ANETA TOR-ŚWIĄTEK
Lublin University of Technology
Department of Polymer Processing
ul. Nadbystrzycka 36, 20-618 Lublin
e-mail: a.tor@pollub.pl

Characteristics of physical structure of poly(vinyl chloride) extrudate modified with microspheres

RAPID COMMUNICATION

Summary — Microcellular extrudates based on poly(vinyl chloride) with blowing agents in the form of microspheres added in an amount of 0.5, 1.0, or 2.5 % by weight of the polymer were prepared. Extrudates in the form of rods were studied to determine the geometric characteristics of the microporous structure. The influence of the quantity of blowing agents used on the number, size and surface fraction of micropores formed was tested. Five consecutive sections of prepared extrudate starting from the surface layer and ending with the core were studied. Significant changes in the structure of successive layers, associated with different cooling rates were observed.
Keywords: microcellular extrusion, poly(vinyl chloride), blowing agent, microspheres, physical structure.

CHARAKTERYSTYKA STRUKTURY FIZYCZNEJ WYTŁOCZYNY Z POLI(CHLORKU WINYLU) MODYFIKOWANEGO MIKROSFERAMI

Streszczenie — W ramach tej pracy wykonano mikroporowate wytłoczyny na bazie poli(chlorku winylu) z dodatkiem środka porującego w postaci mikrosfer dodawanego w ilości 0,5, 1,0 lub 2,5 % w stosunku do masy polimeru. Wytłoczyny w postaci prętów poddano badaniom mającym na celu określenie cech geometrycznych ich mikroporowatej struktury. Stwierdzono wyraźny wpływ użytej ilości środka porującego na liczbę, rozmiary oraz udział powierzchniowy powstałych mikroporów. Przebadano po pięć kolejnych przekrojów przygotowanych wytłoczyn zaczynając od warstwy powierzchniowej, a kończąc na rdzeniu. Zaobserwowano znaczne zmiany struktury kolejnych warstw, co jest związane z różną szybkością ich chłodzenia.
Słowa kluczowe: wytłaczanie mikroporujące, poli(chlorek winylu), środek porujący, mikrosfery, struktura fizyczna.

INTRODUCTION

Microcellular extrusion process is in recent years one of faster developing polymer processing methods. Results of the process are products of different shapes like pipes, bars, cables and conduit coatings, with specific new physical and functional properties resulting from modification of the structures [1—4]. The essence of the process is to insert a suitable proportion of a blowing agent to obtain homogeneous microcellular structure without altering the properties [1, 5]. Blowing agent is inserted into polymer material during the extrusion process. Under the influence of high temperature, the blowing agent undergoes decomposition or expansion which causes changes in the polymer material structure from solid into microcellular [6].

The final physical structure of cellular and microcellular products depends upon:

— the type of blowing agent used and its ability to decompose [7—9],

— processing conditions of the polymer used [10—12].

The elementary values that allow defining physical structure of the products with more than one component, *e.g.* microcellular products, are these which enable to determine the amount and quality of each component. The resulting physical structure has to be characterized by measuring the volume porosity, the surface porosity, the dimension of pores, the pores distribution and finally the volume fraction of pores (porosity of the system) [13, 14]. This can be done with computer image analysis methods which are known to allow qualitative and quantitative measurements of dispersed particular quantitie [15, 16].

EXPERIMENTAL

Materials

Plastified, transparent poly(vinyl chloride) (PVC) with trade name Alfavinyl GFM/4-TR was studied. PVC used has density of 1210 kg/m^3 and melt mass-flow rate *MFR* (10 kg, 160 °C) of 2.19 g/10 min.

The blowing agent (BA) was used in the form microspheres with name Expancel 930 MB 120, produced by Akzo Nobel. The diameter of microspheres was from 28 to 38 μm, with density lower than 6.5 kg/m^3 and processing temperature range 140—200 °C. Unexpanded microspheres are made of spherical polymeric capsules containing gas-liquid hydrocarbon. Under the influence of higher temperature microspheres absorb heat, what caused growth of its volume up to 50 times. BA used in this research was a blend containing 65 % of microspheres in copolymer of ethylene and vinyl acetate (EVA).

Samples preparation and testing

Samples were obtained by means of microcellular extrusion process with constant screw rotation speed 1.30 s^{-1} and using different content of BA (0.5, 1.0 or 2.5 % of the mass in relation to the mass of input plastic). The process was conducted in the following temperatures of heating zones in the plasticizing system and extruder head: 100, 110, 120, 130 and 140 °C. Extrusion products are in the shape of a rod with diameter 5 mm.

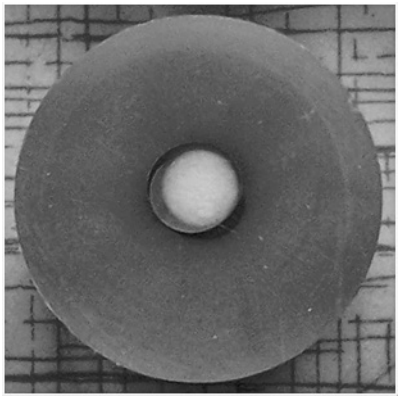

Fig. 1. View of exemplary sample in the form of microsection inserted in a chemoset acrylic resin

In order to assess physical structure of the extrudate obtained, samples in the form of microsections, as it is shown in Figure 1, were prepared. The samples were inserted into a chemoset acrylic resin with trade name Duracryl Plus, produced by SpofaDental a.s. Then samples were ground and polished for imaging analysis.

Physical structure of the extrudate was tested with computer image analysis techniques by using a confocal laser scanning microscope LEXT OLS3100. Analysis of microscopic images was carried out by computer program Pixel-Fox. Microcellular extrudate samples were examined in cross-sections in five measuring plots, starting from surface layer and next moving the measuring area towards the core, numbered from 1 to 5, respectively. Area of each measuring plot was 0.292 mm^2.

RESULTS AND DISCUSSION

Figure 2 presents images of the microcellular structure of samples with various contents of BA in the form of microspheres. Based on the analysis of obtained images it can be stated, that new microcellular structure of the extrudate is not homogeneous in the whole section. There is greater concentration of micropores formed in the core of the extrudate. Faster cooling of the extrudate prevents expansion of microspheres in the skin layer. Changed

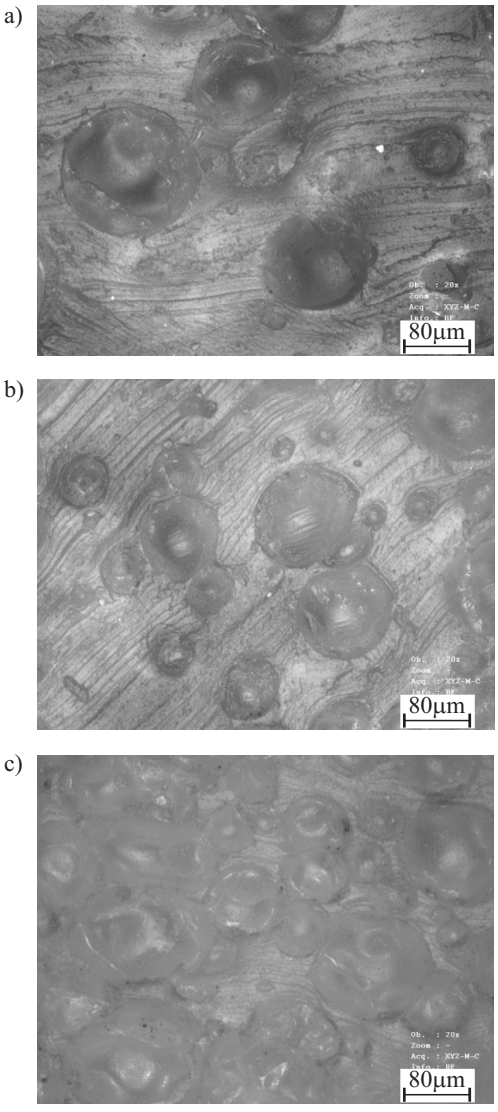

Fig. 2. View of physical structure of obtained microcellular extrudate with content (in relation to PVC mass) of BA in the form of microspheres: a) 0.5 %, b) 1.0 %, c) 2.5 %

content of microspheres has a significant influence on size, distribution and amount of expanded micropores. The diameter of micropores in the skin layer decreased from 70.18 μm (for 0.5 % of BA) and 69.48 μm (for 1.0 % of BA) to 47.95 μm (for 2.5 % of BA).

As a result of the study, characteristic quantities of the physical structure such as the amount, diameter, elementary surface and the surface fraction of micropores were obtained. Research results of physical structure of obtained microcellular extrudate are shown in Tables 1—3.

T a b l e 1. Results of investigation of physical structure of microcellular extrudate with 0.5 % content of BA

Number of measuring cross-section	Amount of micro-pores	Diameter of micro-pores, μm	Elementary surface of micropores μm^2	Surface fraction of micropores %
1	6	70.18	9 313.98	15.65
2	10	76.30	6 894.70	25.10
3	13	89.62	8 262.66	27.08
4	14	96.40	11 269.50	44.31
5	15	114.10	12 196.25	48.24

T a b l e 2. Results of investigation of physical structure of microcellular extrudate with 1.0 % content of BA

Number of measuring cross-section	Amount of micro-pores	Diameter of micro-pores, μm	Elementary surface of micropores μm^2	Surface fraction of micropores %
1	33	69.48	4 813.43	41.20
2	31	78.45	5 387.59	48.35
3	25	80.34	6 045.65	52.33
4	31	86.41	7 869.59	60.48
5	37	96.49	5 707.33	67.96

T a b l e 3. Results of investigations of physical structure of microcellular extrudate with 2.5 % content of BA

Number of measuring cross-section	Amount of micro-pores	Diameter of micro-pores, μm	Elementary surface of micropores μm^2	Surface fraction of micropores %
1	34	47.95	2 452.84	56.60
2	35	55.62	3 168.32	66.58
3	36	57.03	3 471.20	80.81
4	41	57.10	3 026.98	94.44
5	49	57.40	2 763.27	98.40

Increasing content of BA in PVC from 0.5 to 2.5 % caused decrease in size of micropores by about 30—50 % depending on cross-section. Simultaneously in consecutive measuring layers of the samples one observes a gradual increase in micropores diameter ranging from about 48—70 μm in the surface layer (number 1) to 57—114 μm in the core of the extrudate (number 5). The same applies to elementary surface of micropores. Its intensive decrease is observed with 2.5 % content of BA. Growth of this quantity is greatest at the lowest dosage of BA in amount of 0.5 %. However the surface fraction of micropores in the whole range of microspheres proportion is lowest in the surface layer (number 1) of the extrudate and it ranges from 16 % at 0.5 % of BA to 56 % at 2.5 % of BA. While approaching the core of the extrudate increasing surface fraction of micropores is observed. In measuring cross-section in the core of the extrudate (number 5) this fraction ranges appropriately from 48 % for sample with 0.5 % of BA to 98 % for sample with 2.5 % of BA.

Similar dependence holds for the amount of micropores in particular measuring cross-sections. The amount of formed micropores in extrusion products in particular measuring layers increases with increasing content of BA in PVC. The amount of micropores increases also with the change of measuring cross-sections in the direction of extrudate core, however this increase is gradual. The largest increase in the amount of micropores occurs in each measuring area during the change of BA dosage from 0.5 to 1.0 % and it ranges from 450 % in the skin layer to 147 % in the core. This may be due to the cooling intensity of the extrudate, which prevents formation and growth of microspheres in the skin layer.

CONCLUSIONS

The analysis of results showed that both the diameter of the micropores formed, the elementary surface, the surface fraction and amount of micropores varies depending not only on the content of blowing agent (microspheres) in the material, but also on the measuring cross sections. All tested values characterizing the physical structure of microporous extrudate had lower values on the surface layer of the extrudate. These values increased while shifting the measurement cross-section towards the core of the extrudate. This demonstrates the significant impact of extrudate cooling intensity on the test quantities. Contact between the surface layer of extrudate during the extrusion and a cooling factor (water), caused the formation of micropores and the growth just created pores.

Modifying PVC with BA in sufficient quantity is essential to the effective conduct of the extrusion process and the properties and structure of products.

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