

J. JAMROZ<sup>1</sup>, M. HAJNOS<sup>2</sup>, Z. SOKOŁOWSKA<sup>2</sup>

## NEW POSSIBILITIES IN THE STARCH EXTRUDATES INVESTIGATIONS

### Abstract

Potato flour extrudates were obtained by twin screw extruder. The effects of process variables were related to the following extrudate features: expansion, density, and shearing stress. The microstructure of the extruded products was examined by mercury porosimeter. Total porosity was related to physical properties of the extrudates. Total porosity decreased when expansion of products increased. Feed moisture was the principal determinate of physical strength and affected the changes of number, size and distribution of the pores.

### Introduction

It is well known that characteristics of extrudates such as texture, structure, expansion, and sensory properties are affected by many variables in the extrusion processes.

Owusu-Ansah et al, [13] revealed the textural and microstructural changes in corn starch as a function of specific extrusion variables.

They found out that such variables as feed moisture and screw speed significantly influenced breaking strength, as measured by Warner-Bratzler shear. Porosities of the extrudates increased with decreased feed moisture, parallel to an increase in expansion and a decrease in breaking strength. Texture and sensory properties of extruded products are related to the degree of their expansion [5, 8, 16].

According to Park [15], the main factor causing puffing of extrudates is the vaporization of heated water during extrusion-cooking. When gelatinized, molten starch is forced out of die. When the extrudate leaves the high pressure barrel the pressure drops rapidly, and water evaporates. Simultaneously, many small air bubbles form inside the extruded material, rendering its puffy structure. The dough cools down as it

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<sup>1</sup> Department of Biological Elements of Food Technology and Feed, Agriculture University, Doświadczalna Str. 48, 20-236 Lublin

<sup>2</sup> Institute of Agrophysics, Polish Academy of Science, Doświadczalna Str. 4, 20-236 Lublin, Poland

looses the latent heat with steam and heat transfer with the air. In the course of cooling the material becomes firmer, less plastic and solidifies.

On further cooling bubbles collapse and shrinkage of the puffed, sponge-like, structure takes place. The product loses its plasticity and elasticity. Extrudates of various expansion ratio differed in the size, number and distribution of the air pockets [6, 11].

Scanning electron microscope and appearance examination have been used to examine the microstructure of extrudates [2, 6, 18]. However, the quantitative characteristics of various pores of extrudates is difficult to perform by common laboratory methods. The study of internal porosity of biological material by mercury porosimeter is known [7, 17].

In this study the mercury porosimeter was used for examination of the microstructure of extrudates. Some relations between physical properties of potato flour extrudates and their microstructure were found.

## Materials and methods

### *Samples*

The samples of commercial potato starch were used (PN-93/A-74710). Approximate analysis gave 0.26 % of ash, 0.02 % of protein (N·6,25), 0.03 % of fat and 19.5 % of moisture.

### *Extrusion*

Extrusion was carried out in a Polish S 9/5 industrial twin screw extruder, described by Mościcki [12]. Barrel temperature was differentiated over the length of the barrel from feed to die as follows:

sample 1 – 80-120-150-170-100, sample 2 – 80-110-140-160-100°C.

Feed moisture content was adjusted to 10.5 %, or 19.5 % (d.b.). The moisture 10.5 %, (d.b.) was achieved by air-drying of native starch.

### *Expansion*

The expansion ratio was defined as the ratio of the diameter of the extrudate and the diameter of the die (expansion ratio = diameter of product/diameter of the opening). An average of 10 determinations was obtained.

The diameters of air-dried extrudates of each sample were measured with vernier caliper to the nearest 0.05 mm.

*Density*

Ten measurements of the extrudate stick per extrusion duplicate were carried out. Density of extrudate in  $\text{kg/m}^3$  was available from Eq. (1);

$$\zeta = \frac{m}{\pi r^2 l} \quad (1)$$

where:  $r$  - radius,  $l$  - length,  $m$  - weight

*Shearing stress*

A shear test was run with Instron type 4302 to determine the texture of starch extrudates. The shear stress ( $\text{N/cm}^2$ ) was calculated by dividing shear by the cross-sectional area of extrudate. Averages of 10 readings were taken.

*Mercury porosimetry*

Estimation of the porosity with the mercury porosimetry was based on the assumption, than the liquid which does not wet the surface, cannot penetrate the pores of the surface. It is likely softly after applying a pressure. At relatively low pressure, big pores are filled, and at higher pressure – smaller pores are available for the liquid. The external pressure is a function of the size of the pores. The size of the pores is related to the pressure, assuming the cylindrical pore model (the Washburn equation (2) [19]):

$$P = -2\delta\cos\theta/r \quad (2)$$

where:  $P$  – external pressure,  $r$  – cylinder radius,  $\delta$  – mercury surface tension,  $\Theta$  – wetting angle in the system : solid surface – mercury.

The radius derived from that equation, so-called equivalent radius, corresponds to the radius of the ideal capillary, because pores in natural materials have irregular shape.

The Olsen scheme can be useful for porosimetric measurements of the volume of pores and solid body:

Solid body particles	Pores inaccessible to mercury at max. porosimeter pressure	Pores filled with mercury during porosimetric analysis	Pores filled with mercury at initial pressure
$m/\zeta$	$V_b$	$V_{\max}$	$V_\alpha$
$V_o$			

The Olson equation (3), allows to measure the volume of pores beyond the range available for the mercury porosimetry:

$$V_b = V_o - V_{\max} - m/\zeta \quad (3)$$

where:  $V_b$  – pore volume below the range,  $V_o$  – volume not taken by mercury, at the initial pressure of the porosimeter,  $V_{max}$  – volume of mercury forced at the maximum pressure,  $m$  – sample weight,  $\zeta$  – sample density measured pycnometrically.

The Carlo Erba 2000 Hg intrusion porosimeter, compiled with the Carlo Erba CUT/HEC 960 computer (Carlo Erba Strumentazione, Rodano, Italy), was used in the determination of the pore radius distribution.

One gram of < 0.2 mm air-dried extrudate or whole samples were used; out-gassing for 24 h was performed before measurements.

To find a link between intrusion pressure and pore radius a cylindrical shape of pores was assumed in the calculations, and a surface tension value for Hg of 0.48 N/m extrudate contact angle of  $141^\circ$  were used in the Laplace equation. The pressure varied from 100 kPa to 200 MPa, and corresponded to a pore radius range from 3.6 nm to 7.5  $\mu\text{m}$ .

## Results and discussion

Internal porosity (TP) of extrudates evaluated by the mercury porosimeter

Not too much attention was paid to the analysis of the micropores, which appear in air cell walls. Their sizes ranged from tenth parts of a microne to several micrones. Their size and distribution depended on the extrusion variables and feed composition. Increased fiber content in corn feed increased the number of apertures in cell walls and reduced big air cells. Thus, the expansion ratio reduced with the fiber addition [9, 11].

In corn meal extrudates with 10-30 % of wheat bran addition, in air cell walls an abundance of spherical particles was observed [11]. According to Abdel-Aal E.-S.M. et al., after coarse grinding the extrudates turned into micro-flakes distinctly different in size, thickness, and appearance [1].

Table 1

Effect of extrusion variables on physical and textural properties of potato starch products

Sample	Barrel temp. [°C]	Feed moisture [%]	Expansion ratio	Density [kg/m <sup>3</sup> ]	Shearing stress [N/cm <sup>2</sup> ]	Total porosity [%]	
						N*	N**
1	80–170	10.50	6.10	51.94	4.03	14.42	2.87
2	80–160	19.50	1.86	365.14	301.93	4.30	3.29

N\* – natural extrudate,

M\*\* – ground extrudate.

Microstructure of natural and ground potato extrudates was analysed with the mercury porosimeter. The extrudate porosities (TP), calculated under assumption of cylindrical pore model, depended on the expansion ratio. That dependence was more straightforward for the natural extrudate samples (Tab. 1).

Grinding of the samples clearly reduced the porosity and the TP distribution making it practically independent of the expansion ratio. The TP of the natural samples ranged from about 4 % to 15 %, and these of the ground samples ranged from about 2.5 % to 3.5 %.

The link between the amount of pores and their radius is illustrated by the cumulation curve (integral) (Fig. 1) and pore size distribution curve (PSD) (Fig. 2). Fig. 2 shows the PSD curves for selected ground extrudate samples (1M and 2M). Their character is similar indicating a low porosity of the material despite of the expansion ratio. The PSD of the natural extrudate (Fig. 2; 1K and 2K) samples shows a broad

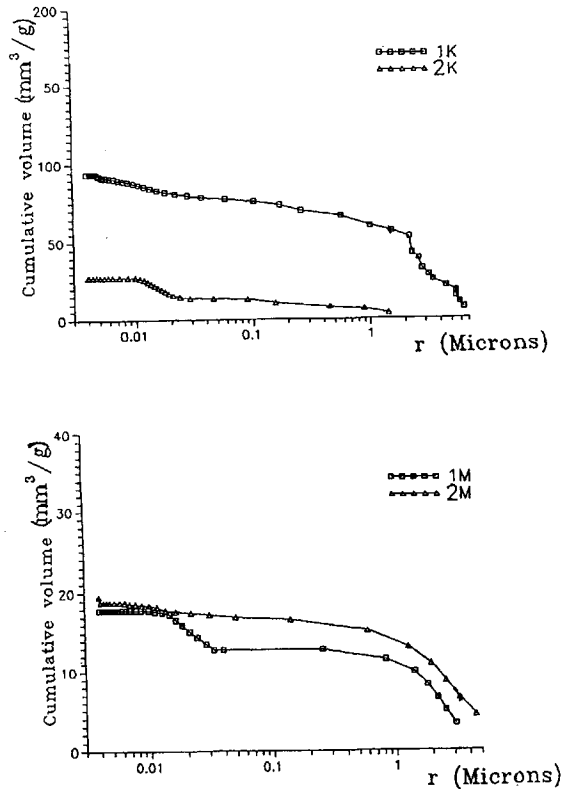


Fig. 1. Cumulative pore size distribution:  
for sample 1 (expansion ratio = 6.10),  
for sample 2 (expansion ratio = 1.86),  
K – natural extrudate, M – ground extrudate.

distribution indicating their porous structure. In samples 1, a small number of pores with the radius ranging from  $0.01 \mu\text{m}$  to  $5.5 \times 10^{-2} \mu\text{m}$  was observed, and this range, in sample 2, was wider (Fig. 1 sample 1K and Fig. 2 sample 2K).

The grinding of the highly expanded product might cause pore vanishing of the range below  $1 \mu\text{m}$ .

In the sample of high density (Fig. 2, sample 2) pores below  $0.01 \mu\text{m}$  were observed, which to a certain degree, could constitute the reflection of the micropores in the native material.

The extrusion conditions might influence the microporosity changes in the range

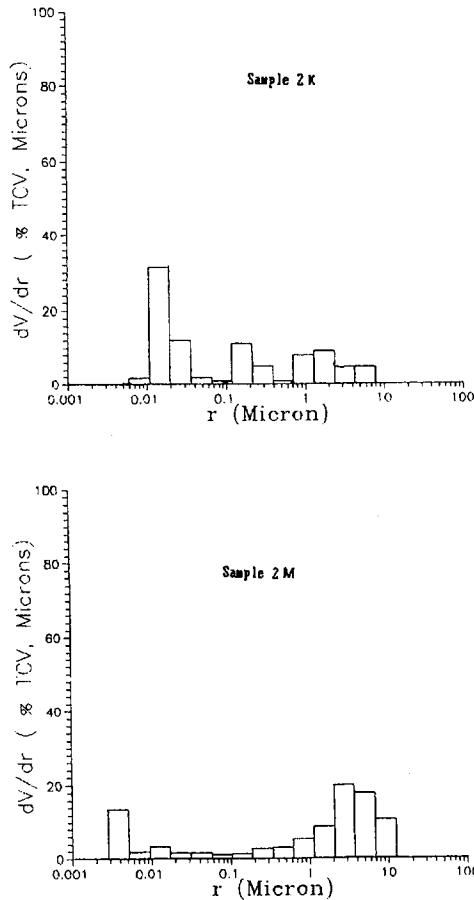


Fig. 2. Pore size distribution:  
 for sample 1 (expansion ratio = 6.10),  
 for sample 2 (expansion ratio = 1.86),  
 K – natural extrudate, M – ground extrudate.

from 3.6 nm to 7.5  $\mu\text{m}$ . Relatively high TP values were noted in the samples of natural extrudates (14.42 %) (Fig. 1, sample 1K), and in the most dense sample (Fig. 2 sample 2K and sample 2M), the TP (4.30 %) was close to that of the ground sample (3.29 %). In the sample with high expansion (1), usually containing big air pockets, relatively the lowest internal porosities were observed, which could be due to the smooth surface of the puffed extrudates.

In the analysed sample, higher content of pores with the radius of over 1  $\mu\text{m}$ , as well as small content of pores with the radius of  $5.5 \times 10^{-3} \mu\text{m}$  to  $5.5 \times 10^{-1} \mu\text{m}$  were observed.

The samples with decreasing expansion ratio, contained the highest number of pores with the radius below 1  $\mu\text{m}$  up to 0.1  $\mu\text{m}$ .

## Conclusion

The microstructure of the extrudate is reflected in both the expansion and the physical strength of products. The shear stress of the extruded starch products were inversely proportional to the expansion volume.

The total porosity, pore size distribution and cumulative curve were significantly lower for the ground extrudates as compared with the natural material.

Highly expanded samples showed lower internal porosity. One could conclude that after the radial expansion, the cell walls remained smoother. In extrudates of increasing density, greater number of micropores was noted.

Extrudates were made in Department of Food Engineering of the Agriculture University in Lublin, Poland.

## REFERENCES

- [1] Abdel-Aal E.-S.M., Sosulski F.W., Adel A., Shehata Y., Youssef M.M., Ibaev J.L.: *Lebensm.-Wiss. Technol.*, **25**, 1992, 21-25.
- [2] Barrett A.M., Peleg M.: *J. Food Sci.*, **57**, 1992, 146-148, 154.
- [3] Chiang B.-Y., Johnson J.A.: *Cereal Chem.*, **54**, 1977, 436-443.
- [4] Chinnaswamy R., Hanna M.A.: *J. Food Sci.*, **53**, 1988, 834-836, 840.
- [5] Chinnaswamy R., Hanna M.A., Zobel H.F.: *Cereal Foods World*, **34**, 1989, 415-422.
- [6] Cohen S.H., Voyle C.A.: *Food Microstructure*, **6**, 1987, 209-211.
- [7] Grundas S., Hnilica P.: *Zesz. Probl. Post. Nauk Roln.*, **304**, 1985, 113-121.
- [8] Guy R.C.E., Horne A.W.: *Food Structures – Its Creation and Evaluation*. M.V. Blanshard and J.R. Mitchell, eds. Butterworths: London. 1988, pp. 331-349.
- [9] Jin Z., Hsieh F., Huff H.E.: *Cereal Chem.*, **71**, 1994, 227-234.
- [10] Liang M., Hsieh F., Huff H.E., HU L.: *J. Food Sci.*, **59**, 1994, 890-894.
- [11] Lue S., Hsieh F., Peng I.C., Huff H.E.: *Lebensm. -Wiss. Technol.*, **23**, 1990, 165-173.
- [12] Mościcki L.: *Post. Techniki Przetw. Rolno Spozyw.*, **1**, 1994, 46

- [13] Owusu-Ansah J., van de Voort F.R., Stanley D.W.: *Can. Inst. Food Sci. Technol. J.*, **17**, 1984, 65-70.
- [14] Padmanabhan M., Bhattacharya M.: *Cereal Foods World*, **34**, 1989, 945-951.
- [15] Park K.H.: Ph.D. Thesis, University of Illinois Urbana-Champaign, Urbana, IL., 1976.
- [16] Stanley D.W.: *Food Technol.*, **40**, 1986, 65-68, 76.
- [17] Szot B., Stawiński J., Grundas S.: 4 Tagung, Agrophysik. Rostock. 1987, 247-251.
- [18] Tan J., Gao X., Hsieh F.: *J. Food Sci.*, **59**, 1994, 1247-1250.
- [19] Washburn E.W.: *Nat. Acad. Sci. Proc.*, **7**, 1921, 115-116.

## NOWE MOŻLIWOŚCI BADANIA EKSTRUDATÓW SKROBIOWYCH

### Streszczenie

Ekstrudaty ziemniaczane wyprodukowano w dwuślimakowym ekstruderze. Wpływ zmiennych procesu określono w stosunku do takich cech ekstrudatów jak: ekspansja, gęstość i siły ścinania. Siły ścinania i gęstość ekstrudowanych produktów skrobiowych były odwrotnie proporcjonalne do wartości ekspansji. Mikrostrukturę produktów ekstrudowanych badano w porozymetrze rtęciowym. Ogólna porowatość (porowatość wewnętrzna) była określona w relacji do fizycznych właściwości ekstrudatów. Ogólna porowatość zmniejszała się kiedy wzrastała ekspansja produktów. Wilgotność wsadu była szczególnym determinantem fizycznej wytrzymałości i powodowała zmiany liczby, wymiarów i rozmieszczenia porów.

Rozmieszczenie wymiarów porów naturalnych próbek ekstrudatów pokazywało zróżnicowany charakter i wyróżniało ich porowatą strukturę. Względnie wysokie wartości porowatości wewnętrznej odnotowano w próbkach ekstrudatów naturalnych (14.42 %), a w próbkach o największej gęstości, porowatość wewnętrzna (4.30 %) była zbliżona do wartości próbek zmielonych (3.29 %). ☒