

# Effect of PVA and PDE on selected structural characteristics of extrusion-cooked starch foams

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#### Abstract

The aim of this work was to determine selected physical properties of biodegradable thermoplastic starch (TPS) filling foams manufactured by extrusion-cooking technique from different combinations of potato starch and two additives: poly(vinyl alcohol) PVA and Plastronfoam PDE. Foams were processed with seven starch/additives combinations at two different extruder-cooker's screw rotational speeds. The densities of starch foams depended significantly on the additive type and content. The linear relationship between the Young modulus and the ultimate compression force and apparent density was found. The foams processed with the addition of PVA had low density, porosity and lower values of the Young modulus than the foams prepared with PDE.

**Keywords:** *extrusion-cooking, thermoplastic starch foams, protective loose-fill materials, physical properties, functional additives.* 

# 1. Introduction

With the current focus on environmental credentials, producers of packaging foam materials are under growing pressure to develop products based on natural renewable raw materials<sup>[1-4]</sup>. The pro-environmentally oriented consumers are becoming increasingly aware that the use of petroleum-based non-degradable packaging for short-term use is not adequate<sup>[5-7]</sup>. Therefore, the search for safer, environmentally friendly packaging materials has become an important issue in developed countries and promoted the development of biopolymer-based materials, such as the packaging materials manufactured on the basis of completely biodegradable thermoplastic starch (TPS)<sup>[8-12]</sup>. To succeed, the TPS-based packaging must comply with the quality and safety requirements<sup>[13,14]</sup>. It is also important, that it could be processed with the standard equipment used in plastic processing.

According to the granular structure of starch there have been observed several negative characteristics for strachy products. Increased amount of starch in blends resulted lowering the mechanical properties of biopolymers as reported by Nafchi et al.<sup>[15]</sup>. There are many works available describing the mechanical properties of TPS-based materials<sup>[16-22]</sup>.

The mechanical features of starch-based bipolymers are strongly dependent on the type and amount of additives used, mainly plasticizers and elastomers. Many authors also examined the behavior of starch-based blends<sup>[23-25]</sup>. In this case starch was bound with other biopolymers, often with biodegradable polyesters, like polycaprolactone, polyester amide or with polylactide acid (PLA)<sup>[26-28]</sup>. For example, the addition of PLA increased the mechanical properties and resistance to water of final products<sup>[15]</sup>. TPS/PLA blends are characterized by improvement in thermal stability, inhibition of starch retrogradation and higher resistance to water at high relative humidity<sup>[29-31]</sup>.

Other auxiliary materials, like poly(vinyl alcohol), assist in controlling the adhesive properties of the material surface<sup>[32-34]</sup>.

The results of application of glycerol as plastycizer showed reduced stiffness and improved fracturability of starch-based foams. Moreover, poly(vinyl alcohol) has to be indicated as an effective additive able to decrease water absorption in biobased foams<sup>[35,36]</sup>. These types of materials may be used as alternative environmentally friendly packaging materials with cushioning features.

As described by Kaisangsri et al.[37] the addition of plant fibers to foamed materials based on starch improved the mechanical characteristic of these products, especially as bending resistance and compression resistance The opposite characteristics were found by Carr et al.<sup>[38]</sup> if manioc fibers was used as an additive and decreased mecanical strength of foamed materials was observed. The increase in fibers quantity has resulted in foams with higherdensity and less flexibility, whatever the fiber type. Mostfibers quantity did not improve the foam strength. Steven et al.[39] concluded that strength of foams at the bending tests was similar for starch-lignin foams compare to polystyrene foams, and the maximum stress results were lower while elastic modulus was higher for starch-lignin biopolymers. Classification of foamy materials could be based on the mechanical characteristics, especially elastic modulus<sup>[4]</sup>. Physical properties of foams are extremaly important for practical application of starch-based products. For these reasons several tests as compression, elongation, or bending could be performed for the evaluation of Young modulus, compression stress and strain or breaking resistance. The idea of polymer-starch blends gave the possibility to achieve products undergo dezintegration or degradation without lowering the functional and mechanical properties<sup>[3,6,40]</sup>.

Several works have been done for imporovement of physical and mechanical properties of starch-based biopolymers, but still there are a lot of possibilities to apply several components and functional additives to create desirable characteristics of final products depend on applications and needs of final users.

This paper provides an approach aimed at examining the selected structural characteristics as density, porosity and mechanical strength of new type of biodegradable loose-fill cushioning foams based on potato TPS enriched with two functional additives– poly(vinyl alcohol) PVA and Plastronfoam PDE processed under various screw speed of extruder.

# 2. Materials and Methods

#### 2.1 Materials and samples preparation

Potato starch of the Superior Standard type (PPZ Trzemeszno, Trzemeszno, Poland) with amylose content of 26.97% was used in this study. The moisture content of the starch was 16.7% and the pH 7.4. The extrusion-cooking process parameters and raw materials and additives were selected on the base of the preliminary study. First step included application of selected additives as glycerin, PDE, PVA, guar gum, xanthan gum, gelatin, sodium dicarbonate, albumin, carrageenan, monoglyceride E471, mineral talc. These additives were used as foaming, emulsifying or stabilizing agents for the proper structure of starch-based foams. The results of most of the additives used were not satisfactory for products' quality so the only two were selected for further tests. As the functional additivies Plastronfoam PDE as a white powder (VGT Polska Sp. z o.o., Kraków, Poland) and poly(vinyl alcohol) PVA as a white powder (Avantor-POCH S.A, Gliwice, Poland) were used. The control blend, containing only potato starch was prepared and six experimental blends, enriched with functional additives, PVA or PDE were prepared as presented in the Table 1. In total, seven various raw materials blends were prepared. Also, the necessary amount of water was added to the mixtures, so the total moisture content of all prepared blends was 18%. The blends were mixed for 20 minutes in a laboratory ribbon mixer until homogeneous mass was obtained.

The extrusion-cooking process was carried out using a single screw extrusion-cooker TS-45 (Z.M.Ch. Metalchem, Gliwice, Poland) with L/D = 12. The screw rotational speed was set at 100 or 130 rpm, so 14 different types of foams were produced (7 different blends processed at two different screw rotations), as presented in the Table 1.

The temperature profile along the barrel sections (from the feeding zone to the die) was similar for all treated blends and varied from 80 up to 100 °C. A forming circular die with the internal diameter of 5 mm was selected for the experiment, and thus annular cross-section samples were obtained, single foam characterized the size of 20 mm in length and approx. 10 mm in diameter depends on expansion

intensity. In total, After the extrusion-cooking, the foams were cooled down at room temperature and dried in an air oven at 40 °C for 24 h. Examples of the tested foams are presented in the Figure 1.

### 2.2 Determination of true and appearent densities

Measurements of the true and apparent densities as well as porosity were evaluated for all the samples processed at various conditions. The apparent (bulk) density was measured by a GeoPyc1360 dry flow pycnometer (Micromeritics, Inc., Norcross, GA, USA) with the consolidation force of 50 N, while the measurements of the true density (or material density) of foam slices was performed with the AccuPyc 1330 helium gas pycnometer (Micromeritics, Inc., Norcross, GA, USA). All measurements were performed with 5 repetitions. The parameters of the measurements of both densities were described in details by Muszyński and coworkers<sup>[41]</sup>.

The apparent  $\rho_e$  and true  $\rho_t$  densities (g·cm<sup>-3</sup>) of samples were determined and the results were used to calculate porosity P (in %) and specific pore volume SPV (cm<sup>3</sup>·g<sup>-1</sup>), according to Equations 1 and 2:

$$P = \left(1 - \frac{\rho_e}{\rho_t}\right) 100\% \tag{1}$$

$$SPV = \frac{1}{\rho_t} + \frac{1}{\rho_e} \tag{2}$$

where: P = porosity (%),  $\rho_e$  = apparent density (g·cm<sup>-3</sup>),  $\rho_e$  = true density (g·cm<sup>-3</sup>), SPV = specific pore volume (cm<sup>3</sup>·g<sup>-1</sup>).

#### 2.3 Mechanical measurements

The mechanical properties were examined with a Zwick BDO-FBO0,5TH universal testing machine (Zwick GmbH & Company KG, Ulm, Germany), linked to a computer with testXpert II 3.3 test software (Zwick GmbH & Company KG, Ulm, Germany). Compression test was applied between two flat plates with dimensions of 100×100mm each, bottom plate was stationar and upper plate was movable with the test speed of 3.0 mm s<sup>-1</sup>. The foams were placed horizontally

 Table 1. Sample coding scheme depending on the functional additive percentage content and speed of the extruder-cooker screw.

C	Blend	compositio	Extruder-cooker	
code	Potato starch	PVA	PDE	screw speed (rpm)
C100	100	0	0	100
1PVA100	99	1	0	100
2PVA100	98	2	0	100
3PVA100	97	3	0	100
1PDE100	99	0	1	100
2PDE100	98	0	2	100
3PDE100	97	0	3	100
C130	100	0	0	130
1PVA130	99	1	0	130
2PVA130	98	2	0	130
3PVA130	97	3	0	130
1PDE130	99	0	1	130
2PDE130	98	0	2	130
3PDE130	97	0	3	130



Figure 1. The structure of starch foams obtained at 130 rpm: (a) control foam; (b) foam with 3% PDE addition; (c) foam with 3% PVA addition.

on a flat plate, so that both compression plates were perpendicular to the axis of the sample, therefore the samples were compressed in the direction parallel to their diameter. The measuring head compressed the sample in one cycle to 50% of its original diameter. Measurements for each foam type were performed with 10 repetitions. From the obtained stress-strain curves the following structural parameters were determined: the ultimate compression force (N) as the force causing sample fracture, and the Young modulus (MPa) as the deformation resistance of the foam<sup>[42-44]</sup>.

# 2.4 Statitical analysis

The statistical analysis of the structural characteristic (densities, porosity) and mechanical properties (Young modulus, ultimate compression force) of starch foams was performed with the software package Statistica 12.0 (StatSoft Inc., Tulsa, OK, USA) following a general linear model (GLM). The statistical model included the following variables: the functional additive type and content and the screw rotational speed as well as interactions between the screw rpm and functional additive type. Mean values were compared by Tukey's multiple comparison test, the probability level of p<0.05 was considered as statistically significant. A two-dimensional linear Pearson analysis was used to calculate the corresponding correlation coefficients

(r values) between the structural characteristics of tested foams and the mechanical strength (Young modulus and ultimate compression force); p value <0.05 was considered as statistically significant.

# 3. Results and Discussions

The two of functional additives showed various effect on starch-based foams according to its functionality nd technological approach. Plastronfoam PDE is the foaming agent dedicated for processing temperature up to 210 °C for a maximum gas yield, suitable as a blowing agent in plastic extrusion as well as injection molding. This is endothermic multi-component system, based on sodium bicarbonate and citric acid derivatives, starting its decomposition at 140 °C. Polyvinyl alcohol (PVA) is a particularly well-suited highly polar synthetic polymer for the formulation of blends with natural polymers. In the study of Shen et al.<sup>[45]</sup> polyvinyl alcohol (PVA) was used to improve the toughness of UF foam. Polyvinyl alcohol has a high tensile strength and flexibility and has been used in UF resin synthesis for commercial use. Therefore, to obtain an excellent foam material the proper amount of added PVA affected on superior toughness, compression strength, and morphological characterization of UF foams. According to Wang et al.[46] chitosan/PVA foams demonstrated interconnected and open-cell structures with large pore size.

The control foams (C100 and C130) varied considerably compared with the foams enriched with functional additives, in terms of their structural characteristics (Figure 1a), its structure was irregular with the presence of big internal empty cells after water evaporation during expansion. Addition of PDE influenced positively on foams structure giving the homogenous internal structure with a large number of smalluniform cells formed during the expansion of samples (Figure 1b). This structure was the most desirable and the samples with PDE addition, especially with 2 and 3% of PDE content, characterized compact structure less resistant for external deformations. If PVA was used as an additive the samples characterized with higher dimension of internal cells but much smaller than observed in control sampled without additives (Figure 1c).

The results of both apparent and true densities found for samples with the addition of PDE and PVA were significantly lower than for the control foams, especially there is clear evidence of decreased the apparent density of foams with foaming agents (Table 2). Similarly, the porosity and specific pore volume were the lowest for the extruded foams processed with addition of PDE among the all the tested extrudates.

Moreover, the screw speeds applied during the extrusion-cooking of control samples have no significant effect on the structural characteristics and mechanical properties of control foams, the only values of the apparent density significantly decreased from 0.439 to 0.379 g·cm<sup>-3</sup> (reduction of 14% was observed, p<0.05) when increased screw speed was applied during processing. In contrast, there was observed an effect of the screw speed on the structural properties of foams containing functional additives. In the case of foams with PVA there were significant differences between the true density of the foams containing 1% of PVA extruded at 100 and 130 rpm (increase of 32%; values of

Table 2. The structural characteristics of starch foams.

0.983 and 1.300 g·cm<sup>3</sup> for 100 and 130 rpm, respectively). The apparent density of all tested foams with PVA addition decreased when the blends were extruded at higher screw speeds (decrease of 22%, 35% and 25% for foams with 1, 2 and 3% of PVA, respectively). However, the porosity values increased for 1PVA130 and 2PVA130 (for 15% and 8%, respectively) while the specific pore volume increased for 2PVA130 and 3PVA130 blends (for 45% and 31%, respectively).

In the case of foams extruded from blends containing the PDE functional additive, the extrusion higher speed significantly increased the true density (by 48%, 81% and 74% for blends containing 1, 2, and 3% of PDE, respectively) and porosity (by 14%, 15%, 29%, for blends containing 1, 2, and 3% of PDE, respectively) of tested extrudates, and decreased the specific pore volume of samples containing 1, 2 and 3% of PDE (8, 25 and 15%, respectively). Generally, with PDE application it could be noticed that with an increase of the functional additive content a significant reduction of true and apparent densities was observed. Also, the values of true and apparent densities were significantly greater for PVA supplemented foams than for those extruded with PDE. Similarly, both the porosity and specific pore volume depended significantly on the functional additive type and content and a greater value of specific pore volume was observed for PDE foams. The results of compression tests, presented in the Table 3, showed that control starch foams extruded with various screw speed were characterized with the greatest ultimate compression force (204.1 and 162.9 N, for C100 and C130, respectively).

Moreover, these samples were characterized by a significantly greater Young modulus. For both functional additives it can be noticed, that with an increasing amount of the functional additives in blends the decrease of mechanical parameters was observed, irrespective of the functional additive type. Typical curves of compression tests for samples extruded at 130 rpm are shown on Figure 2.

E	True density	Apparent density	Porosity	Specific pore volume
Foam type	(g·cm <sup>-3</sup> )	(g·cm <sup>-3</sup> )	(%)	(cm <sup>3</sup> ·g <sup>-1</sup> )
C100	$1.227\pm 0.107^{\text{gh}*}$	$0.439 \pm 0.068^{\rm h}$	$64.14\pm4.74^{ab}$	$3.141\pm0.69^{\rm a}$
1PVA100	$0.983 \pm 0.062^{\rm ef}$	$0.295 \pm 0.022^{\rm f}$	$69.81\pm3.44^{bcd}$	$4.422 \pm 0.245^{\rm bc}$
2PVA100	$1.198\pm0.086^{\text{fgh}}$	$0.289 \pm 0.032^{\rm f}$	$75.61\pm4.06^{\rm def}$	$4.334 \pm 0.369^{bc}$
3PVA100	$1.406\pm0.164^{\rm h}$	$0.235 \pm 0.026^{\rm e}$	$83.15\pm2.10^{\text{gh}}$	$5.02\pm0.589^{\rm c}$
1PDE100	$0.635 \pm 0.126^{\rm bc}$	$0.184\pm0.029^{\text{cde}}$	$70.78\pm2.65^{\text{cd}}$	$7.159 \pm 0.980^{\rm ef}$
2PDE100	$0.411\pm0.070^{ab}$	$0.130\pm0.007^{ab}$	$67.79\pm4.27^{\rm bc}$	$10.222 \pm 0.819^{\rm g}$
3PDE100	$0.262\pm0.031^{\text{a}}$	$0.109\pm0.006^{\rm a}$	$57.96\pm5.05^{\rm a}$	$13.07 \pm 0.721^{\rm h}$
C130	$1.202\pm0.068^{\text{fgh}}$	$0.379\pm0.038^{\text{g}}$	$68.43\pm3.30^{\mathrm{bc}}$	$3.496\pm0.273^{ab}$
1PVA130	$1.300\pm0.265^{\rm h}$	$0.231\pm0.034^{\text{de}}$	$81.77\pm3.75^{\rm fgh}$	$5.508\pm0.729^{\text{cd}}$
2PVA130	$1.068\pm0.133^{\rm efg}$	$0.187\pm0.011^{\text{cde}}$	$82.27 \pm 1.85^{\text{gh}}$	$6.298 \pm 0.399^{\rm de}$
3PVA130	$1.238\pm0.160^{\text{gh}}$	$0.175\pm0.019^{\mathrm{bc}}$	$85.74\pm1.85^{\rm h}$	$6.595 \pm 0.627^{\text{ef}}$
1PDE130	$0.938\pm0.077^{\text{de}}$	$0.181\pm0.016^{\text{bcd}}$	$80.63 \pm 1.55^{\text{efgh}}$	$6.624 \pm 0.531^{\rm ef}$
2PDE130	$0.743\pm0.119^{\rm cd}$	$0.160\pm0.018^{abc}$	$78.15\pm2.98^{\text{efg}}$	$7.69\pm0.845^{\rm f}$
3PDE130	$0.457\pm0.041^{ab}$	$0.113 \pm 0.011^{a}$	$75.05\pm3.89^{\text{de}}$	$11.14\pm0.81^{\text{g}}$
<i>p</i> -value F.A.*	< 0.001	< 0.001	< 0.001	< 0.001
p value S.S.	< 0.001	< 0.001	< 0.001	0.723
p value F.A. x S.S.	< 0.001	< 0.001	< 0.001	< 0.001

a-hMeans in the same column with different superscripts differ significantly (p<0.05). \*F.A. = functional additive; S.S. = screw speed.

Table 3. The	e mechanical	properties	of foams	with	functional
additives.					

Foam type	Young modulus (MPa)	Ultimate compression force (N)
C100	$563.8\pm 87.7^{\rm f^{*}}$	$204.1 \pm 33.1^{\circ}$
1PVA100	$404.0\pm71.8^{\text{e}}$	$100.0\pm18.6^{\rm b}$
2PVA100	$414.5\pm95.3^{\text{de}}$	$88.0\pm14.9^{\rm b}$
3PVA100	$373.1\pm50.8^{\text{de}}$	$88.4\pm23.8^{\rm b}$
1PDE100	$387.3\pm55.4^{\text{e}}$	$69.0\pm19.4^{\rm ab}$
2PDE100	$314.7\pm58.0^{\text{cde}}$	$54.3\pm15.8^{ab}$
3PDE100	$197.0\pm56.3^{abc}$	$30.0\pm12.1^{\rm a}$
C130	$566.5\pm55.6^{\rm f}$	$162.9 \pm 71.6^{\circ}$
1PVA130	$371.4\pm53.0^{\text{de}}$	$86.4\pm23.3^{\rm ab}$
2PVA130	$310.2\pm69.2^{\text{bcd}}$	$76.0\pm43.0^{\rm ab}$
3PVA130	$206.1\pm57.7^{ab}$	$60.7\pm11.2^{\rm ab}$
1PDE130	$342.4\pm57.9^{\text{de}}$	$64.0\pm31.1^{\rm ab}$
2PDE130	$311.9\pm68.9^{\text{cde}}$	$57.2\pm18.3^{\rm ab}$
3PDE130	$127.1\pm33.4^{\mathrm{a}}$	$33.6\pm14.0^{\rm a}$
p value F.A.*	< 0.001	< 0.001
p value S.S.	< 0.001	0.004
p value F.A. x S.S.	< 0.001	0.632

 $^{\rm a-f}$  Means in the same column with different superscripts differ significantly (p<0.05). \*F.A. = functional additive; S.S. = screw speed.

However, some evident differences between PVA and PDE foams could be observed. The Young modulus of foams with PDE was among the lowest recorded values and their ultimate compression forces were the lowest of all types of examined extrudates (Table 3). Moreover, it can be stated that foams with the same amount of PVA as functional additive were characterized by a greater Young modulus when extruded at 100 rpm than at 130 rpm. However, the general influence of the interactions between the functional additive level and the screw speed applied during processing was not statistically significant at the ultimate compression force (p = 0.632). All of the above results allow drawing the conclusion that the foams extruded without functional additives were more flexible and were characterized by a higher mechanical strength.

Figure 3 shows the correlation between the apparent density and the measured structural properties – the Young modulus of the extruded starch foams. The calculated Pearson's correlation coefficient revealed that significantly positive correlations were found. On the Figure 4 the correlation between the apparent density and the ultimate compression force of the extruded starch foams have been presented. The linear correlation coefficient values were relatively high and ranged from r = 0.963 for correlation



**Figure 2.** Typical curves of compression tests for samples extruded at 130 rpm: (a) control foam; (b) foam with 3% PDE addition; (c) foam with 3% PVA addition.

between the apparent density and the Young modulus and r = 0.973 for correlation between the apparent density and the ultimate compression force were calculated.

In this study, selected physical properties were characterized on different foams, and the effect of the functional additive and extruder screw speed on the properties of starch-based



Figure 3. The correlation between apparent densities and measured structural properties - Young modulus of extruded starch foams: ♦ control foams extruded without functional additive; + foams extruded with poly(vinyl alcohol) PVA; ▲ foams extruded with Plastronfoam PDE. In each graph the determination coefficient of linear regression R<sup>2</sup> and Pearson's r coefficient of correlation are presented.



**Figure 4.** The correlation between apparent densities and ultimate compression force of extruded starch foams:  $\blacklozenge$  control foams extruded without functional additive; + foams extruded with poly(vinyl alcohol) PVA;  $\blacktriangle$  foams extruded with Plastronfoam PDE. In each graph the determination coefficient of linear regression R2 and Pearson's r coefficient of correlation are presented.

loose-fill cushioning foams was studied and evaluated. The observed decrease of the apparent density of starch foams with an increasing screw speed is in accord with previously published results<sup>[47,48]</sup>. The higher screw speeds during the cooking extrusion increased the shearing forces inside the barrel and thus affected on more intensive thermomechanical treatment resulting better expansion and lower density of final products<sup>[9,10]</sup>.

The structure of the matter in the cell walls of extruded starch products depends on ingredients and process conditions and results in different wall properties, which partially affects the global mechanical characterization of the product<sup>[48]</sup>. In our study, the high correlation between the apparent density and mechanical traits was observed. For all the tested foams an increase of the ultimate compression force and the Young modulus was noticed with an increasing apparent density, showing that the mechanical properties of foams depend on blends composition used. For extruded starch products, a power relation between mechanical strength and structural traits is generally found<sup>[49]</sup>. Ashby<sup>[50]</sup> proposed a power law model for nonfood cellular solids, in which the exponent n gave an indication of the type of cavities. In compression, the power index equal to 2 means that the cells are open, while it is equal to 3 when the cells are closed. However, in our study, since the linear relation was found, the power index was equal to 1.

These results are in accordance with other studies, where linear relationships between the modulus of deformability<sup>[51]</sup> or crushing strength<sup>[52]</sup> and structural parameters were found. It is assumed that this type of relation is true of extrudates for which the basis formulae of blends are rather similar<sup>[51]</sup>. Furthermore, this clearly shows the specific tendencies in the mechanical properties of extrudates, according to the ingredients and process conditions.

# 4. Conclusions

Several key properties relevant to protective loose-fill cushioning foams were identified and experimentally determined. They include density, porosity, strength properties and the compression modulus. The densities of the starch foams depended significantly on the functional additive type and content. In general, foams made with PDE as the functional additive had better performance in term of stiffness than PVA foams, since PDE foams had low density, porosity and good shock absorbance (lower values of the Young modulus). The structural parameters of foams, as well as their mechanical strength, were significantly different between foams produced at different extruder screw speed. Mechanical properties showed a mediocre correlation with the Ashby model for solid foams, as the linear relationship between the Young modulus and the ultimate compression force and structural parameters was found.

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Received: Mar. 16, 2017 Revised: July 28, 2017 Accepted: July 31, 2017