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APPLICATION OF THE MERCURY POROSIMETRY TO THE POROSITY STUDY OF WHEAT FLOUR EXTRUDATES

J. Jamroz[1], *M. Hajnos*[2], *Z. Sokołowska*[2]

[1]Department of Biological Elements of Food Technology and Feed, University of Agriculture, Doświadczalna 48
20-236 Lublin, Poland
[2]Institute of Agrophysics, Polish Academy of Sciences, Doświadczalna 4, 20-290 Lublin 27, Poland

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Abstract. Wheat flour products manufactured with a single and twin screw extruder at different extrusion-cooking variables were studied. The effects of extrusion-cooking conditions were related to the following extrudate features: expansion, density and shearing stress. The microstructure of the extruded products was examined by mercury porosimetry in order to determine whether their physical properties were related to the presence of pores. The number, sizes and distribution of pores resulted from structure changes in the extruded wheat flour.

Keywords: wheat flour, extrudates, internal porosity, mercury porosimetry

INTRODUCTION

Extrusion-cooking is an important technique for the modification of starch and protein-based flours to produce a wide variety of traditional and modern food products. Characteristics of these products, e.g., structure, expansion ratio, and sensory properties are affected by many variables of the extrusion-cooking process. Numerous research works on the effects of moisture content and extruder barrel temperature profile on the expansion properties of starch extrudates from single or twin screw extruder-cooker have been reported [2,3,5,12]. According to Barrett and Peleg [1], the texture of puffed extrudates is primarily determined by their cellular structure and the mechanical properties of the solid matrix. Evaluation of density or shearing stress may inform about the changes of product s structure. However, the extrudates with the same density could have different combinations of cell size wall thickness. According to Owusu *et al.* [12] the microstructure of the extrudate is reflected in both the expansion and the physical strength of the extrudate, with highly expanded samples more porous and showing numerous peaks before breakage. Conditions of the extrusion-cooking affect morphology of the extrudate big structures (air pockets) of about 50 to several thousand microns in size [10], as well as small structures in air cell walls in the range of about 5 m and less [4].

So far not much attention has been paid to the analysis of the microporous structure of extrudate cell walls. Distribution and size of pores depend on extrusion-cooking variables and chemical composition of the feed. Guy and Horne [6] reported that fibre supplement in corn feed materials results in the size decrease of air pockets as the fibre, which prevented full expansion, ruptured the walls. The most important processing variable was feed moisture [14]. Pore size and thickness of the walls were related to moisture content [7]. These findings were based on SEM data.

The internal microstructure of the extruded products was usually examined under a scanning electron microscope. The micrographs showed air pockets and their wall surface. Recently, in the study of internal structure of extrudate, a new technique - Small angle X-ray scattering (SAXS) was employed. This method demonstrated numerous electronic inhomogeneities in the density formed in the extrudates after extrusion-cooking process [9]. Additionally, mercury porosimetry method applied to the study of internal porosity in the cell wall of potato extrudates was used [8].

The objective of our work was to investigate internal porosity of wheat flour extrudates in relation to some extrudates features.

MATERIALS AND METHODS

Samples - Commercial wheat flour [13] was used. It contained 0.46% of ash, 12.01% of protein (N x 6.25), 1.82% of lipids, 79.89% of starch, 1.93% of fibre and 14.50% of moisture [13].

Extrusion-cooking process - Extrusion -cooking was accomplished using a Polish S 45 and S 9/5 industrial single- and twin-screw cookers with the specifications described by Mościcki [11]. Prior to extrusion-cooking, portions of wheat flour were equilibrated overnight to five moisture levels (db): 14.20, 11.20, 13.40, 11.80 and 14.50% (Table 1).

Expansion - Expansion ratio was defined as the ratio of the extrudate and die diameters. Each mean was an average of ten estimations. The diameters of the air-dried extrudates were determined with a vernier caliper with 0.05 mm precision.

Density- Density (ς) was derived using the formula:

$$\varsigma = w(\pi r^2 l)^{-1}$$

with r, l and w the stick radius, length and weight, respectively. Ten estimations (kg m^{-3}) were made on the extrudate sticks available from the duplicated cooking.

Shearing stress - Shearing stress (N cm^{-2}) was calculated as the extrudate shear force to the cross-section area ratio. Instron 4302 instrument was used for the starch extrudate (6-6.5%, of moisture, dry basis) texture determinations (in ten replicates).

Mercury porosimetry- A Carlo Erba 2000 Hg intrusion porosimeter, linked to a Carlo Erba CUT/HEC 960 computer (Carlo Erba Strumentazione, Rodano, Italy), was used to determine the distribution of pore radii. The extrudates were ground on a laboratory coffee mill to pass through a 0.2 mm sieve. One gram of air-dried extrudate was used; outgassing for 24 h was performed before the measurements.

To find correspondence between intrusion pressure and pore radius values, in the calculations we assumed that pores were cylindrical, and a surface tension value for Hg of 0.48 N m^{-1} extrudate contact angle of 141° was used in the Laplace equation. The pressure varied from 100 kPa to 200 MPa, and corresponded to the pore radius range of 3.6 nm to 7.5 μm.

RESULTS AND DISCUSION

Table 1 presents the characteristics of extrusion-cooked wheat flour. The final product is shown in Fig. 1. Differences in the properties of the extrudates could be related to feed moisture, temperature profile in the extruder

Table 1. Characteristics of wheat flour feed and extrudates

Sample*	Feed moisture (%)	Barrel temperature profile (°C)	Die opening (mm)	Density (kg m^{-3})	Expansion ratio	Shearing stress (N $\pi^{-1} r^{-2}$)
P1	14.20	80-120-180-200-100	6	365.30	2.39	48.47
P2	11.20	145-165-120	4	145.86	3.55	13.26
P3	13.40	100-140-180-200-100	6	206.61	3.27	14.04
P4	14.50	80-150-190-210-140	3.5	147.24	4.20	15.32
P5	11.80	140-160-120	5	150.36	3.10	12.12
P6	14.50	80-150-190-210-140	3.5	148.88	4.11	16.90
Control	14.50					

*Samples: P1-P6, extrudates after applying various extrusion-cooking variables; P2, P5 - using single- and twin-screw; P1, P3, P4, P6 - extrusion-cookers; P0 - control - raw wheat flour.

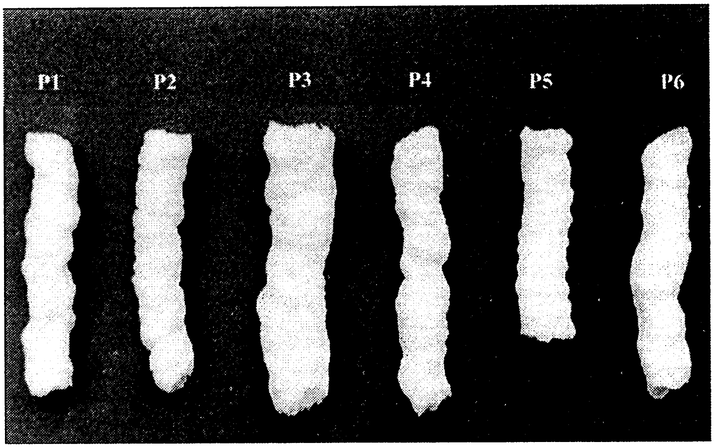

Fig. 1. Physical appearance of wheat extrudates: P1-P6-samples cooked with different cooker variables. See Table 1 for notes.

barrel, die diameter, feeding rate, and obviously, extruder type.

Extrusion processes affect the structure of the examined samples. Figures 2a and 2b show cumulative curves (TCV) and Fig. 3 pore size distribution (PSD) obtained for the examined materials by means of mercury porosimetry. Curves (means for 3 experiments) are given as dV/dr where V is a percentage share of the pores of a given size in a total pore volume obtained by the porosimetric measurement, and r is an equivalent radius of these pores. Such a presentation of results allows to compare distribution of pores for the samples with considerably diverse total volume of pores.

Parallely to the changes of the cumulative volume (Fig. 2a), values of total porosity (TP) either increase or decrease. For wheat flour (raw material) the highest values of TCV was 170.19 $mm^3 g^{-1}$ and of TP was 18.20%. Extrudates were characterised by lower values of TCV and TP. In the P1 extrudate obtained at 14.20% feed moisture and die diameter of 6 mm, the lowest expansion ratio related to the highest density and shearing stress was observed. In this extrudate, the values of TCV and TP were close to those of the feed materials. Supposedly, the feed material moisture had an important effect on the changes of product porosity. From the analysis of the sample 3 structure (die diameter 6 mm) at feed moisture lower by 5.6%, the values of TCV and TP lower by about 50% (as compared to extrudate P1) were obtained.

Faubion and Hoseney [5] showed that by decreasing feed moisture, expansion and textural strength of wheat flour extrudates increased.

Apparently, smoothing of the cell walls (sample 3) was related to the increase of the expansion ratio (3.27). In our previous study [9], internal porosity of potato starch extrudates was related to expansion ratio. The largest air cells with the smallest pore sizes were obtained at the biggest expansion ratio. Apart from feed moisture, die diameter of extruder can affect the structure of the product, evaluated by TCV and TP. Extrudates P2 and P5 were obtained by a single screw extruder. The TCV value of sample 2 (∅=4 mm, die diameter) was about two times higher than that of the sample 5 (∅=5, die diameter). Rate of feed (sample 4-120 kg h^{-1}; sample 6-180 kg h^{-1}) can affect changes in the product structure. Sample 4, fed at 120 kg h^{-1}, was characterised by about 31.84 % higher TCV and about 27.96% higher TP comparing to the respective values for sample 6.

Different structures of samples P4 and P6 are well illustrated by cumulative curves (Fig. 2b). There are two distinct ranges of pores in both samples, which is the reason for different character of the curves which final parts visibly rise. From the analysis of the distribution curve

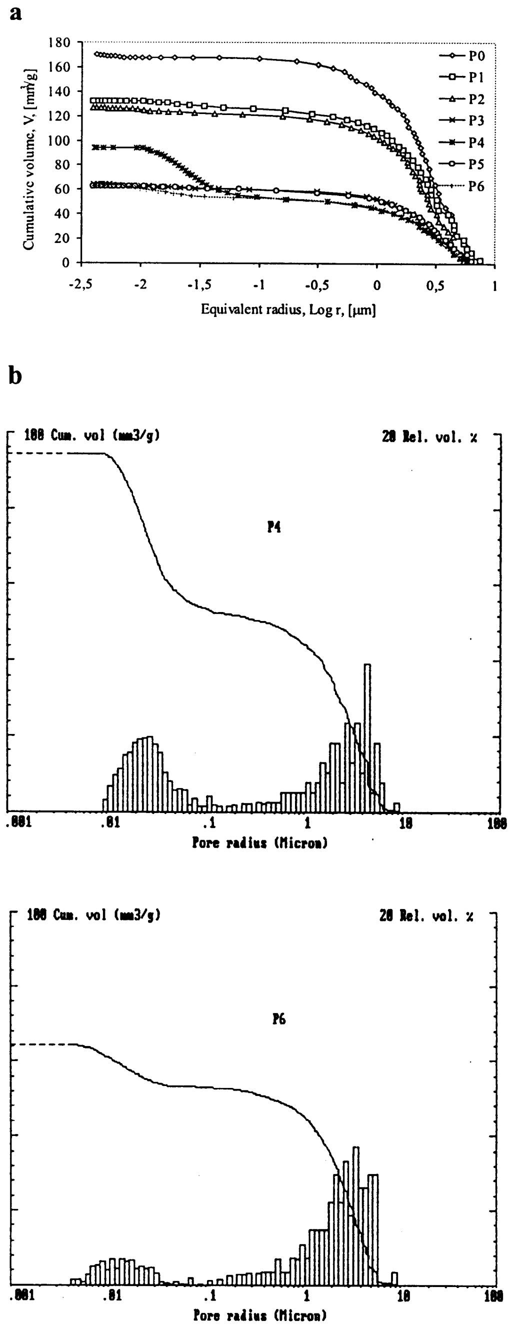

Fig. 2. Cumulative curves for wheat flour extrudates: a) samples P1-P6 and for native material (P0) and b) samples P4 and P6.

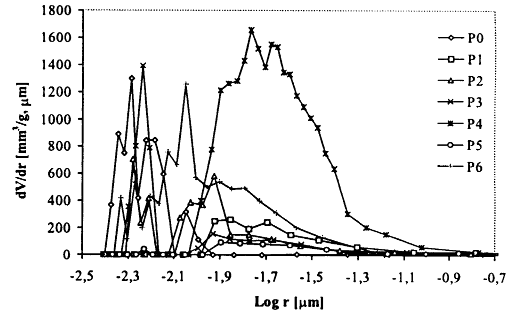

Fig. 3. Pore size distribution for wheat flour extrudates and for native material.

(PSD) data the range of the radius and the respective range of porosity may be assessed. In the extrudate P4 about 50% of the total pore volume was noted in the range of $1 \cdot 10^{-2}$-$7 \cdot 10^{-2}$ μm whereas in the P6 sample 15% of the total pore volume was noted in the range of $5 \cdot 10^{-3}$-$5 \cdot 10^{-2}$μm. In other samples small volume of small pores was noted in the range of $1 \cdot 10^{-2}$-$6 \cdot 10^{-1}$ μm radii. In the samples PS, P2, P3 only negligible porosity was stated in the range $5 \cdot 10^{-3}$-$1 \cdot 10^{-2}$μm radii. For all the samples the maximum amount of big pores was stated in the range $6 \cdot 10^{-1}$- 8 μm of radii, where a distinct rise of the cumulative curves appears. Conditions of the extrusion did not have a great effect on the changes of the average pore radius (AVR), which was about 3.81 μm. The exception was a high density extrudate P1, with an AVR of 2.49 μm. However, the extrusion-cooking process definitely resulted in lowering of TP and TCV of the product.

CONCLUSION

This study showed that the porosity of extruded wheat flour could be influenced by controlling process conditions. Thus, it should be feasible to produce extruded wheat flour with specific porosity properties by setting appropriate processing variables.

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