Moisture sorption characteristics of extrusion-cooked starch protective loose-fill cushioning foams

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A b s t r a c t. The aim of this work was to determine the water vapour sorption properties of thermoplastic starch filling foams processed by extrusion-cooking technique from various combinations of potato starch and two foaming agents: poly(vinyl) alcohol and Plastronfoam, in amount of 1, 2 and 3% each. Foams were processed with the single screw extruder-cooker at two different screw rotational speeds 100 and 130 r.p.m. The sorption isotherms of samples were determined and described using the Guggenheim-Anderson-de Boer model. Also, the kinetics of water vapour adsorption by foams, as a function of time, was measured and fitted with Peleg model. On the basis of the analysis the influence of the applied foaming agents, as well as the technological parameters of extrusion-cooking process in relation to water vapour adsorption by thermoplastic starch foams was demonstrated. There was no difference between the shapes of the isotherms for poly(vinyl) alcohol foams while for Plastronfoam foams a notable difference among foams extruded at 100 r.p.m. was observed in the regions of low and high humidity content. The analysis of the Guggenheim-Anderson-de Boer model parameters showed that the water molecules were less strongly bound with the foam surface when extruded at a lower screw speed.

K e y w o r d s: extrusion-cooking, thermoplastic starch foams, protective loose-fill materials, sorption isotherms, foaming agents

INTRODUCTION

Extrusion-cooking is a technique of fast and intensive processing of materials in which the pressure and temperature of the process have a great influence on the characteristics of the final products (Agbisit *et al.*, 2007; Chanvrier *et al.*, 2013; Conti e Silva *et al.*, 2010; Ilo *et al.*, 1999; İbanoğlu *et al.*, 2006). The temperature of the treatment during the process can be high (up to *ca*. 200°C), the pressure can reach 20 MPa, while the total time is usually very short (30-45 s) (Combrzyński, 2012; Mitrus, 2012; Mościcki and Van Zuilichem, 2011; Suknark *et al.*, 1997). Therefore, the extrusion-cooking process is an example of the so called HTST processes (high temperature, short time) (Frame, 1994; Guy, 2001; Lui and Peng, 2005; Mościcki and Van Zuilichem, 2011; Wójtowicz *et al.*, 2013). Generally, the extrusion-cooking technique can be successfully applied to starch-based foam production (Altskär *et al.*, 2008; Mitrus, 2012; Wang *et al.*, 2005; Wu *et al.*, 2010). The application of food extrusion-cookers gives much better results in processing of starch-based blends than conventional plastic extruders, due to the origin of plant components (Mitrus and Mościcki, 2014).

The types of used foaming agents, as well as the parameters of the extrusion-cooking process, such as temperature, screw rotation, raw material water content, have a direct impact on the density, porosity, mechanical parameters and other physical properties of obtained foams. For example, Van Soest *et al.* (1996) have shown that with increasing screw rotation increased level of crystallization of amylose-rich starch was observed, which influenced the mechanical properties of the final product. The relationship between the extruder-cooker screw rotation and the rheological properties of the processed liquefied blend was also demonstrated in other studies (Tolstoguzov, 1993). The

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functional properties of thermoplastic starch (TPS) depend on the quantity and type of added plasticizers and auxiliary materials, as well (Combrzyński *et al.*, 2012; Mitrus *et al.*, 2010; Mościcki *et al.*, 2007). The best improvement of TPS mechanical properties was obtained with glycerol, as materials became more pliable and easier to work. The structure of a TPS surface depends to a great extent on the starch type used. TPS materials obtained from potato starch exhibited a relatively smooth structure and gradual smoothing of the surface with increasing glycerol content. Pores are the results of excessive granulate expansion during barothermal extrusion-cooking, creating a characteristic honey comb structure (Janssen and Mościcki, 2009).

This paper provides an approach aimed at examining water vapour sorption properties of TPS loose-fill cushioning foams, enriched with two foaming agents: poly(vinyl alcohol) PVA and Plastronfoam PDE. A systematic study of the sorption and adhesion properties of TPS materials will be performed to determine their possible usage as biodegradable packaging materials and its behavior during storage.

MATERIALS AND METHODS

Potato starch of the Superior Standard type (PPZ Trzemeszno, Trzemeszno, Poland) was used in this study. The water content of the starch was 16.7% and the pH 7.4. As the foaming agents: Plastronfoam PDE (VGT Polska Sp. z o.o., Kraków, Poland) and poly(vinyl alcohol) PVA (Avantor-POCH S.A, Gliwice, Poland) were used in amount of 1, 2 and 3% (w/w). In total, 7 various starch-foaming agent blends were prepared. The control blend, containing only potato starch and experimental blends, enriched with a PVA or PDE foaming agents were prepared and moistened up to 18%. The blends were mixed for 20 min in a laboratory ribbon mixer (type 357651, Rowag Rogoźno Wlkp., Poland) until homogeneous mass was obtained.

The extrusion-cooking process was carried out using a single screw extruder-cooker TS-45 (Z.M.Ch. Metalchem, Gliwice, Poland) with L/D = 12. The screw rotation was set at 100 or 130 r.p.m., the temperature profile along the barrel sections (from the feeding zone to the die) was from 80 up to 100°C. A forming circular die with the internal diameter of 5 mm was chosen for the experiment, and thus annular cross-section samples were obtained. Technological experiments were carried out in 3 repetitions. After the extrusion-cooking, the foams were stabilized in room temperature, dried using the shelf dryer (self-constructed) for 24 h at 40°C in natural air convection and stored in closed bags before tests. The coding scheme for the samples name is as follows: XYZ, where X denotes the content of foaming agent (1, 2, 3% w/w), Y denotes the foaming agent type (C – control foam, PVA – poly(vinyl alcohol), PDE – Plastronfoam), Z denotes the extruder screw rotation speed (100-100 r.p.m., 130-130 r.p.m.).

Determination of sorption isotherms was evaluated under various water activities conditions. The equilibrium water content of foam samples at 20°C was determined using the static gravimetric method. Samples weighing *ca.* 2 g were placed inside desiccators conditioned to a constant <u>relative</u> humidity (RH) with saturated salt solutions in order to achieve a water activity range of 0.11-0.84. The following salt solutions were used: LiCl (a_w=0.113), KCH_3COO (a_w=0.225), $MgCl_2$ (a_w=0.329), K_2CO_3 (a_w=0.438), $Mg(NO_3)_2$ (a_w=0.529), $NaNO_2$ (a_w=0.648), NaCl (a_w=0.753), KCl (a_w=0.843), moreover, anhydrous $CaCl_2$ was used to obtain a_w=0.0. Crystalline thymol was used to avoid the growth of mold in desiccators with relative humidity over 70%. Desiccators were placed inside the thermostat cabinet for the period of 3 months, and after this period the weight change of each sample was determined. The analyses were performed for each foam type in triplicate, and the presented data represent mean values. The moisture adsorption data were fitted into the GAB model, defined as follows:

$$u = \frac{u_m C k a_w}{[(1-ka_w)1+(C-1)ka_w]}, \quad (g_{H_2O}\,100\,g_{d.b.}^{-1}) \qquad (1)$$

where: u – the water equilibrium content (g_{H_2O} 100 g of sample dry mass, d.b.), u_m – the monolayer water content (g_{H_2O} 100 $g_{d.b.}^{-1}$), a_w – the water activity of foam samples, C – the kinetic constant related to the sorption in the first layer, k – the kinetic constant related to multilayer sorption (Figura and Teixeira, 2007).

From the BET equation, the water activity of the monolayer water content was calculated (Oniszczuk *et al.*, 2015). Finally, the specific surface area a_{sp} (m² g⁻¹) of the adsorbent was calculated according to the equation:

$$a_{sp} = \frac{u_m \omega N}{M}, \qquad (g\,g_{d.b.}^{-1}) \qquad (2)$$

where: a_{sp} – specific surface area (m² g⁻¹), u_m – the monolayer water content (g $g_{d.b.}^{-1}$), N – the Avogadro number (6.023 10²³ molecules per mol), ω – the water setting surface (1.05 10⁻¹⁹ m² per molecule), M – the water molecule mass (18.015 g mol⁻¹) (Figura and Teixeira, 2007).

The measurements of the water vapour sorption kinetics was performed by continuous recording of the foam weight values with the precision of 0.0001 g, under constant relative humidity level and temperature. Before measurement the samples were dried for 24 h in a vacuum oven at 60°C to remove water. To maintain a constant relative humidity level the saturated solution of NaCl was used (RH = 75.3%). The measurements were performed at 20°C for 18 h without access of light. In an attempt to describe the kinetics of water vapour adsorption, a two-parameter, non-exponential, empirical equation proposed by Peleg (1988) was used. The major advantage of the Peleg model is timesaving by

predicting water sorption kinetics of materials, including equilibrium water content. In a modified form, it can be formulated as (Muszyński *et al.*, 2016):

$$M(t) = \frac{S_0 M_e t}{M_e + t S_0}, \qquad (\text{g g}_{\text{d.b.}}^{-1}) \qquad (3)$$

where: $M(t)$ – the water content at the time t (g_{H_2O} $g_{d.b.}^{-1}$), S_0 – equals the rate of moisture intake during the first period ($t = 0$) (g_{H_2O} h^{-1}), M_e – equal to the final equilibrium water content ($t = \infty$) (g_{H_2O} $_{d.b.}^{-1}$) (Turhan *et al.*, 2002).

The statistical analysis of the models and equations fitting was performed on the base on experimental data. The parameters of the BET model and of the modified Peleg equation were determined with Table Curve 2D software (SYSTAT Software Inc., San Jose USA). The goodness of fit was assessed on the basis of the coefficient of determination R^2, root mean square error RMS and coefficient of residual variation VCR (Nowacka *et al.,* 2012).

RESULTS AND DISCUSSION

The relationship between the water activity and the equilibrium of the water content is shown by the isotherms (Fig. 1). The GAB model provided a very good fit to the experimental sorption data for a whole range of water activity (Table 1) with the worst fit for 1PDE100 and 3PDE100 ($R^2 = 0.971$ and $R^2 = 0.977$, respectively). Further, the coefficient of residual variation showed the highest values for this two foams (VCR = 9.66 and 8.15% for 1PDE100 and 3PDE100, respectively). However, as for all analysed foams RMS and VCR values are below 10 and 20%, respectively it can be stated that BET model described the experimental data correctly. In general, there was no difference between the shapes of the isotherms for PVA foams (Fig. 1, panel A and B). With PDE foams, a notable difference for foams extruded at 100 rpm was observed, especially in the regions of low and high humidity content (Fig. 1, panel C). Therefore, the parameter C in the GAB equation, related to the sorption in the first layer, has the greatest values for PDE100 foams (Table 1). In the middle region of water activity, where the complete moisture monolayer is formed, the shape of the isotherm was similar for all foams. However, the calculated monolayer capacity u_m was the lowest for 1PDE100 foam (Table 1). In general, for the control and all PVA foams, the monolayer capacity u_m, the water activity of monolayer and the specific surface area a_{sp} were greater when foams were extruded at the screw speed of 100 r.p.m. than at 130 r.p.m. (Table 1). In contrast, for 1PDE and 3PDE foams the increase of
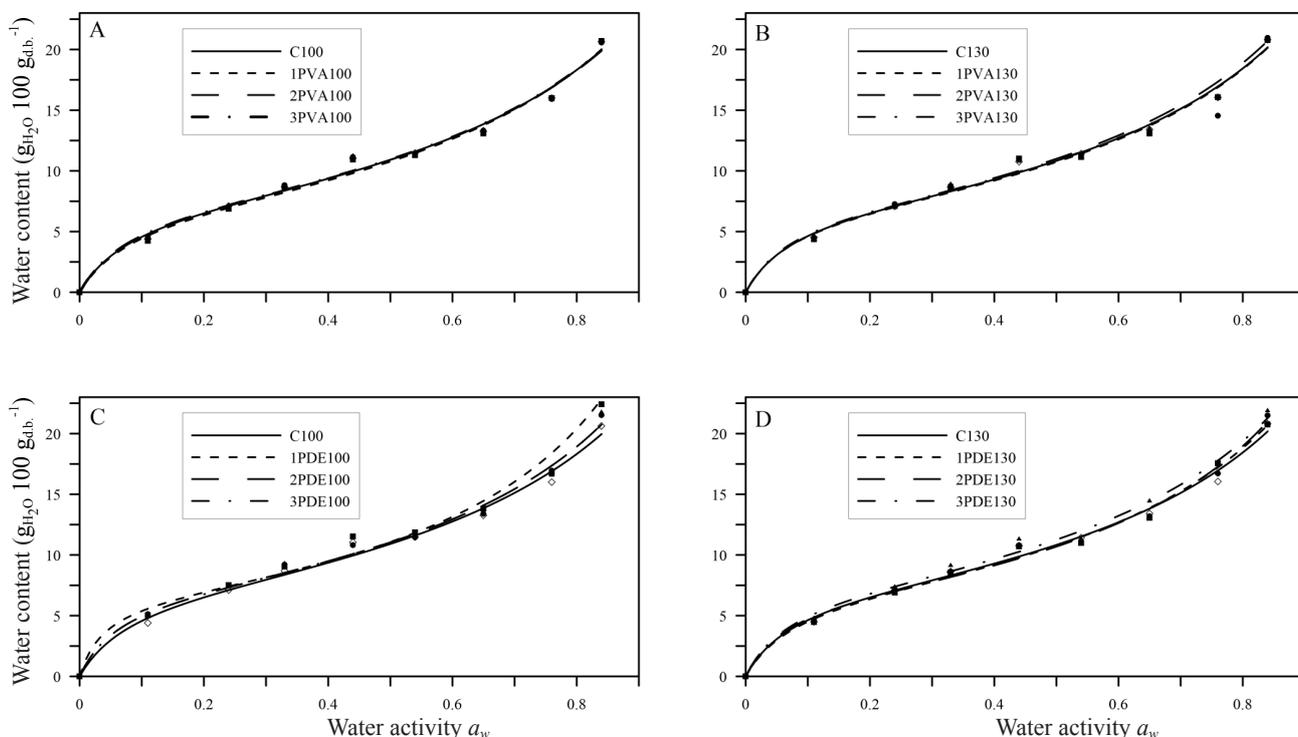

Fig. 1. Sorption isotherms for different types of starch foams (points) approximated with the GAB model (lines). A – control foam (C100) and foams enriched with 1% (1PVA100), 2% (2PVA100) or 3% (3PVA100) of poly(vinyl alcohol) PVA extruded at the extruder screw speed of 100 r.p.m.; B – control foam (C130) and foams enriched with 1% (1PVA130), 2% (2PVA130) or 3% (3PVA130) of poly(vinyl alcohol) PVA extruded at the extruder screw speed of 130 r.p.m.; C – control foam (C100) and foams enriched with 1% (1PDE100), 2% (2PDE100) or 3% (3PDE100) of Plastronfoam PDE extruded at the extruder screw speed of 100 r.p.m.; D – control foam (C130) and foams enriched with 1% (1PDE130), 2% (2PDE130) or 3% (3PDE130) of Plastronfoam PDE extruded at the extruder screw speed of 100 r.p.m.

T a b l e 1. GAB equation parameters, the water activity of the monolayer and the surface area of adsorbent of foams stored at 20°C

Foam type	GAB parameters			Model accuracy			Water activity a_w of monolayer	Specific surface area a_{ap} (m^2 g^{-1})
	u_m (g_{H_2O} 100 gd.b.$^{-1}$)	C	k	R^2	RMS%	VCR%		
C100	7.45	15.82	0.76	0.984	5.34	6.94	0.264	261.5
1PVA100	7.32	15.15	0.77	0.982	5.85	7.36	0.265	257.0
2PVA100	7.41	17.04	0.76	0.989	5.32	7.26	0.257	260.1
3PVA100	7.44	16.55	0.76	0.981	5.05	7.47	0.260	261.2
1PDE100	6.66	30.61	0.85	0.971	7.00	9.66	0.180	233.8
2PDE100	7.21	20.00	0.79	0.988	4.27	5.97	0.231	253.1
3PDE100	7.22	20.00	0.79	0.977	5.62	8.15	0.231	253.5
C130	7.23	17.11	0.78	0.987	4.64	6.13	0.250	253.8
1PVA130	7.13	17.26	0.78	0.982	5.89	7.37	0.249	250.3
2PVA130	7.33	16.53	0.78	0.985	5.22	6.23	0.253	257.3
3PVA130	7.29	18.08	0.77	0.987	4.91	6.04	0.247	255.9
1PDE130	7.09	16.29	0.79	0.990	4.54	5.64	0.251	248.9
2PDE130	6.82	19.68	0.81	9.987	5.77	6.52	0.227	239.4
3PDE130	7.40	18.28	0.79	0.984	5.01	6.84	0.240	259.8

the screw speed resulted in the increase of those parameters. Also, the values of the C parameter of PVA foams differed from those of PDE, especially for foams extruded at 100 r.p.m., where it showed a considerably higher value for PDE foams (Table 1). The k parameter, related to multilayer sorption, was similar for all foams, and ranged from 0.76 (C100) to 0.85 (1PDE100).

All sorption isotherms were of sigmoidal shape therefore can be classified as a type II according to Branauer classification. This type of isotherms is common for starch-rich products (Marzec and Lewicki, 2006; Oniszczuk *et al.*, 2015). The value of C parameter is related to the sorption energies of the monolayer (Enrione *et al.*, 2007). The comparison showed that the lower values were observed for the control foams and the foams extruded with a lower amount of foaming agents. That would suggest that water molecules in those types of foams are more weakly bound to the active site of the polymer matrix compared to the foams containing 2 or 3% of the foaming agent in their blends. As a result, the lower value of C parameter, the higher value of monolayer water activity is generally observed (Table 1). Moreover, for all PDE foams, the C constant of samples extruded at 100 r.p.m. was lower than of samples extruded at 130 r.p.m. This result suggests that the water molecules were less strongly bound with the foam surface of the foams extruded at a lower screw speed (Enrione *et al.*, 2007; Figura and Teixeira, 2007). For the control and PVA foams this effect was not noticed. Furthermore, for all types of foams the k parameter of the GAB equation was significantly lower than 1. Therefore, it could be concluded that there was a difference between the energy associated with the isosteric heat of sorption in a multilayer and condensation

of pure water, as the closer the value of k parameter to 1, the lower the mutual interaction between the absorbed water molecules in a multilayer is observed (Enrione *et al.*, 2007).

Analyzing the shape of the moisture sorption curves (Fig. 2) as a function of time it can be concluded that all the foams absorb it in a similar manner and the parameters of the Peleg equation (Table 2) allow for a proper estimation of these properties. The adsorption curves are all of hyperbolic shape, and a very fit of the model to experimental data was achieved, as coefficient of residual variation VCR did not exceed the value of 20% in all cases. It is worth noting the for all PDE foams extruded at 100 r.p.m. the RMS% exceeded the value of 10% (VCR = 16.60, 12.45, 12.48% for 1PDE100, 2PDE100 and 3PDE100, respectively). However, the worst fit in terms of root mean square error value was obtained for 3PDE130, where calculated RMS% value reached 22.84%.

For all samples, the foams exhibited typical adsorption behavior, absorbing moisture rapidly in the initial phase and slowing down as the water content began approaching equilibrium values. The highest initial sorption rate (S_0 parameter) was observed for PDE foams, especially for 2PDE100 and 3PDE100 (0.108 and 0.087 g_{H_2O} h^{-1}, respectively). Furthermore, it can be noticed that for PVA foams extruded at 130 r.p.m. S_0 value was identical irrespectively of the PVA content in the extruded blend (ranging from 0.040 to 0.041 g_{H_2O} h^{-1}). The equilibrium water content (M_e parameter) during the 18 h long experimental run, was not achieved for any foam. (Table 2). The highest saturation with water vapour was observed for C100 an 1PDE130 foams, which reached in both cases about 86% of their theoretical equilibrium water content predicted by Peleg
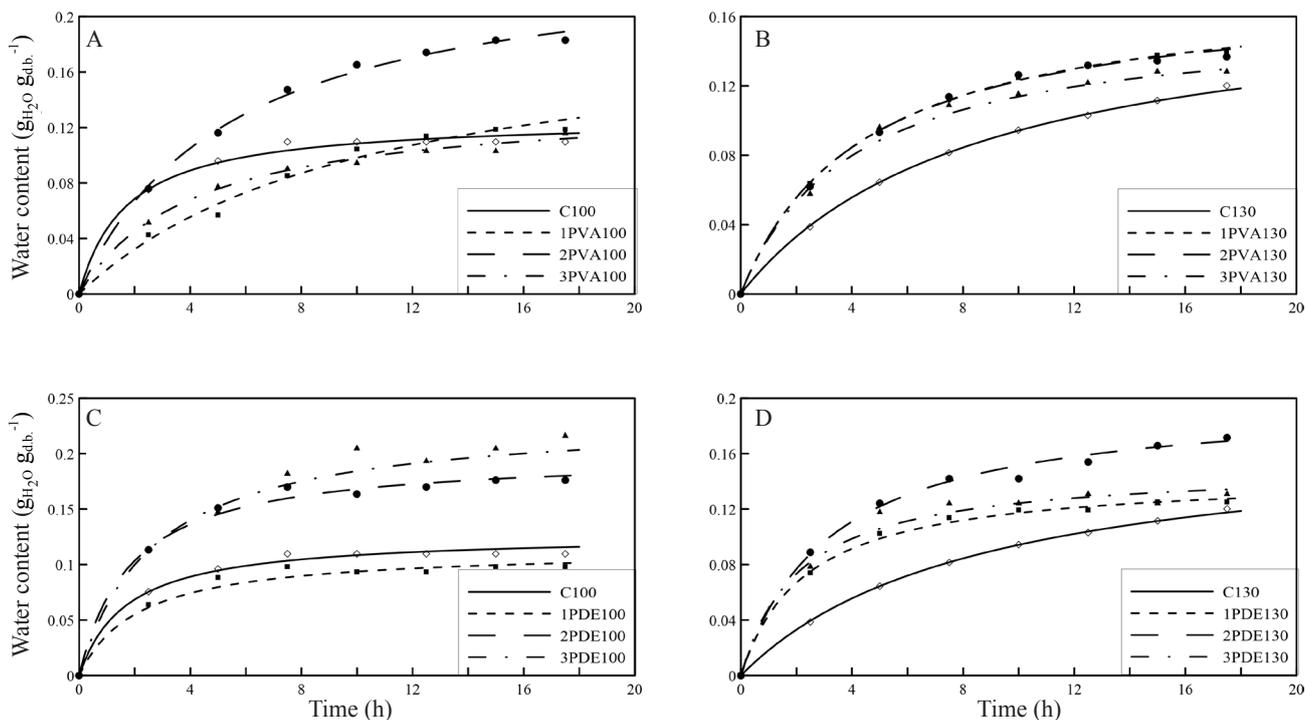
Fig. 2. Changes of water content as a function of the storage time of starch foams in RH 73.5% (points) approximated with Peleg equation (lines). A – control foam (C100) and foams enriched with 1% (1PVA100), 2% (2PVA100) or 3% (3PVA100) of poly(vinyl alcohol) PVA extruded at the extruder screw speed of 100 r.p.m.; B – control foam (C130) and foams enriched with 1% (1PVA130), 2% (2PVA130) or 3% (3PVA130) of poly(vinyl alcohol) PVA extruded at the extruder screw speed of 130 r.p.m.; C – control foam (C100) and foams enriched with 1% (1PDE100), 2% (2PDE100) or 3% (3PDE100) of Plastronfoam PDE extruded at the extruder screw speed of 100 r.p.m.; D – control foam (C130) and foams enriched with 1% (1PDE130), 2% (2PDE130) or 3% (3PDE130) of Plastronfoam PDE extruded at the extruder screw speed of 100 r.p.m.

T a b l e 2. Parameters of the modified Peleg equation of kinetics of water vapour adsorption for starch foams stored in RH 73.5%

Foam type	Peleg equation parameters		Model accuracy		
	S_0 (g_{H_2O} h^{-1})	M_e (g_{H_2O} $g_{d.b.}^{-1}$)	R^2	RMS%	VCR%
C100	0.074	0.127	0.978	4.52	3.89
1PVA100	0.019	0.201	0.982	16.12	5.91
2PVA100	0.046	0.248	0.996	2.52	2.17
3PVA100	0.033	0.139	0.992	5.59	3.02
1PDE100	0.053	0.114	0.944	16.60	7.11
2PDE100	0.108	0.199	0.976	12.45	4.42
3PDE100	0.087	0.233	0.941	12.48	7.66
C130	0.020	0.175	0.997	5.08	1.85
1PVA130	0.040	0.177	0.999	1.23	1.07
2PVA130	0.041	0.176	0.995	2.78	2.42
3PVA130	0.040	0.159	0.992	7.37	3.15
1PDE130	0.062	0.144	0.992	3.98	2.62
2PDE130	0.062	0.200	0.988	9.13	3.58
3PDE130	0.072	0.150	0.952	22.84	6.68

model. Generally, the screw speed did not affect the adsorption rate in further phases or the final equilibrium of water content (M_e parameter) and for all types of foams the effect of initial moisture sorption rate (S_0) on the final equilibrium water content (M_e) was not found.

Water activity is one of the basic parameters characterizing the hygroscopic properties of the material (Figura and Teixeira, 2007). In systems that have not reached their equilibrium water activity a transport of water molecules occurs, which may alter the physical properties of the product. The effect of water on the texture of starch materials can be related to its plasticizing effects by increasing the free volume in the starch polymer (Enrione *et al.*, 2007; Fontanet *et al.*, 1997). Water molecules lead to the relaxation of the starch polymer structure, unveiling new active centers of adsorption. As a result, the extruded starch undergoes transition from its amorphous state to a viscoelastic state and the adsorption capacity increases (Fontanet *et al.*, 1997; Marzec and Lewicki, 2006).

During the extrusion process starch forms amylose complexes, which reduce the availability of free sorption sites in amylose. Also, amylopectin as a highly-branched polymer is likely to have a number of water-binding sorption sites compared to amylose (Enrione *et al.*, 2007). Therefore, the sorption capacity of the final product could also be related to the ratio of amylopectin to amylose in a given starch type. As the increase of water content or water activity increases molecular mobility it also could influence the resistance of the material to deformation (Marzec and Lewicki, 2006; Świetlicka *et al.*, 2015). In our study, all foams had equilibrium water activity of monolayer below 0.300. The additional humidification can lead to an increase in rigidity and brittleness, as a phenomenon of antiplasticization caused by water could occur (Marzec and Lewicki, 2006). Our further studies will be concerned with testing additional fillers to improve the hydrophobic characteristics of extruded foams in order to expand their usage in the packaging market.

CONCLUSIONS

1. The foams produced with various foaming agent content differed in terms of water vapour isotherms and moisture sorption.

2. In general, there was no difference between the shapes of the isotherms for foams containing poly(vinyl) alcohol.

3. With foams containing Plastronfoam, a notable difference for foams extruded at 100 r.p.m. was observed, especially in the regions of low and high humidity content.

4. In general, for the control and all foams containing poly(vinyl) alcohol, the monolayer capacity, the water activity of monolayer and the specific surface area were greater when foams were extruded at the screw speed of 100 than at 130 r.p.m.

5. All sorption isotherms were of sigmoidal shape therefore can be classified as a type II according to Branauer classification.

6. The water molecules were less strongly bound with the foam surface of the foams extruded at a lower screw speed.

7. For all samples, the foams exhibited typical adsorption behavior, absorbing moisture rapidly in the initial phase and slowing down as the water content began approaching equilibrium values. The highest initial sorption rate was observed for foams containing Plastronfoam.

8. The screw speed did not affect the adsorption rate in further phases or the final equilibrium of water content and for all types of foams the effect of initial moisture sorption rate on the final equilibrium water content was not found.

9. The extruded starch undergoes transition from its amorphous state to a viscoelastic state and the adsorption capacity increases.

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