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EVALUATION OF THE EFFECTIVENESS OF THE MICROCELLULAR EXTRUSION PROCESS OF LOW DENSITY POLYETHYLENE

OCENA EFEKTYWNOŚCI PROCESU WYTŁACZANIA MIKROPORUJĄCEGO POLIETYLENU MAŁEJ GĘSTOŚCI*

From a processing point of view, evaluation of effectiveness is essential for the proper conduct of each polymer processing process. However, it is particularly important in the case of modified plastics processing aids, such as blowing agents, the addition of which causes changes in both the process course and the physical properties and structure of the resulting product. Carrying out a proper analysis of the efficiency of the process, taking into account the criteria and methods can efficiently and reliably carry out a extrusion process. This paper presents the results of assessing the effectiveness of the microcellular extrusion process of LDPE modified with selected microblowing agent in the form of microspheres.

Keywords: *microcellular extrusion process, effectiveness of process, low density polyethylene, blowing agent, microspheres.*

Z przetwórczego punktu widzenia ocena efektywności ma istotne znaczenie dla właściwego prowadzenia każdego procesu przetwórstwa tworzyw polimerowych. Jednak szczególnie ważna jest ona w przypadku przetwórstwa tworzyw modyfikowanych środkami porotwórczymi, których dodanie powoduje zmiany zarówno w przebiegu procesu, jak i we właściwościach fizycznych oraz strukturze otrzymanego wytworu. Przeprowadzenie właściwej analizy efektywności procesu, uwzględniającej odpowiednie kryteria i metody pozwala wydajnie i niezawodnie prowadzić proces wytłaczania. Artykuł prezentuje wyniki badań oceny efektywności procesu wytłaczania mikroporującego polietylenu małej gęstości modyfikowanego środkiem mikroporującym w postaci mikrosfer.

Słowa kluczowe: *wytłaczanie mikroporujące, efektywność procesu, polietylen małej gęstości, środek porujący, mikrosfery.*

1. Introduction

Microcellular extrusion process is now more frequently used method for processing of thermoplastics. The process according to the distribution characteristics of blowing agent can be carried out using standard production lines, but with regard to the proper selection of processing conditions. Consequence of the introduction of special blowing agents to plastic is [4] to obtain the products of a certain physical properties and structure.

Due to the presence of blowing agents with the characteristics of the distribution endo-or exothermic on the processed material, contained in the plasticizing system, affects variables conditions such as high temperature, high pressure, large shear stresses. This makes the microcellular extrusion process difficult to proceed [1, 2].

Considering the effectiveness of the extrusion process takes into account certain criteria and methods for assessing the effectiveness and impact of various features design particular processing machines and tools [6, 7]. Among the criteria to assess the effectiveness of the extrusion process should be distinguished quantitative, qualitative and operational criteria [13]. Quantitative criteria include, for example, the physical parameters characterizing the extrusion process, such as the degree of plasticity, volumetric flow rate of material, the mass flow rate of material (efficiency extrusion) and energy efficiency. Among the criteria of quality, replace the distribution and fluctuation of processing temperature, the longitudinal and cross degree of mixing, stability of the process efficiency (pulsation of the flow rate and pressure), structural changes and physical properties of the products. Operational criteria include durability, the availability of production and process automation and robotics.

In the plastics processing with the addition of various auxiliary agents, including blowing agents, mixing efficiency is very important. It is primarily determined by the design of the plasticizing system, in particular a screw and cylinder design [5, 8, 12].

Quality evaluation of microcellular extrusion process is very complex. Requires consideration of a large number of direct and indirect indicators describing the quality and the proper conduct of the process [9]. Direct indicators include physical quantities characterizing the plasticization of the process and added blowing agents because of the stability of the process [15]. The measurement of these values is made directly during the process or after the relevant calculations. Direct indicators include mainly factors determining the extrusion process course, the stream of input and dissipation of heat, distribution of temperature and pressure, distribution of the residence time of material in the extruder, the degree of plastic homogenization, the energy consumption and rheological properties [14]. Indirect indicators are physical quantities that describe in detail the process of plasticization and instability. These include temperature fluctuation of plastic, pulse of the pressure, thermal and mechanical homogenization of the material. Specific criteria and methods of extrusion evaluation are used depending on variety of extrusion process [3].

Microcellular extrusion efficiency is the ability of the plasticizing system, which processor the plastic modified with blowing agent, to manufacture of high-quality components with the possible greatest effectiveness and energy efficiency of the process.

(*) Tekst artykułu w polskiej wersji językowej dostępny w elektronicznym wydaniu kwartalnika na stronie ***www.ein.org.pl***

The purpose of this study is to evaluate the extrusion process on the basis of factors characterizing the effectiveness, such as pressure and temperature distribution of the plastic in the extruder plasticizing system and extrusion head, the mass flow rate of material from the extrusion head (process efficiency), the Barus effect, extruder energy efficiency and process energy efficiency of LDPE porous extruded. Great importance for the assessment of the process is extrudate quality, which is determined by the degree of mixing of polyethylene and blowing agent.

2. Working stand and characteristic of research

Microcellular extrusion process was carried out in technological line for shapes (fig. 1), equipped with single screw extruder W-25D, extrusion head with slot die for tape extrusion, calibration device, the cooling bath and collecting device. The extruder is made up of motor, reduction gear and plasticizing system. Extruder plasticizing system consist of segment cylinder assembled from three segments. The plasticizing system can be supplied with different segments, which together made up of three screw length: 18D, 23D and 28D. In the first set, used to test of the extrusion process efficiency of the porous LDPE, the cylinder has two heating zones and provided with a screw with a diameter of 25 mm and length of 450 mm. Screw drive is implemented using a DC motor with a capacity of 3.57 kW with infinitely variable number of rotations, belt transmission, gear and overload clutch. This ensures the operation of the extruder screw rotation speed varying in the range from 0 to 5.35s^{-1}. Extrusion head for tape is equipped with a heating zone with an annular electric heater, sensor of pressure and temperature of the plastic and the second temperature sensor – a thermocouple. The head has a removable flat slot die, the width of the die used in the studies is 22.00 mm, and the height of 1.40 mm.

To the studies the low density polyethylene LDPE with the trade name Malen E FABS 23 D022 produced by Basel Polyolefins was used. The plastic contains an antioxidant, reducing the degradation of material in terms of processing and slip additives and anti blocking. This material is characterized (by the manufacturer) density of 922 kg/m^3, a nominal melt flow rate MFR at 190°C and 2.16 kg equal to 2g/10 min, tensile strength of 18 MPa, hardness 48°Sh and softening point VST (50°C /10N) of 91°C.

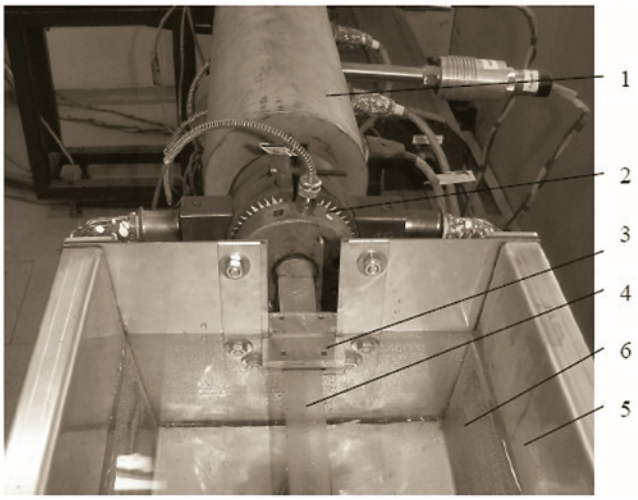


Fig. 1. Fragment of technological line of tape extrusion: 1 – plasticizing system of extruder , 2 – extrusion head, 3 – calibration device, 4 –extruded porous LDPE tape, 5 – cooling bath, 6 – cooling water

Low density polyethylene was modified with a special blowing agent in the form of polymer microspheres called Expancel 950 MB 120 manufactured by Akzo Nobel. Microspheres used are made of thermoplastic polymer capsules comprising a gas - a liquid hydrocarbon, which, under the influence of elevated temperature increases the volume of the microspheres. Polymer coat of microspheres is formed during their manufacture by polymerization of a mixture of acrylonitrile and methacrylonitrile. The most important characteristics of this agent are primarily density of 6.5 kg/m^3, size before processing from 28 to 38 μm and a processing temperature of 180 to 200°C.

3. Programme of research

In order to know the impact of the microspheres on LDPE extrusion process the test program was developed, taking into account the content of dispensed microspheres, the temperature in the extruder plasticizing system and extrusion head, the screw rotation speed, the rotational speed of the collecting device.

The process was carried out under specified processing conditions, taking into account the type of the plastic and the characteristics of the distribution of selected blowing agent. Set temperature of the zones of the extruder plasticizing system and the extrusion head, with values 130, 150, 180°C, read after process stabilization for each screw rotational speed and the content of microspheres in the base material being processed.

It was found that the quantity of the dispensed microspheres will be 0%, 0.1%, 0.3% and 0.5% by mass relative to the mass of LDPE. These values are based on data from the manufacturer of the Expancel and own preliminary tests.

During microcellular extrusion of LDPE the extruder screw rotation speed was changed and it was 50, 100 and 150 rpm. Withdrawing shaft rotational speed of collecting device amounted respectively to a 4, 8 and 15 rpm.

As part of the established research program of microcellular extrusion process effectiveness was made the measurements of real plastic temperature and pressure in the plasticizing system and extrusion head, the mass and dimensions of the extruded measuring sections, extrusion time of measuring section and time of extruder power consumption during measuring section extrusion.

After a series of calculations was determined the mass flow rate of the extrudate, which is a measure of extrusion process efficiency, the plastic enthalpy growth, flow of energy supplied to the extruder and the extrusion head, the heat flux carried by the material, the energy efficiency of extruder and microcellular extrusion process. The designation of the above figures is important for the physical changes taking place and the obtained microporous structure of extrudate.

Barusa effect studies was made in order to determine the expansion of the extrudate stream flowing from the die of extrusion head due to different normal stresses resulting from plastic flow. Measurements were performed after the cooling of extrudate at the normal temperature. The value of this effect was determined from the dependence, which is the ratio of the cross-section of extrudate to the cross-sectional area of the die, expressed as a percentage.

To properly determine the quality of the microcellular extrusion process and the degree of mixing polyethylene and microspheres the resulting extrudate microporous structure was also evaluated by performing microscopic image.

4. Research results

Studies of plastic temperature distribution (Table 1) in the extruder plasticizing system and the extrusion head has shown that the addition of microspheres to the LDPE occurring temperature differences. The highest temperature increase of about 40% was observed at the dosage of 0.1% of the microspheres irrespective of the change of extruder screw rotational speed. On further increasing the content of the microspheres in the plastic in an amount of 0.3% and 0.5% the

Table 1. Research results of plastic temperature and pressure in extruder plasticizing system (I, II) and extrusion head (III)

No.	Dosage of microspheres, %	Screw rotational speed, rpm	Temperature in plasticizing system and extrusion head, °C			Pressure in plasticizing system and extrusion head, MPa	
			I	II	III	I	III
1.	0	50	128,8	154,4	175,1	17,1	2,0
		100	129,5	154,6	175,6	10,3	2,6
		150	131,6	159,6	179,6	9,9	3,4
2.	0,1	50	142,6	186,2	181,7	16,9	2,0
		100	143,6	186,8	178,0	12,9	2,4
		150	143,8	179,4	177,9	10,9	2,8
3.	0,3	50	140,5	175,5	200,0	18,8	1,2
		100	139,8	163,1	190,0	11,5	2,4
		150	142,4	165,3	198,6	9,9	2,3
4.	0,5	50	132,1	162,5	187,0	12,4	1,1
		100	140,0	169,4	197,9	10,9	1,7
		150	141,0	169,0	199,0	7,0	2,0

material temperature is changing in relation to the set temperature, but this increase is not as intense and is about 9%.

The sharp increase of material temperature in the first zone of the plasticizing system, occurring after the addition of the microspheres can result from the use of the innovative design of the extruder, which provides high-intensity mixing process of plastic. In the process, heating of the LDPE takes place by means of electric heaters arranged on the cylinder of the extruder. However, in the second zone of the plasticizing system and after the process stabilization, reach a certain temperature and screw rotational speed about 100 rpm the autothermal extrusion process be started [11]. Then heat generation that is necessary for the of plasticization occurred as a result of external friction plastic granules themselves and cylinder working surfaces and the screw and the internal friction of the plastic macromolecules and the added blowing agent. This results in a significant increase in plastic temperature with an increase in extruder screw rotational speed.

In the case of pressure measurements (Table 1) of the plastic in the extruder plasticizing system and extrusion head, the pressure is increasing and then rapidly falls in extrusion head, which is according to the data available in the literature [14]. As a result of increasing the extruder screw rotational speed, the pressure in the plasticizing system of plastic decreases on average by 30% with the increase in screw rotation speed of 50 to 100 rpmin and a 17% at the next increase screw rotational speed of 150 rpm. Pressure of the plastic in extrusion head increases with a light intensity with increasing screw rotational speed. The plastic pressure changes in the extruder plasticizing system and extrusion head does not affect the content of the microspheres or this influence is very small.

Research of the microcellular extrusion allowed to determine the efficiency of this process and to determine occurring the Barus effect. Based on the results of measurements the dependences of the tested factors described in the test program, as a function of the screw rotational speed were done, which are shown in Figures 2÷5.

On the basis of the measurements results it can be concluded that with increasing screw rotational speed the microcellular extrusion process efficiency increases by an average of about 30%. In addition, with increasing content of microspheres in LDPE takes place a gradual slight increase of efficiency of the ongoing process. For the screw rotational speed of 50 rpm the process efficiency is increased by 3.4% for a dosage of 0.1%, by 4.7% for a dosage of 0.3% and by 1.5% for a dosage of 0.5%. At higher screw rotational speeds the process efficiency changes slightly with increasing dosage of microspheres in LDPE.

Barus effect tests have shown a slight expansion of extrudate stream flowing from the extrusion head only with the extruder screw rotational speed of 150 rpm. As a result of increasing the dosage of the microspheres in LDPE a gradual decrease in the effect is observed, which may be associated with decreased amounts of extruded plastic, which is accompanied by porous processes. When the screw rotational speed of 50 rpm the Barus effect changes by 5.3% for a dosage of 0.1% and 0.3% and by 8% for a dosage of 0.5%. When the screw rotation speed of 100 rpm the value of the effect is reduced: by 3.1% for a dosage of 0.1%, by 12.4% for a dosage of 0.3%, and by 5% for a dosage of 0.5%. The largest effect Barus changes occur when the screw rotational speed of 150 rpm and are: 13.4% for a dosage of 0.1%, 8.6% for a dosage of 0.3% and 2% for a dosage of 0.5%.

Measurements of heat flux carried by the LDPE containing microspheres and flow of energy supplied to the extruder made it possible to determine the energy efficiency of the extruder (Fig. 4) and extrusion process (Fig. 5).

Experimental studies have shown a gradual impact of both variables factors, such as dosage of microspheres and screw rotational speed on the value of energy efficiency of extruder and extrusion process. With the increase of screw rotational speed increases the energy efficiency of both the extruder and the extrusion process of porous LDPE, which is confirmed by the information from the literature [10]. The energy efficiency of the extrusion process increases by about 20% when increasing the screw rotational speed to 100 rpm, and about 15% at a speed of 150 rpm. For energy efficiency of extruder this increase is about 20% at a speed of 100 rpm and 11% at a speed of 150 rpm.

Simultaneously, the addition of microspheres to the polyethylene caused a gradual decrease of the energy efficiency of the extrusion process in the whole range of changed extruder screw rotational speed. This may be due to the characteristics of the microspheres, which in order to increase their dimensions take heat from the plasticized material.

Following the addition blowing agent in the form of microspheres to the polyethylene extrudate in the form of a strip with microporous structure was obtained. The appearance of samples in the cross-section are shown in Figure 6. On microscopic images are visible micropores with different diameter and distribution. The largest difference in the dimensions of the micropores take place at dosage of 0.1%, which

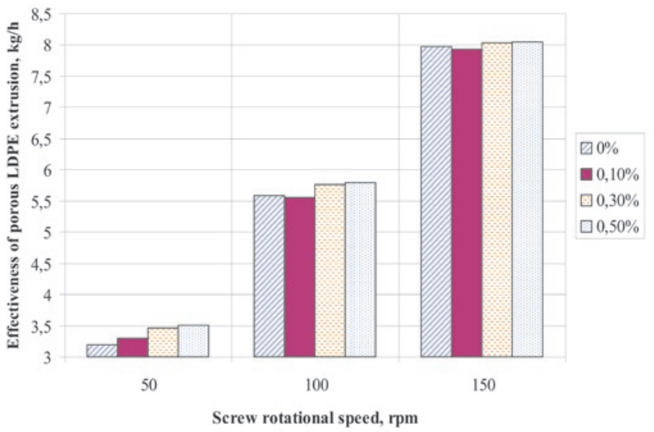


Fig. 2. Dependence of efficiency of LDPE microcellular extrusion on screw rotational speed and dosage of microspheres

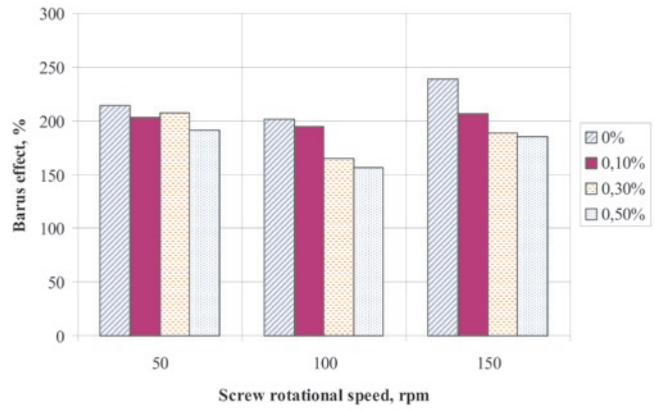


Fig. 3. Dependence of Barus effect on screw rotational speed and dosage of microspheres in LDPE

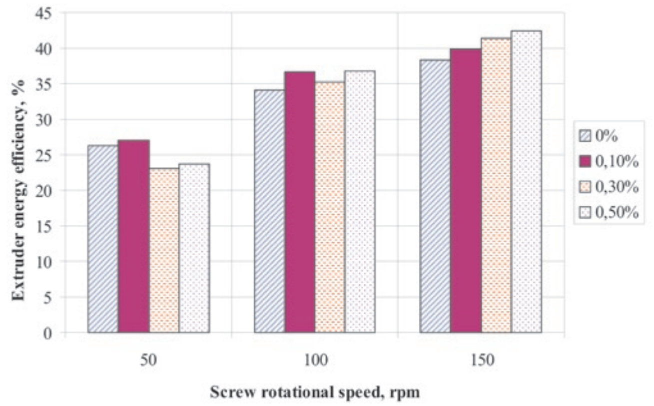


Fig. 4. Dependence of extruder energy efficiency on screw rotational speed and dosage of microspheres in LDPE

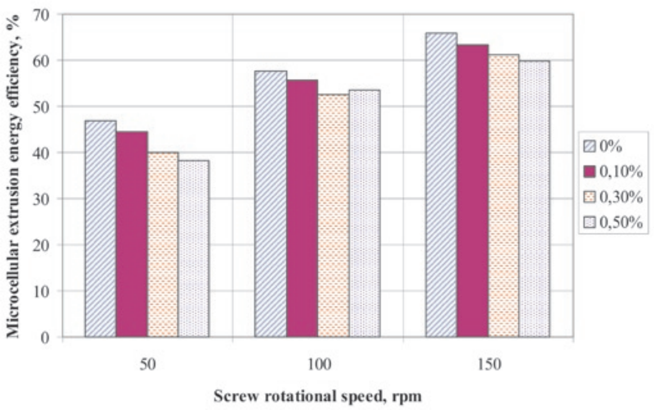
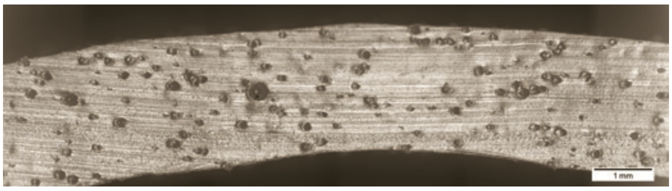
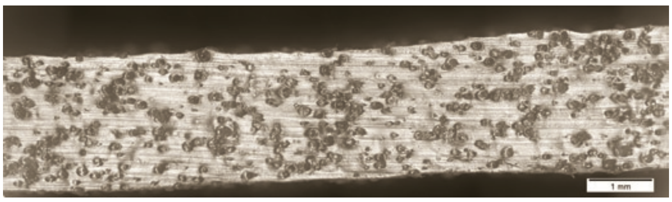


Fig. 5. Dependence of microcellular extrusion process energy efficiency on screw rotational speed and dosage of microspheres

a)



b)



c)

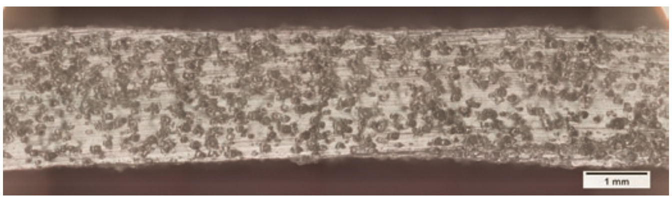


Fig. 6. View of microporous extrudate physical structure (cross section) obtained with screw rotational speed of 50 rpm and with content of microspheres: a) 0,1%, b) 0,3%, c) 0,5%

may be due to a greater possibility of the growth of microspheres in the solid LDPE. Increasing the amount of the dispensed microspheres resulted in growth reduction due to its amount in extrudate.

Based on evaluation of the obtained microporous extrudate structure can be concluded that the degree of mixing of the LDPE and the microspheres is not sufficient. The newly created structure is not homogeneous across the cross-section of extrudate, which can affect the particular physical properties of the product. It is necessary to use additional devices such as dosing dispenser capable of blowing agent in very small quantities or changes in screw construction.

5. Summary

Analyzing the effectiveness of the microcellular extrusion process it has to be consider a number of factors affecting the course and the quality of the process. The most important include primarily changes of the temperature in the extruder plasticizing system and in the extrusion head and the associated pressure fluctuations, changes of the screw rotational speed and increased dosage of microspheres contained in the plastic being processed.

Based on the research it can be concluded that the low density polyethylene modified with blowing agent in the form of microspheres is efficiently extruded by the extruder screw rotational speed of 150 rpm and with a dosage of 0.5% of the microspheres. This is due to the low temperature and pressure changes of the plastic, which positively influences on the degree of mixing, the smallest Barus effect, highest effectiveness and energy efficiency of the extruder and the process.

Presented the results of the temperature and pressure changes of plasticized material, and also dependings of the effectiveness, energy efficiency of the extruder and microcellular extrusion process as a function screw rotational speed is reflected in the literature. However, changes of the efficiency tested as a function of quantity dispensed microspheres show necessity of a broader analysis of the impact of blowing agents on particular process parameters, taking into account different method of agent dosage, the extruder screw design and the residence time of the material in the plasticizing system.

During the evaluation of the effectiveness of this process must take into account its efficiency, which has significant influence on weight reduction of extrudate, resulting from the addition of microspheres to a plastic. These changes result from the amount of energy flux fed to the extruder and the extrusion head, the heat flux (thermal power) carried by the material in the plasticizing system, plastic enthalpy, energy flux heaters of extruder cylinder and extrusion head and also mechanical power supplied to the screw shaft. The values of these factors significantly affect the energy efficiency of the extruder and the extrusion process, which in addition to changes of temperature and pressure as well as the mixing degree of plastic and microspheres are the basic criteria to assessing the effectiveness of the microcellular extrusion process.

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