the effect of different drying methods on the investigated properties of aerated apple puree gels. The relations between the glass transition temperature, water activity, and water content were also investigated for selected snacks.

2. Materials and Methods

2.1. Materials and Preparation of Gels

Apple snacks were produced by the freeze-drying of 2% agar gel with the addition of apple puree (25%, 40%) and with or without maltodextrin. Additionally, one type of gel was obtained by foaming of agar sol solution with apple puree, maltodextrin and methylcellulose (foaming agent). The composition of gels (concentration of ingredients) and type of added gelling agent and carrier were selected based on preliminary studies. This variant of the gel was subjected to different drying methods: air-drying, vacuum-drying, and freeze-drying. The pure agar-agar gel was also freeze-dried. Table 1 presents the composition of the investigated gels. The ingredients used in the formulation of the fruit gel were: Idared apple puree (16°Brix, thermally preserved, provided by a local manufacturer), agar-agar powder (Hortimex Sp. z o.o., Konin, Poland), methylcellulose (Methocel 65HG, Sigma Aldrich Co., Louis, MO, USA), maltodextrin DE 7-13 (Pepees S.A., Łomża, Poland).

Table 1. Composition of gels.

Type of Gel	Apple Puree	Agar-Agar Powder	Maltodextrin	Methylcellulose	Water
		g/100 g			
P0%	0.0	2.0	0.0	0.0	98.0
P25%	25.0	2.0	0.0	0.0	73.0
P25%DE	25.0	2.0	2.0	0.0	71.0
P40%DE	40.0	2.0	3.2	0.0	54.8
P40%DE-MC	40.0	2.0	3.2	0.3	54.5

Before further procedures, puree was rubbed through the sieve with a mesh size of 0.6 mm. The agar-agar powder with or without maltodextrin was dispersed in distilled water, heated to 90 °C and continuously agitated at a speed of 60 rpm using a propeller stirrer R1342 (IKA® Labortechnik, Staufen im Breisgau, Germany). The obtained solution was cooled in the water bath until 60 °C and the apple puree was added and stirred at a speed of 60 rpm for 1 min. In the case of the aerated variant of the gel one more preparation step was used. The mixture of agar-agar sol, maltodextrin, and apple puree with the addition of methylcellulose was foamed using a kitchen mixer (Severin, Sundern, Germany) at a speed of 3500 rpm for 5 min. All variants of the gel product were poured into Petri dishes and allowed to set at temperature 4 °C for 12 h. After storage, the gels were cut by using a cork borer, and cylinders with a diameter of 13.5 mm and height of 13.7 mm were obtained.

2.2. Drying of Gels

2.2.1. Freeze-Drying

The gel samples (all variants) were placed on an aluminium tray and frozen at −40 °C for 4 h using a shock freezer (Irinox, Corbanese, Italy). In the next step, the samples were freeze-dried for 24 h with an application of Gamma 1-16LSC freeze-dryer (Martin Christ Gefriertrocknungsanlagen GmbH, Osterode am Harz, Germany) under the pressure of 63 Pa and at a shelf temperature of 20 °C.

2.2.2. Vacuum-Drying

The gel cylinders (only variant P40%DE-MC) were also dehydrated on a tray in a cabin-vacuum-drier (Conbest, Cracow, Poland) at the temperature of 50 °C and under the pressure of 10 kPa with the tray load of 3.0 kg·m^{-2}. The dryer was equipped with a weighing system (Mensor, Warsaw, Poland), which enabled the control of the mass of the dried material. The material was dried to obtain the constant mass of the product.

2.2.3. Air-Drying

The gel samples (only variant P40%DE-MC) were air-dried with the application of a prototype laboratory convection dryer. The airstreams at a temperature of 70 °C flowed parallel to the material layer with a speed of 1.5 m/s. The tray load was the same as for the vacuum-drying. The gels were dried to equilibrium moisture content.

2.3. Sorption Isotherms

The obtained dried gels were stored in desiccators containing calcium chloride for 1 month to equilibrate the water content in the material. The different saturated salt solutions (LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, $NaNO_2$, NaCl, $(NH_4)_2SO_4$) and anhydrous calcium chloride ($CaCl_2$) were prepared to obtain the water activity in the range of 0–0.810. To avoid the microbial growth in dried samples, a small amount of thymol was placed in desiccators. The triplicate samples of the same variant of dried gels with known weight were placed in open weighing dishes in glass sorption jars with different water activity and stored for 3 months at a temperature of 25 °C. The mass of the samples was controlled every month to obtain the equilibrium value. The water content of the samples after storage was calculated based on the change in the measured initial and final (at equilibrium state) weight of the dried gels.

Table 2 presents the applied models for sorption isotherms used to predict the water sorption characteristics of dried gels. The regression analysis was performed with the application of the Table Curve 2D programme (Systat Software Inc., San Jose, CA, USA). The determination coefficient (R^2), the root mean square error (*RMS*), and parameter *P* were used to estimate the compliance of the model with empirical data (Table 2).

Table 2. List of sorption isotherm models.

Model	Equation		Description
GAB [37]	$u = \frac{u_m C k a_w}{[(1-ka_w)[1-ka_w+Cka_w]}$	(1)	u—equilibrium water content g/g $_{d.m.}$, u_m—monolayer moisture content (g/g $_{d.m.}$); C, k—constants, a_w—water activity
BET [38]	$u = \frac{u_m c a_w}{(1-a_w)[1+(c-1)a_w]}$	(2)	c—constant

Table 2. *Cont.*

Model	Equation		Description
Lewicki [39]	$u = \frac{F}{(1-a_w)^G} - \frac{F}{1+a_w{}^H}$	(3)	F, G, H—constants
Peleg [40]	$u = Aa_w{}^D + Ba_w{}^E$	(4)	A, B, D, E—constants
Hasley [41]	$a_w = exp\left(\frac{-g}{u^n}\right)$	(5)	g, n—constants
Statistical parameters			
'RMS	$RMS = \sqrt{\frac{\sum_{i=1}^{N}\left(\frac{u_e-u_p}{u_e}\right)^2}{N}}100\%$	(6)	u_e—an experimental value of water content, u_p—the predicted value of water content, N—number of observations
P	$P = \frac{\sum_{i=1}^{N}\left\|\frac{u_e-u_p}{u_e}\right\|}{N}100\%$	(7)	

2.4. Water Sorption Kinetics

The water sorption kinetics of all kinds of dried samples were determined according to the procedure described by Jakubczyk, et al. [42]. The sample was weighed automatically (PW-Win software, Radwag, Warsaw, Poland) every minute up to 48 h during storage in a chamber with a relative humidity of 75.3% (saturated NaCl solution). The water uptake (g/gd.m.) by samples was calculated and presented during the time of the sorption experiment as the sorption kinetics curves.

2.5. Glass Transition Temperature

Modulated DSC experiments were carried out using TA Instruments Q200 differential scanning calorimeter (New Castle, DE, USA). Modulated differential scanning calorimetry (MDSC) was used to determine the glass transition temperature of the selected samples (freeze-dried gels: P0%, P25%, P25%DE and air-dried as well as vacuum-dried gels: P40%De-MC) in the range of water activity 0–0.75. The cell was purged with dry nitrogen (at 50 mL/min) and temperature calibration was performed on an empty oven with an application of standard pure indium and distilled water. The sapphire standard was tested for calibration of specific heat capacity. An empty hermetically sealed aluminium pan was used as a reference. The samples of mass 10–15 mg were hermetically sealed in aluminium pans and cooled from room temperature to −90 °C at 5 °C/min, then equilibrated for 5 min. During MDSC procedures, the samples were scanned from −90 to 150 °C at parameters: a constant heating rate of 2 °C/min, an amplitude of ±1 °C, period of modulation −60 s. The obtained data were analysed with respect to the total, reversible and non-reversible heat flow [28,42,43]. TA Instruments Universal Analysis software was applied to determine the onset, midpoint and endpoint of transition. The midpoint of the glass transition was

selected as the transition temperature. The mean value and standard deviation of the glass transition were calculated based on three replicates of measurements.

The Gordon-Taylor model [44] and Roos model [45] represented by Equations (8) and (9) were used to describe changes of T_g with water content and water activity. The goodness fit was estimated based on R^2, *RMS* and parameter *P*.

$$T_g = \frac{(1 - x_w)T_{gs} + kx_w T_{gw}}{(1 - x_w) + kx_w} \quad (8)$$

where: T_{gs}, T_{gw}, T_g—glass transition temperatures of solids, water, and their mixture, respectively; x_w—mass fraction of water; k—the Gordon–Taylor model parameter.

$$T_g = Aa_w + B \quad (9)$$

where: a_w—water activity; A, B—parameters of the Roos model.

The critical water content x_{wc} and critical water activity a_{wc} were defined as the values of these parameters at which the glass transition temperature was equal to the storage temperature of 25 °C.

A one-way ANOVA test (Tukey's method) was applied to determine the significance of differences among samples at the 95% significance level for constants.

2.6. Microstructure

The inner microstructure of the gel samples after drying (a_w~0.1–0.2) was observed using an environmental scanning electron microscope Quanta 200 ESM (FEI Company, Hillsboro, OR, USA) at an accelerating voltage of 30 kV in a high-vacuum mode. The cylindrical samples were cut lengthwise using a new razor blade each time. The images were taken at magnification in the range from 30 to 220×.

3. Results and Discussion

3.1. Sorption Properties of Dried Apple Puree Gels

The sorption isotherms characterise the relation between water content and the water activity of the product. The selection of the best model describing the sorption isotherms can be a challenge due to the different compositions and complex structure of food materials [46].

Table 3 presents the parameters of analysed models of the sorption isotherms of different dried apple puree snacks. The goodness of fit for models was estimated based on the determination coefficient (R^2), the root mean square error (*RMS*), and *P* parameter. The lower values of *RMS* and *P* values and the higher R^2, the better goodness of fit [26]. The GAB and Peleg models were described by high values of R^2, which ranged from 0.993 to 0.998 for all dried samples (Table 3). The *RMS* values varied from 5.99 to 27.59% for GAB model and from 5.79 to 17.64% for Peleg model (Table 3), which can be considered as satisfying. These models were also characterised by *P* values lower than 12.24%. Lee and Lee [47] as well as Filho et al. [48] stated that the sorption model can be acceptable when the parameter *P* is lower than 10%. It can be noticed that parameter *P* of the investigated models was very close to this limit. On the other hand, the Hasley model provided the worst representation of data, with *P* values ranging from 10.21 to 39.55%.

Table 3. Parameters of sorption isotherms models with goodness fit coefficients (R^2, *RMS*, *P*) of freeze-dried (FD), air-dried (AD) and vacuum-dried (VD) gels.

Model	Parameter	FD-P0%	FD-P25%	FD-P25%De	FD-P40%De	FD-P40%DE-MC	AD-P40%DE-MC	VD-P40%DE-MC
GAB	u_m	0.096 ± 0.009	0.074 ± 0.006	0.065 ± 0.005	0.066 ± 0.005	0.079 ± 0.008	0.112 ± 0.021	0.106 ± 0.035
	C	5.351 ± 0.941	2.904 ± 0.625	2.422 ± 0.308	2.493 ± 0.266	2.306 ± 0.412	0.689 ± 0.098	1.224 ± 0.197
	k	0.747 ± 0.022	0.999 ± 0.015	0.989 ± 0.011	1.020 ± 0.008	0.995 ± 0.019	0.943 ± 0.029	0.925 ± 0.018
	R^2	0.993	0.995	0.996	0.995	0.995	0.997	0.996
	RMS	5.99	15.57	14.48	13.02	10.06	27.59	11.26
	P	4.48	10.34	9.97	9.46	6.72	12.24	7.35
BET	u_m	0.068 ± 0.004	0.084 ± 0.009	0.080 ± 0.006	0.073 ± 0.009	0.105 ± 0.018	0.135 ± 0.022	0.072 ± 0.005
	c	7.138 ± 0.780	2.108 ± 0.613	1.514 ± 0.390	2.107 ± 0.711	1.202 ± 0.357	0.462 ± 0.105	2.997 ± 0.464
	R^2	0.986	0.961	0.973	0.948	0.974	0.993	0.978
	RMS	6.29	15.48	13.43	24.23	15.29	25.64	26.06
	P	4.92	12.29	10.33	21.33	12.18	14.20	19.59
Lewicki	*F*	1.598 ± 0.234	1.732 ± 0.828	1.302 ± 0.538	1.034 ± 0.286	1.535 ± 0.602	0.744 ± 0.094	0.350 ± 0.261
	G	0.089 ± 1.541	0.092 ± 0.041	0.101 ± 0.039	0.130 ± 0.032	0.102 ± 0.102	0.135 ± 0.080	0.341 ± 0.132
	H	282.20 ± 5.03	14.596 ± 2.171	14.087 ± 1.859	10.516 ± 1.386	13.458 ± 1.812	6.905 ± 0.483	6.632 ± 1.171
	R^2	0.852	0.995	0.996	0.995	0.995	0.994	0.992
	RMS	28.61	12.95	13.82	12.89	11.45	43.52	17.31
	P	22.88	8.59	9.17	9.14	7.91	18.16	10.97
Peleg	*A*	0.160 ± 0.011	0.995 ± 0.124	1.122 ± 0.111	0.845 ± 0.029	1.672 ± 0.031	0.741 ± 0.071	0.574 ± 0.021
	B	2.774 ± 0.086	8.642 ± 0.975	10.897 ± 1.412	6.813 ± 0.980	11.999 ± 1.076	7.089 ± 0.881	4.559 ± 0.467
	D	0.142 ± 0.021	0.258 ± 0.025	0.256 ± 0.018	0.184 ± 0.039	0.325 ± 0.034	0.226 ± 0.035	0.135 ± 0.029
	E	0.620 ± 0.015	1.226 ± 0.095	1.420 ± 0.101	1.063 ± 0.147	1.499 ± 0.132	1.569 ± 0.154	0885 ± 0.147
	R^2	0.994	0.995	0.996	0.995	0.995	0.998	0.998
	RMS	5.79	11.68	12.50	12.62	17.64	14.73	15.53
	P	4.37	8.18	8.64	9.25	11.77	8.19	7.93
Hasley	*g*	0.019 ± 0.001	0.078 ± 0.002	0.067 ± 0.002	0.082 ± 0.002	0.083 ± 0.002	0.085 ± 0.009	0.076 ± 0.003
	n	1.602 ± 0.035	0.973 ± 0.022	0.965 ± 0.023	0.904 ± 0.019	0.949 ± 0.027	0.829 ± 0.021	0.954 ± 0.022
	R^2	0.975	0.993	0.993	0.994	0.992	0.992	0.991
	RMS	17.60	34.27	36.99	27.04	20.30	94.24	37.23
	P	10.21	16.79	17.58	14.78	12.63	39.55	16.55

The GAB equation has been frequently applied to describe the sorption isotherms of many products due to the physical meaning of the model, which facilitates the interpretation of obtained results [38,49,50]. However, the constant *C* of the GAB model should be in the range of $5.67 \leq C \leq \infty$ because outside of this region the monolayer capacity is estimated with an error larger than 15.5% [51]. Table 3 shows that the values of *C* constant for GAB model did not fulfil this requirement in the case of investigated dried gels. For this reason, the Peleg model was selected to describe the sorption isotherms of apple dried snacks, since it presented the highest R^2 and the lowest *RMS* and *P* values. The Peleg model was also the most appropriate for the representation of sorption behaviour of freeze-dried avocado with maltodextrin and inulin [52], freeze-dried vegetable soups [53], as well as apple puree powders [42].

Figure 1 shows the sorption isotherms plots as experimental data and the Peleg fitted model for freeze-dried apple gels with different compositions (Figure 1a) and P40%DE-MC snacks obtained by the application of different drying methods (Figure 1b). The sorption curves obtained for the freeze-dried agar gel without other ingredients (P0%) and the vacuum-dried foamed gel (P40%De-MC) were sigmoidal in shape, which is characteristic for type II isotherms. The application of the procedure described by Blahovec and Yanniotis [54] based on nonlinear regression analysis of the data and using the plot of a_w/w vs. a_w (plots are not shown) enabled confirming that these isotherms represented type II. Additionally, the values of the *c* constant of the BET model obtained for these dried samples were higher than 2, which also indicated the isotherm type II. However, the BET model can be only applied to predict sorption behaviour at the water activity range lower than 0.5 [55]. In the case of isotherms type II, the interactions between water (sorbate) and food material (sorbent) are stronger than between sorbate–sorbate molecules. The freeze-dried agar gels showed a higher hygroscopicity at low water activity, which may affect the softer texture of the material. The material seemed to behave as a sponge.

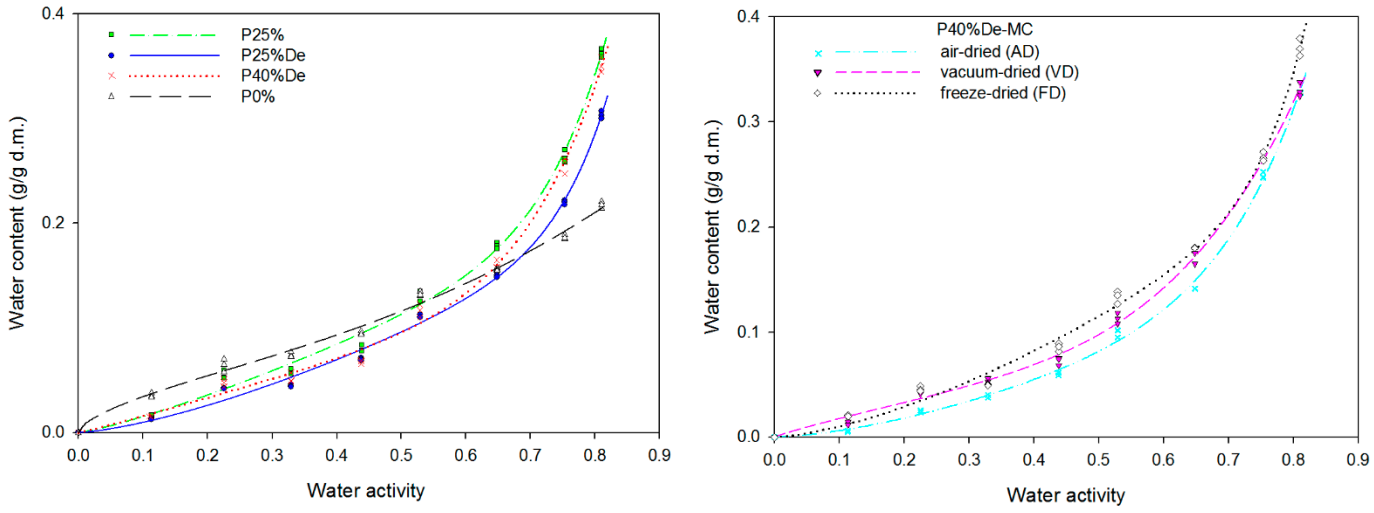


Figure 1. Water sorption isotherms of dried gels: (**a**) freeze-dried (non-foamed) gels with different compositions; (**b**) P40%DE-MC gels dried by AD, VD and FD techniques.

The foamed gel dried by the vacuum-drying method adsorbed a higher amount of moisture than the freeze-dried and the air-dried gels with the same composition at low water activities ($a_w < 0.2$) (Figure 1b). The vacuum-dried gels were probably characterised by more developed surface area as a result of foaming of material as well as expansion of matrix during the dehydration. However, this material (AD-P40%DE-MC) adsorbed less amount of water than freeze-dried gel (FD-P40%DE-MC) in the range of water activity from 0.20 to 0.65. It means that vacuum-dried gels were more stable products with a lower

sorption capacity during the storage (in the analysed water activity range) than lyophilised material.

The other dried gels showed type III behaviour according to the Brunauer–Emmet–Teller (BET) classification [56]. These isotherms presented a convex shape without an inflection point (Figure 1). This behaviour is typical for products with high sugar content and for most fruits. The water content increased linearly with water activity in the range of low and intermediate a_w, this is the so-called multilayer sorption region. The capillary condensation region is characteristic at high water activities, then the rapid increase in water content with a_w can be observed due to sugar dissolution and solute–solvent interactions [57]. The slight sigmoid shape of the isotherm at low water activity is caused by the water sorption by biopolymers, but the sharp increase in water content at high water activities is caused by the presence of sugars [30].

The drying method affected the sorption properties of foamed gel with the same composition (Figure 1b). The isotherms type III were observed for the freeze-dried and the air-dried samples. Air-dried gel was characterised by the lowest moisture content in the investigated range of a_w. It may indicate the presence of less porous structure with closed pores of material obtained by convective drying. This structure of air-dried gels may affect the reduced water sorption by material. Tsami et al. [30] and Lee and Lee [47] concluded the freeze-drying created a porous structure and little shrinkage of products with a higher adsorptive capacity than other drying techniques. However, the addition of different amounts of apple puree and maltodextrin as well as the foaming process modified the sorption properties of dried gels. The foamed gel with the same composition absorbed a higher amount of moisture than non-foamed samples at the intermediate water activities ranging from 0.4 to 0.7. More porous structure of foamed and dried gels may enhance their hygroscopicity.

The composition, structure and size of molecules of food products may affect their sorption behaviour. The addition of maltodextrin (P25%, P25% De) caused the decrease in adsorbed water for the entire range of a_w. Water content of the material significantly decreased during water sorption when the crystallisation of solutes occurred [58]. The freeze-dried apple puree gels with the addition of maltodextrin were probably in amorphous state during the moisture sorption. Additionally, the presence of maltodextrin increased the glass transition temperature and maintained lower hygroscopicity of dried gels.

The increase in the apple addition to freeze-dried agar gels from 25 to 40% for samples with maltodextrin (P25%De, P40%De) caused the significant increase in water content at a given water activity (Figure 1a). It can be linked with the higher concentration of low molecular substances (sugars such as sucrose, fructose and glucose and organic acids) in gels that contained a larger amount of apple puree.

Figure 2 shows the water vapour sorption kinetics of the dried apple puree snacks. Higher water sorption was observed for apple puree dried gel prepared without maltodextrin. A similar effect of maltodextrin addition was noted for lemon juice powder [31] and apple leather [59]. The air-dried and vacuum-dried apple puree gels absorbed less water than freeze-dried samples. The shapes of kinetics curves for all freeze-dried gels were similar, and the samples reached an equilibrium of water uptake after 800 min. However, the amount of absorbed water differed with the composition of gels. Higher addition of apple puree caused the increase in water uptake of freeze-dried samples. Air-dried and vacuum-dried gels were less hygroscopic than freeze-dried gels with apple puree, which is in agreement with the results obtained for water sorption isotherms. The highest rate of water uptake was noted for freeze-dried pure agar gels because the material reached the equilibrium water content after 250 min. The kinetics of water uptake explains the changes of chemical properties during the time of storage with consideration of the rate of change. This type of measurement provides useful information during packaging, which enables estimating the time before the texture and colour of the product become unacceptable at a

given relative humidity [59]. It is possible to observe the sorption behaviour of products after opening the packaging.

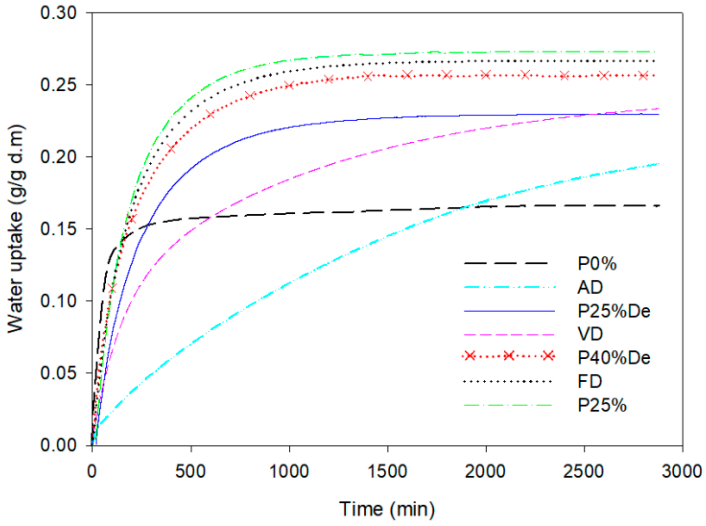


Figure 2. Effect of composition of gels and drying method on the water kinetics uptake of apple puree snacks.

3.2. Effect of Water Activity on the Glass Transition Temperature of Dried Gels

The water activity and glass transition temperature are crucial parameters in the evaluation of changes occurring in food products. Sugar recrystallisation is the main problem of stored products that contain low-molecular sugars in an amorphous state. Dried fruit products are commonly in an amorphous state. These materials are not stable due to a lack of thermodynamic equilibrium [24].

Values of glass transition temperature T_g obtained for selected dried apple puree gels and freeze-dried agar gel as a function of equilibrium water content are shown in Figure 3a. The experimental data and the Gordon–Taylor fitted model were presented. The glass transition temperature decreased with increasing water content due to the plasticising effect of water. Additionally, the addition of maltodextrin to apple puree gels caused the increase in T_g of the freeze-dried sample. A similar effect of a carrier on the T_g of different dried products was observed for mango powder [60], apple puree powder [42] and date syrup powder [61]. The highest glass transition temperature at the investigated range of water content was observed for freeze-dried agar gel without the addition of apple puree. The application of the air-dried method enabled obtaining apple snacks with slightly higher values of T_g in comparison with other dried apple puree gels. It means that the air-dried product will be more stable during the storage because the phase transitions occur at higher water activity at room conditions than for other dried gels. In addition, based on the sorption behaviour, it can be stated that the air-dried gel was less hygroscopic than other materials.

The parameter T_{gs} of the Gordon–Taylor model (Equation (8)) showed that the drying method of gels with the same composition (P40%DE) did not affect the T_{gs} values, but the k parameter obtained for the vacuum-dried sample was significantly higher than for the air-dried gel (Table 4). The k parameter is related to the plasticising effect of water. Higher value of k can be linked with a decrease in the glass transition temperature of the sample [60,62]. Lower susceptibility to water sorption of air-dried gels can be linked to the higher glass transition temperature. The increase in pulp addition caused a slight increase in T_{gs} (Table 4). Fongin et al. [60] stated that the glass transition temperature of anhydrous sample T_{gs} should be independent of pulp content in freeze-dried mango powder because the solid matrix of pulp is intrinsically crystalline. However, the increase in apple pulp addition in freeze-dried gels caused the increase in low molecular sugars and their presence affected a slight decrease in glass transition temperature. The addition of maltodextrin

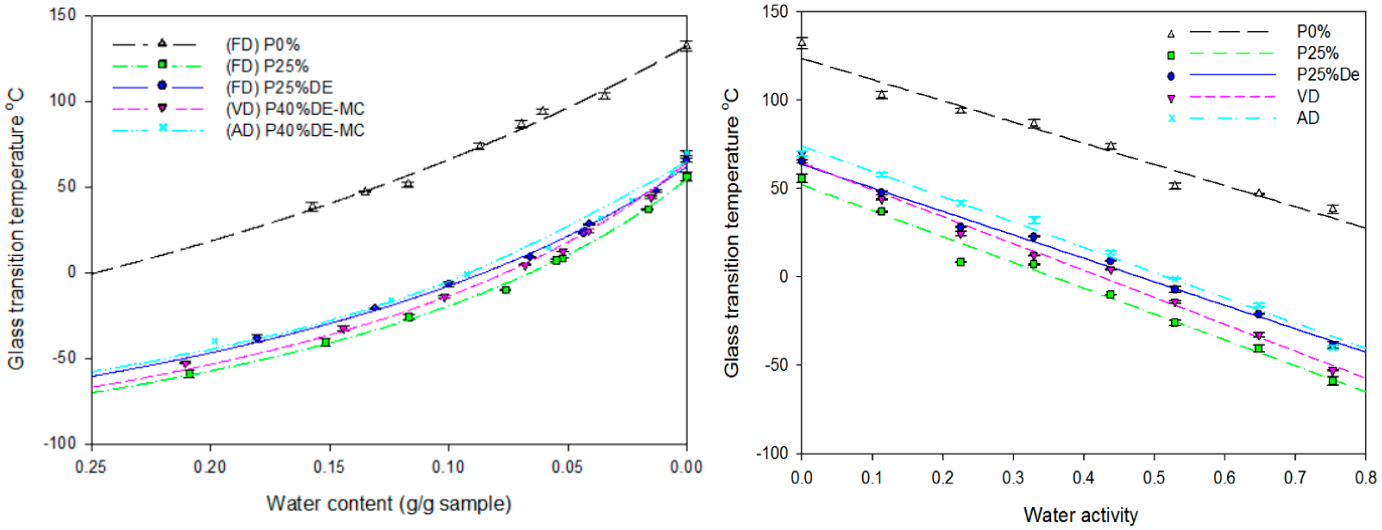
and a lower amount of incorporated pulp affected a fabrication of dried gels with higher stability (the possibility of structure collapse and the changes in texture of the product can be limited).

Table 4. Parameter of Ross and Gordon–Taylor models and critical values of moisture content (x_{wc}) and water activity a_{wc} at a temperature of 25 °C.

	Parameters of Model	Variants of Dried Gels				
		(FD) P0%	(FD) P25%	(FD) P25%De	(VD) P40%DE-MC	(AD) P40%DE-MC
Gordon-Taylor model	T_{gs}	132.32 ^{d,*}	54.39 ^{a}	61.81 ^{b}	64.24^{c}	64.58 ^{c}
	k	2.97 ^{a}	5.76 ^{c}	4.92 ^{a}	5.71 ^{c}	4.85 ^{a}
	R^2	0.989	0.997	0.995	0.992	0.992
	RMS	4.57	13.13	10.98	16.16	13.76
	P	3.48	7.58	8.79	11.50	4.85
Ross model	A	123.82 ^{d}	52.21 ^{a}	63.82 ^{b}	64.72 ^{b}	74.19 ^{c}
	B	−119.96 ^{d}	−146.48 ^{b}	−132.97 ^{c}	−152.50 ^{a}	−143.26 ^{b}
	R^2	0.965	0.985	0.993	0.988	0.991
	RMS	8.53	38.67	16.27	29.71	18.63
	P	6.83	20.12	11.74	19.54	14.54
T_g = 25 °C	x_{wc} (g/g_{dm})	0.225 ^{a}	0.032 ^{b}	0.047 ^{c}	0.042 ^{c}	0.045 ^{c}
	a_{wc}	0.837 ^{e}	0.181 ^{a}	0.301 ^{c}	0.260 ^{b}	0.353 ^{d}

* the different letters in the rows indicates the significant difference between the obtained values for samples, $p \leq 0.05$.

The relation between the water activity and glass transition temperature can be described by Roos model (Figure 3b). However, the calculated RMS, R^2 and P values showed that the experimental data obtained in this study did not fit well for the Roos equation (Equation (9)).



(**a**) (**b**)

Figure 3. Changes of the glass transition temperature of selected dried gels to (**a**) water content; (**b**) water activity.

A higher value of critical water content and critical water activity at the glass transition temperature of 25 °C obtained from the relations of water content-water activity and water content-glass transition temperature may indicate the greater stability of physical properties of products during storage. Dried apple puree gels with maltodextrin were characterised by a significantly higher a_{wc} than dried gel without carrier addition. A similar tendency has been observed for grapefruit and mango powders [20,60]. In the case of the air-dried apple

puree, higher critical values of water activity than for other dried gel with apple puree addition were observed. The results indicate that the freeze-dried agar gel (without apple puree addition) stored at room temperature will change its state from glassy to rubbery at a high-water activity of 0.837. The air-dried sample will be stable at water activity lower than 0.353.

3.3. Microstructure of Dried Gels

Figure 4 presents the structure of dried samples of different compositions or produced with the application of different drying techniques.

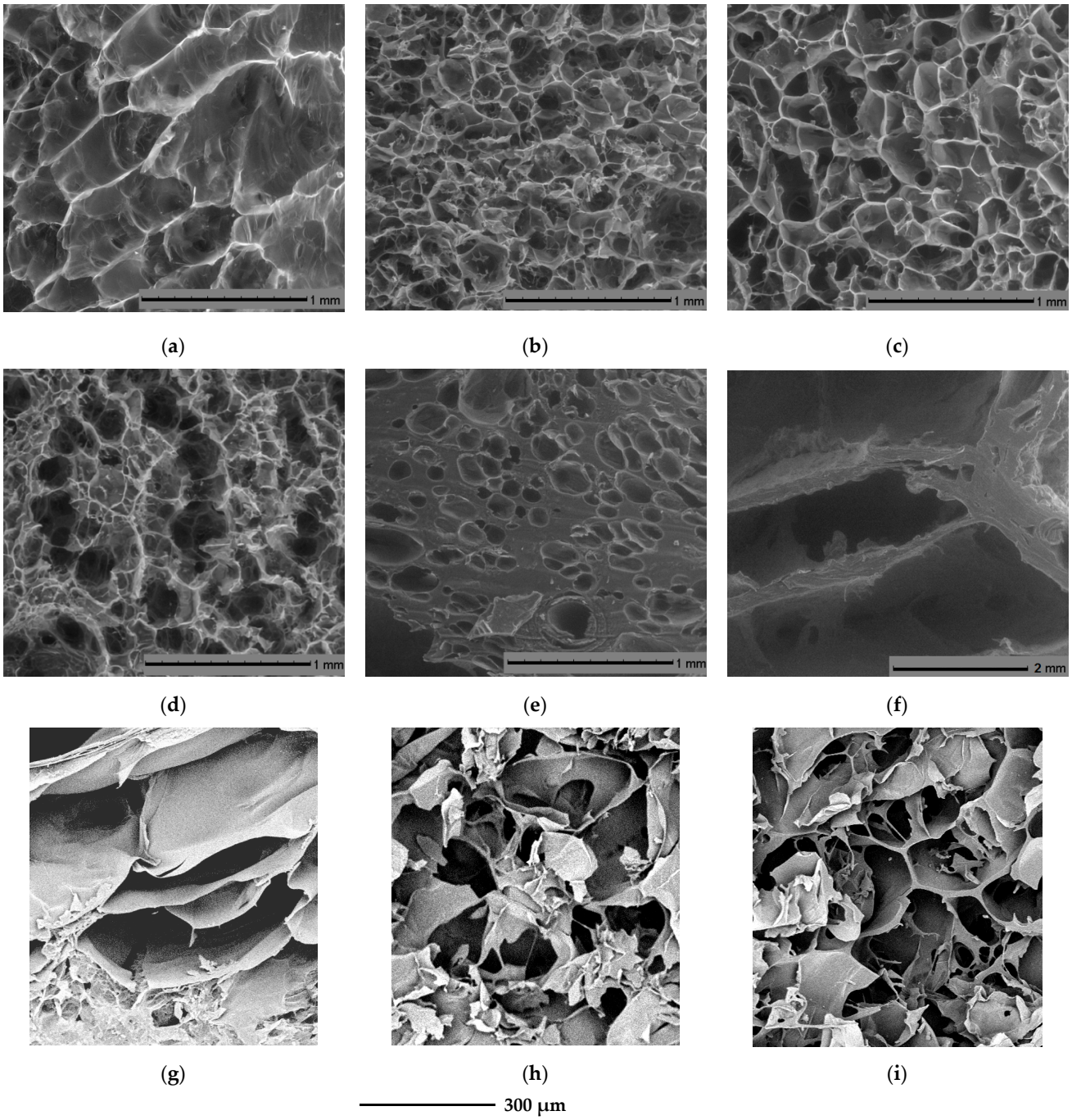


Figure 4. SEM views of different freeze-dried gels; (**a**,**g**) FD-P0%; (**b**,**h**) FD-P25%; (**c**,**i**)-FD- P40%DE; (**d**)-FD-P40%DE-MC; (**e**)-AD-P40%DE-MC; (**f**) VD-P40%DE-MC.

The addition of apple puree to agar gels changed the structure of freeze-dried products (Figure 4a,b). Long pores (Figure 4a,g) with thin walls can be observed for dried agar gels (without other ingredients). Freeze-dried samples with 25% addition of apple puree (Figure 4b) contained more round pores. An increase in puree concentration in gel led to obtaining freeze-dried samples with larger regular pores, but higher concentration of solids contributed to producing the matrix walls with a higher thickness (Figure 4c,i). Aeration of agar sol with the addition of apple puree affected the structure after the freeze-drying of the samples (Figure 4d). These dried gels were very porous and the thickness of pores' walls was reduced. Ciurzyńska and Lenart [63] also obtained the freeze-dried hydrocolloid gel, which was aerated. The structure of materials was very fragile and porous. Based on Figure 4e, it can be stated that air-dried apple puree gel was less porous with more visible shrinkage and contained more closed pores. Application of vacuum-drying of apple gel caused larger holes inside the material with thick walls of pores (Figure 4f). Sundaram and Durance [64] also noticed that the air-drying and the vacuum-drying of locust bean gum-pectin-starch composite gel resulted in samples with big holes. However, after the vacuum-drying, the volume of the sample was increased due to puffing. In our study, the same phenomenon was also observed for the vacuum-dried apple puree gel.

4. Conclusions

The nonlinear regression analysis showed that the Peleg model was adequate to describe the water sorption isotherms of all dried apple puree products as well as freeze-dried agar gel. The water adsorption isotherms of most dried gels represented type III sorption behaviour, which is common for a high sugar food. It can be linked with an increase in sugar concentration in gels due to the addition of apple puree. The different shapes of the sorption isotherm (represented type II) of the freeze-dried agar gel (without apple puree) was a result of the low concentration of solids in the material. The higher amount of adsorbed water at lower water activity was observed for the vacuum-dried apple puree gel, which can be related to expanded structure with large pores. However, the thick walls of pores created during the vacuum-drying impeded the hygroscopicity of the material. Low porosity and thick walls of pores in air-dried gels affected the lower moisture sorption and the increase in glass transition temperature. The open-pore structure and the presence of many pores with thin walls in freeze-dried apple gels led to the higher hygroscopicity of samples. The application of this drying method also caused a decrease in glass transition temperature and critical water activity of studied materials. Additionally, freeze-dried gel without maltodextrin was characterised by the low value of glass transitions, which may indicate the presence of the amorphous state. It may lead to the undesirable changes of texture (collapse of structure). The increase in apple puree addition gels from 25 to 40% caused the increase in hygroscopicity during the storage, which can be linked with the higher concentration of low molecular substances. This study shows that the composition of gel and the application of certain drying methods enabled obtaining products with tailored structure and sorption properties. The air-dried apple puree gels can be recommended as the snack's products with the lowest hygroscopicity and the highest stability at room conditions (the highest glass transition temperature and the critical water activity). In addition, a decrease in the amount of apple puree from 40 to 25% in dried gels improved their sorption properties.

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