Advances in Science and Technology Research Journal

Advances in Science and Technology Research Journal, 18(8), 300–310
https://doi.org/10.12913/22998624/194020
ISSN 2299-8624, License CC-BY 4.0

Received: 2024.08.05
Accepted: 2024.10.02
Published: 2024.11.01

Mechanical and Tribological Characterization of Microporous Low Density Polyethylene Films Obtained in Blown Extrusion

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ABSTRACT

Foaming processes for polymeric materials are usually carried out using extrusion and injection methods. This applies primarily to products whose wall thickness is at least 4 mm. The main advantage of using this type of modification is the continuity of the film, without holes and depressions on the surface and a reduction in the density of the product. Foaming of plastics using the blown extrusion process, where we obtain thin-walled products in the form of film, involves the risk of loss of it continuity. Additionally, as a result of modification with blowing agents, the obtained film is porous throughout its cross-section, which causes surface irregularities that affect its tribological properties. The article presents the results of research on selected mechanical and tribological properties and structure of microporous films obtained in the blown extrusion process. The tests showed the influence of the blowing agent used on the tested properties and structure of the film.

Keywords: polyethylene, film, cellular, properties, blown extrusion.

INTRODUCTION

Foaming processes of polymeric materials can be carried out by main processing methods such as extrusion or injection molding [1, 2, 3]. However, in most cases, the modification of polymeric materials with porophores concerns thick-walled products, usually with a wall thickness of more than 4 mm [4, 5, 6]. This is related, among other, to the type and form of the porophore used, the dosage system used, the need to reduce the consumption of the polymer material or the porosity effect itself, and the measurement recommendations adopted in international standards [7, 8, 9, 10, 11].

Foaming of thin-walled components is not an easy or widely used process. This is due, among other, to the problems of maintaining continuity and stability in the dimensions of the product, the strength and usable requirements of such products, and the sheer cost of their manufacture and subsequent recycling. Polyethylene film waste is generally considered unsuitable for recycling due to its low bulk density and is therefore often sent to landfills or disposed of as energy recovery along with other municipal waste [12]. Research is currently underway to develop porous polymer nanocompositions capable of accommodating any functional components to form stable multifunctional materials. Such work is being carried out, among others, by the team of Chunying Fan and Yan Luo using the thermally induced phase separation (TIPS) method [13]. Another method for producing microporous films is presented in the work of [14] involving hot lamination of individual composition layers. As a result, a new bionic microporous polymer evaporation cooling film was developed.

In polymer materials processing, the most commonly used methods for film production are

flat-head extrusion and blown extrusion. These methods are commonly used for both synthetic and biodegradable solid plastics or blends [15, 16]. In these processes, it is important to fine-tune the viscosity and strength of the extruded material [17], which is influenced by the selected manufacturing parameters and the added additives.

Another important aspect of the manufacture of porous films or membranes is the need to maintain their continuity. This determines the applicability of such products. They can be used as membranes in capacitors [18], packaging films [19], horticultural or agricultural films [20], and filters.

In the paper Aizawa and Wakui [21], LDPE/silica nanocomposites are foamed by two different processes. First one is the pressure quench method which is based on the use of a physical blowing agent and second one is the improved compression moulding technique. As the latter process uses a chemical blowing agent, both types of foamed nanocomposites will provide very useful information about the relationship between foaming process-microstructure and macroscopic properties. Results have revealed how silica nanoparticles are able to act as nucleating sites during foaming step in both processes; however, the optimum amount of particles strongly depends on the foaming route. Thermal and mechanical properties of solid and foamed nanocomposites have been analyzed by means of thermogravimetric analysis and compression tests. Results have revealed that nanosilica particles act as effective nucleating agents, not only reducing cell size and increasing cell density but also achieving more homogeneous cellular structures. Thermal and mechanical properties are improved due to the presence of silica nanoparticles. It has been found that the improvement degree reached for samples produced using chemical blowing agents is greater than that achieved for samples produced using physical blowing agents.

As a result of the addition of the porophore to the polymer material, the structure of the product is changed from solid to porous with open or closed pores, depending on the material components, process parameters and design elements of the processing tools and machinery [22]. The resulting changes in the case of thick-walled products are visible in the structure of the product, mainly in cross-section. For thin-walled products such as films, membranes or coatings, the changes are visible on the surface and can affect their physical mechanical and structural properties and application. The influence of the type and amount of added porophore and processing parameters on the functional properties of specific products becomes important. The paper [21] studied the correlation between gas permeability and porosity of a developed polymer filter. It was shown that permeability increased with increasing porosity. In addition, the gas permeability decreased both due to the pore size and the decrease in porosity due to the increase in compression ratio (a feature of the CAPC method). In another paper [23], the effect of changing porous agent content on the tribological and mechanical properties of polyimide, additionally impregnated with oil, was studied. Porous specimens were produced using a pressing method to control the formation of the porous structure. The study showed that once with the increase of porosity, the surface hardness and elastic modulus decreased, while the elongation at break increased. In addition, as porosity increased, the coefficient of friction first decreased and then increased. The blowing agent (azodicarbonamide) used in the study was almost completely removed during the sintering process, resulting in a molded product whose porosity increased with increasing agent content.

This article presents a study of microporous films obtained by free extrusion with vertical upward blowing. The effects of the amount of added polymer microspheres and the changed rotational speed of the film receiving rollers on selected mechanical and tribological properties of the film were analyzed.

MATERIALS AND METHODS

Low density polyethylene LDPE Malen E FABS23D022 (prod. Basell Orlen Polyolefins Sp. z o.o) in granular form was modified. The tested plastic has a normal density of 926 kg/m^3, MFR (190 °C/2.16 kg) of 1.95 g/10 min, melting point of 112 °C, extrusion temperature in the range of 170–220 °C.

For modification of LDPE, a microporous agent in the form of polymer microspheres Expancel 951MB 120 (prod. Akzo Nobe l) was used, characterized by grade of microspheres in a range 951–120, concentration of microsphere 65% in carrier copolymer of ethylene vinylacetate EVA, height of foaming in a range 100–150 mm (in 210 ºC) and bulk density in a range 400–500 g/cm^3.

Within the framework of the established research program, the blown film extrusion process was carried out on a W25/25 single screw extruder with a vertical upward blowing system (Fig. 1). The diameter of the screw is 25 mm, and the L/D of the screw is 25. The constant factors of the extrusion process included: temperatures in the zones of the extruder plasticizing system 150, 165, 170 ºC; temperatures in the zones of the extruder head 170, 175 ºC; diameter of the extruder head nozzle 90 mm; width of the ring gap 0.8mm; screw speed 2.8 s^{-1}. The variable factors and the thickness measurement of the obtained films are summarized in Table 1.

Tests of mechanical properties, i.e., tensile strength, elongation at break, modulus of elasticity and punching were performed on a Zwick Z010 testing machine on 6 samples for each condition. Tests of mechanical properties in static tension were conducted in accordance with PN-EN ISO 527:2020 [24] at a tensile speed of 50 mm/min. Punching tests were carried out in accordance

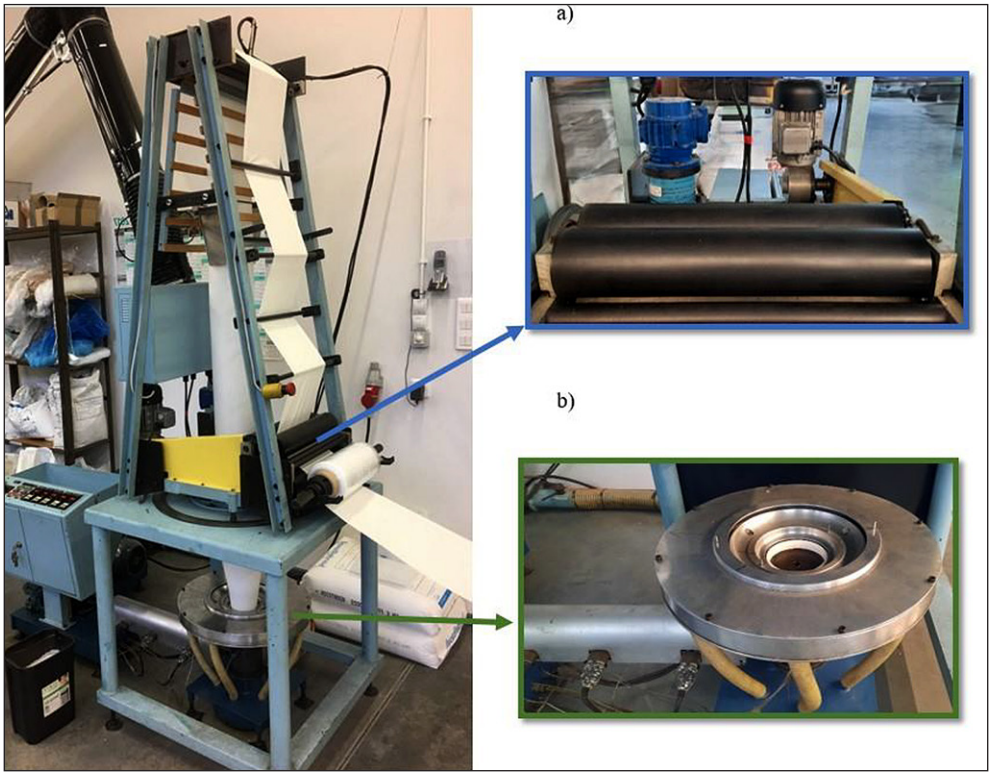

Figure 1. View of blown film technological line: a) receiving rollers, b) extruder head

Table 1. The values of the variable factors and the results of the thickness of the samples

Sample designation	Microspheres dosage, %	Receiving rollers rate, s^{-1}	Sample thickness, mm
PE0M025	0	0.25	0.10
PE0M05		0.5	0.05
PE0M075		0.75	0.03
PE0.5M025	0.5	0.25	0.13
PE0.5M05		0.5	0.08
PE0.5M075		0.75	0.06
PE1M025	1	0.25	0.14
PE1M05		0.5	0.09
PE1M075		0.75	0.06
PE1.5M025	1.5	0.25	0.19
PE1.5M05		0.5	0.08
PE1.5M075		0.75	0.06

with PN-EN 14477:2005 [25] using a standardized blade at a test speed of 50 mm/min.

Frictional contact testing of the film was performed using a test stand that included the Film Friction Tester 7000 from Oakland (Fig. 2). The tester is equipped with a fixed table (known as a sled) with a known mass of 200g and a movable treadmill to which the sample and film countersample are attached. During the test, the treadmill moves relative to the fixed table at a constant speed of 2.54 mm/s. Using an Imada model DS2-1 force gauge, the value of the friction force during the treadmill's movement is recorded. This makes it possible to determine the coefficient of static friction and the coefficient of dynamic friction. The bench meets the requirements of ASTM D-1894, procedure A [26].

Analysis of the surface structure of the samples was performed on a Keyence VHX-7000N digital microscope.

RESULTS

The results of the mechanical properties are shown in the figures 2÷4. The tests showed a decrease in all measured quantities against increasing microsphere content and increasing speed of the receiving rollers.

The results of measuring the elastic modulus EM (Fig. 3) showed a significant and gradual decrease in the modulus, independently for varying receiving rollers rate. At the lowest receiving rollers rate of 0.25 s^{-1}, a decrease in EM was obtained by 31% for 0.5% dosing, 10% for 1.0% dosing and 24% for 1.5% dosing, respectively. Increasing the receiving rollers rate to 0.5 s^{-1} resulted in a further decrease in EM for individual microsphere dosages of 41%, 15% and 9%, respectively. The most intense decrease was obtained for the highest receiving rollers rate of 0.75 s^{-1} after the first 0.5% dosage of microspheres, which amounted to 45% relative to the solid sample. From the point of view of the changed receiving rollers rate, the largest reduction in EM is observed for samples containing 0.5% and 1.0% microspheres at the first rate change from 0.25 s^{-1} to 0.5 s^{-1}, which is 16% and 21%, respectively. Insignificant modulus changes were observed in the remaining samples.

The obtained results of the tensile strength test TS (Fig. 4.) also showed a gradual decrease in the tested size with respect to both the changed content of microspheres in the plastic and the changed film receiving rollers rate. The most intense decrease is seen with the addition of microspheres to LDPE during extrusion at the lowest film receiving rollers rate of 0.25 s^{-1}, where the TS reduction is 40% (for 0.5% microspheres), 19%

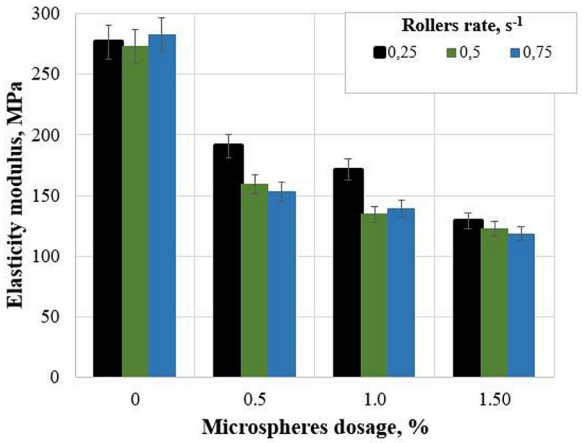

Figure 3. Dependence of elasticity modulus of LDPE films on microspheres dosage and receiving rollers rate

Figure 2. View of the frictional contact testing station: 1 – table with counter sample, 2 – track with measured sample, 3 – dynamometer, 4 – body

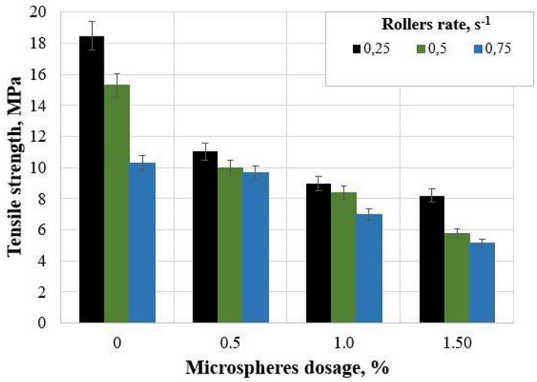

Figure 4. Dependence of tensile strength of LDPE films on microspheres dosage and receiving rollers rate

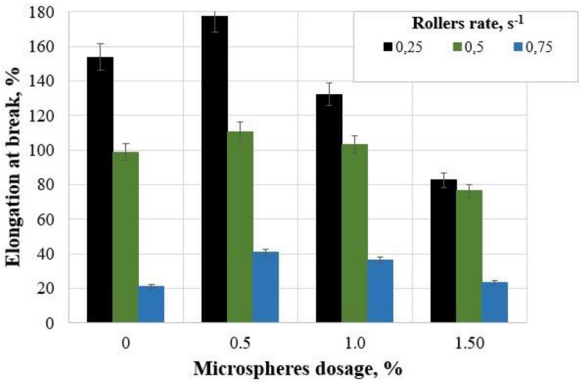

Figure 5. Dependence of elongation at break of LDPE films on microspheres dosage and receiving rollers rate

(for 1.0% microspheres) and 7% (for 1.5% microspheres), respectively. Another change in the receiving rollers rate resulted in a further reduction in TS, with a lower intensity, especially for samples containing 0.5% microspheres, where the decrease in the tested size is 9% (for sample PE0.5M05) and 3% (for sample PE0.5M075), respectively, compared to sample PE0.5M025.

Similar correlations were obtained by Haouari S., Rodrigue D in the work [27], where the addition of corn starch to LDPE caused a significant decrease in the mechanical properties of extruded membranes while foaming the structure.

Investigations of elongation at break EB, shown in Figure 5, revealed a significant effect of the changed receiving rollers rate for all changed dosages of microspheres. The largest EB values are observed for samples extruded at the lowest receiving rollers rate of 0.25 s^{-1}, while the smallest EB values are observed for samples extruded at a receiving rollers rate of 0.75 s^{-1}. The results initially show an upward trend in elongation relative to solid samples over the range of increasing microsphere content to 0.5%, and a subsequent decrease when dosing 1.0% and 1.5% microspheres and over the entire range of increasing the speed of the receiving rollers rate. All samples modified with microspheres and extruded at a receiving rollers rate of 0.75 s^{-1} show an increase in EB with respect to solid samples: by 48% (for sample PE0.5M075), 42% (for sample PE1.0M075) and by 10% (for sample PE1.5M075). Samples containing 0.5% microspheres in all receiving rollers rate ranges show an increase in elongation of 15% (sample PE0.5M025), 12% (sample PE0.5M05) and 48% (sample PE0.5M075) with respect to the corresponding solid samples.

Puncture resistance tests included measurement of film puncture force and elongation at failure. The results were summarized in groups for each dosage of microspheres and are shown in Figures 6 and 7. The puncture force curves of the film samples have a repeatable shape with a clearly marked single maximum, followed by a gradual decrease in the measured quantity. The most intense increase in punching force is observed for samples extruded at the lowest rotational speed of the receiving rollers rate of 0.25 s^{-1}. With a further increase in the rotational speed, there is a decrease in the value of the maximum punching force and the intensity of the increase in the force required to punch the film. The decrease in the maximum punching force of the film is the largest at the first change in the receiving rollers rate (from 0.25 s^{-1} to 0.5 s^{-1}) and is similar for all the samples tested, averaging 38%. Further increasing the film take-up rate to 0.75 s^{-1} results in a further, less intense decrease in the breakthrough force of all samples and is 19%, 24%, 23% and 42% for samples containing 0, 0.5, 1.0 and 1.5% wt. of microspheres, respectively. The study also showed the effect of added microspheres on the shift of the peak of the maximum puncture force relative to the displacement of the arrowhead. As the content of microspheres in polyethylene increases, a change in the displacement of the arrowhead when the sample is punctured is observed. These changes are not the same for each dosage of microspheres, which is due to the uneven distribution and dimensions of the resulting micropores in the film structure.

The study showed the influence of the added microporous agent in the form of microspheres and the changed film receiving rollers rate, which

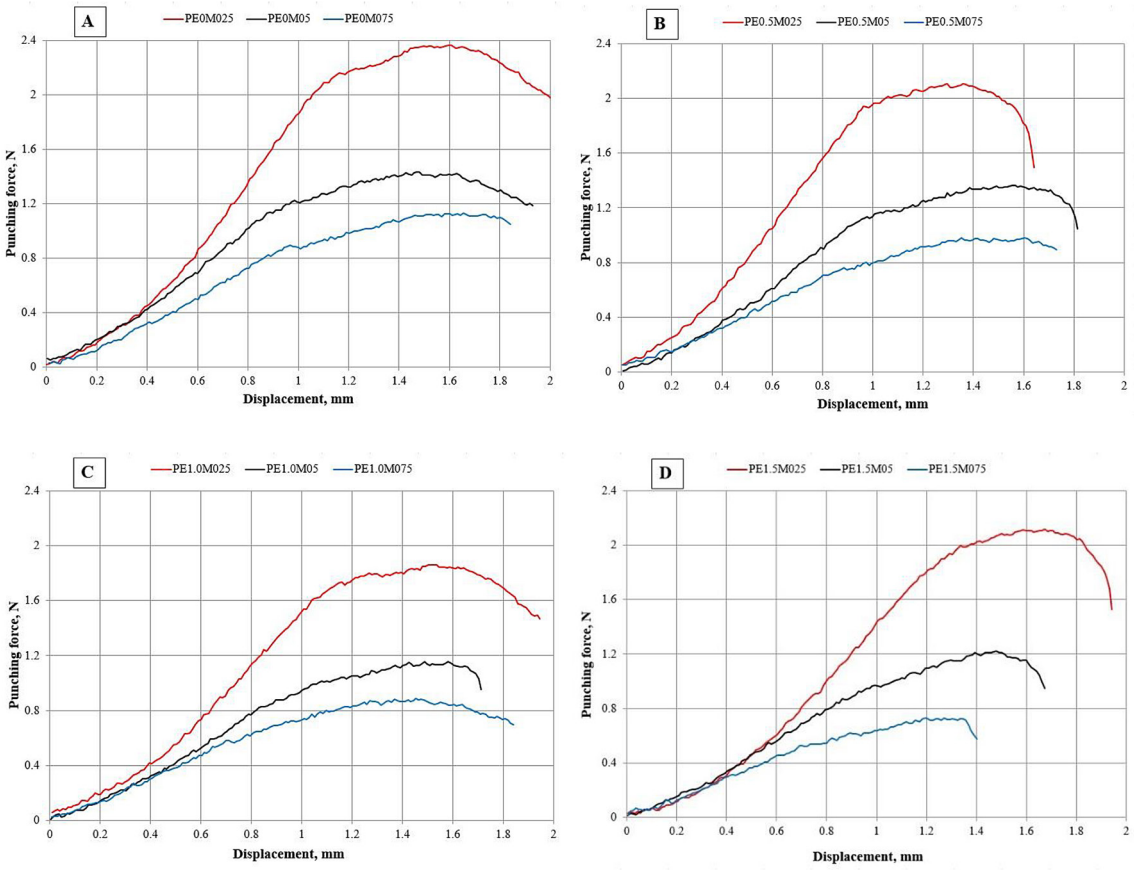

Figure 6. Example force-displacement curves of punching testing for samples containing: A – 0% microspheres, B – 0.5% microspheres, C – 1.0% microspheres, D – 1.5% micropsheres

may be related to the thickness and flexibility of the extruded film. In addition, the study determined the elongation at the punching of the film (Fig. 7) during destruction with the arrowhead. The study showed that the addition of a microporous agent in the form of microspheres results in a decrease in elongation compared to solid film for any changed receiving rollers rate. At the same time, the first dosage of microspheres in the amount of 0.5% turned out to be significant, which resulted in a decrease in elongation at the punching by an average of 14% at the rate

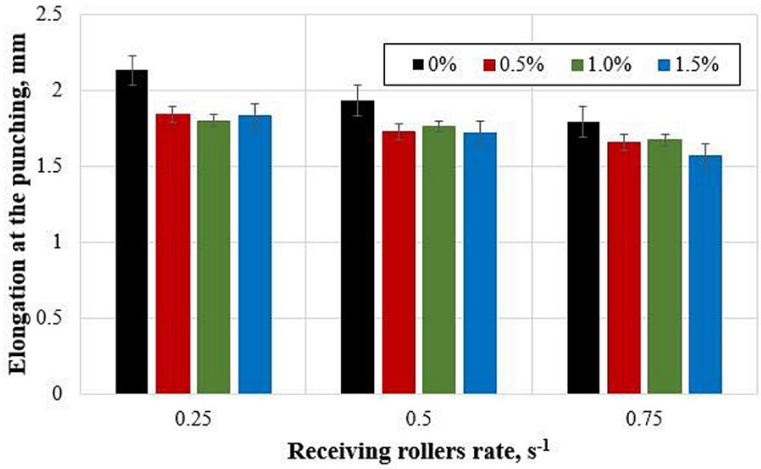

Figure 7. Dependence of elongation at punching of LDPE films with different microsphere content as a function of changed speed of receiving rollers

of 0.25 s^{-1}, 10% at the rate of 0.5 s^{-1} and 7% at the rate of 0.75 s^{-1}. In addition, changing the receiving rollers rate also resulted in a decrease in the tested size by an average of 10% for the solid sample by 5% at the dosage of 0.5% microspheres, 3.5% at the dosage of 1.0% microspheres and by 6.5% at the dosage of 1.5% microspheres. The variation in the test results obtained may be indicative of the inhomogeneity of the film throughout the cross-section due to the free expansion process of microspheres in polyethylene until the expansion temperature of the blowing agent is lowered.

Friction tests

The results of the tribological tests are shown in the graphs of the dependence of friction force on friction contact time (Figs. 8–10) and in Table 2. The study showed the influence of variable factors on frictional force and coefficient of friction. Measurements of friction force during friction contact time showed that at the lowest receiving rollers rate of 0.25 s^{-1} (Fig. 8), the average friction force increases with increasing porophore content and is 0.281 N for PE0M025 solid sample, 0.516 N for PE0.5M025 sample, 0.674 N for PE1M025 sample and 0.858 N for PE1.5M025 sample. Further increasing the receiving rollers rate changes the range of the average friction force. For a receiving rollers rate of 0.5 s^{-1} (Fig. 9.), the range of average friction force is from 0.324 N for the solid sample to 0.601 N for the sample with 1.0% porophore dosage, for 0.75 s^{-1} (Fig. 10.) the range is from 0.305 N to 0.638 N also for the sample with 1.0% dosage. During extrusion at higher receiving rollers rates, a significant reduction in the frictional force curve as a function of contact time and average frictional force of about 60% is observed compared to the receiving rollers rate of 0.25 s^{-1}.

As the porophore content increases in a fixed range, the coefficient of friction (Table 2) increases for samples obtained at all tested values of the receiving rollers rate. At the same time, it

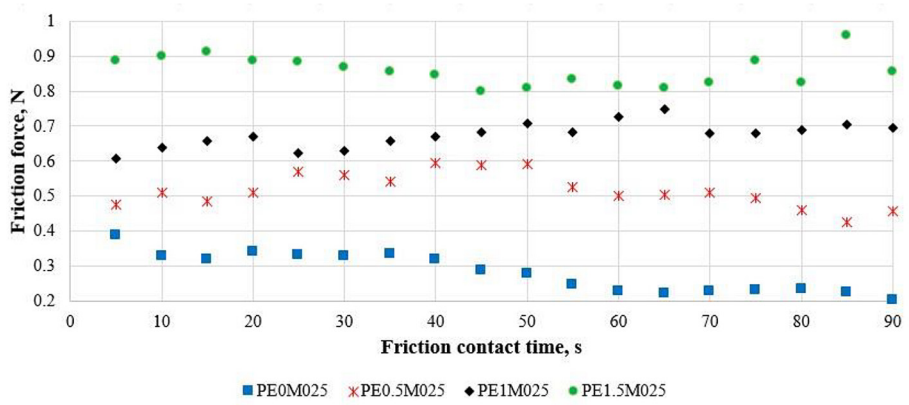

Figure 8. Dependence of friction force on friction contact time of selected LDPE films obtained at the receiving rollers rate of 0.25 s^{-1}

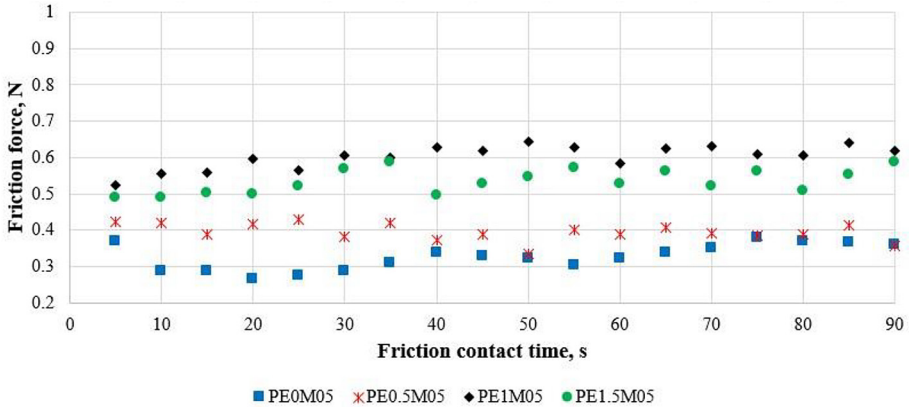

Figure 9. Dependence of friction force on friction contact time of selected LDPE films obtained at the receiving rollers rate of 0.5 s^{-1}

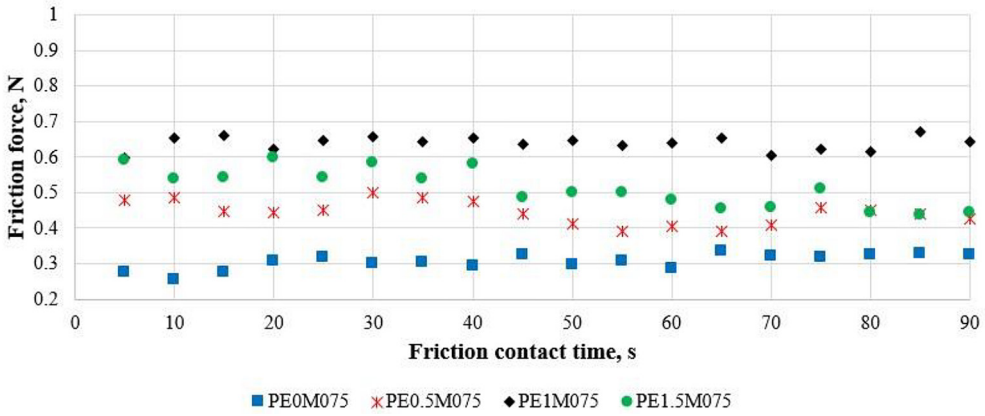

Figure 10. Dependence of friction force on friction contact time of selected LDPE films obtained at the receiving rollers rate of 0.75 s^{-1}

adopts the highest values for samples obtained at the lowest receiving rollers rate (0.25 s^{-1}), and the lowest for the average (0.5 s^{-1}).

In the absence of porophore, the values of the coefficient of friction of samples obtained at different rotational speeds practically do not differ, and are in the range of 0.14–0.17. For samples obtained at the smallest receiving rollers rate 0.25 s^{-1}, the value increases significantly with an increase in the dosage of microspheres by up to 304% (for sample PE1.5M0.25). For the other receiving rollers rate of 0.5 s^{-1} and 0.75 s^{-1}, an increase in the maximum coefficient of friction is observed as the microsphere dosage increases to 1.0% by 186% and 205%, respectively, followed by a decrease of 88% and 80%, respectively.

The obtained changes in the friction coefficient values may be due to the distribution of the formed micropores on the film surface The unregular distribution and the varying number and size of micropores affect the formed tribological contact and adhesion.

An increase in the coefficient of friction with increasing porosity of the samples was observed by Hui Zhang's team [28]. They showed that under dry friction conditions, the wear resistance of porous materials was closely related to their own porosity. NaCl-pored PEEK-based composite samples had relatively smooth surfaces and minimal coefficient of friction. As the porosity increased, the large number of pores changed the smooth surface and compact structure of the sample, and the degree of surface roughness increased. On the other hand, the increase in porosity reduced the mechanical strength of the sample, making the sample more susceptible to wear, creating a more hostile friction environment, and the friction coefficient increased.

In the work of [29], the effect of porosity on the coefficient of friction under various normal loads of EVA foams was studied. These studies indicated, friction coefficients for EVA foam blocks tended to increase with an increase in porosity. and the increase in friction coefficient with an increase in porosity under different normal load conditions was due to an increase in adhesive friction. In order to reduce the coefficient of friction of porous materials or to achieve a self-lubricating effect, the surfaces of the samples are impregnated with silicone oil or polyalphaolefin [23, 30], but this conditions the further applicability of the creations.

Table 2. Research results of coefficient of friction of microporous LDPE films

Coefficient of friction			
Microspheres dosage, %	Receiving rollers rate, s^{-1}		
	0.25	0.5	0.75
0	0.144	0.166	0.159
0.5	0.259	0.206	0.224
1.0	0.337	0.310	0.327
1.5	0.438	0.273	0.261

Morphology

The results of the microscopic structure analysis of the surface of the samples are shown in the Figure 11 and in Table 3. It can be seen that the structure obtained by extrusion blow molding is characterized by an irregular distribution of closed micropores of circular and elliptical shape. The obtained micropores have a peculiar polymer envelope with visible irregularities on its surface (Fig. 11D). Microscopic analysis of the structure of the samples showed that the number of micropores formed is affected by the content of the microporous agent in the plastic. The rotational speed of the receiving rollers does not affect the number, diameter and perimeter of the micropores formed. The area of micropores in relation to the tested area of the sample is 21.11% for samples with 0.5% microspheres, 27.23% for samples with 1.0% microspheres and 56.99% for samples with 1.5% microspheres. The study showed that as the content of microspheres in polyethylene increases, the perimeter and the minimum and maximum diameters of the micropores formed gradually increase. An increase in the perimeter of the micropores by 24% is observed when dosing with 1.0% microspheres and by 15% when further increasing the dosage to 1.5% microspheres. Feret diameter doubles after increasing the dosage to the level of 1.0% microspheres after which it does not increase significantly with another dosage of 1.5% microspheres. The circularity of the microspheres does not change significantly with increased dosage of the agent.

Studies have shown that increasing the content of microspheres in LDPE increases the size of the micropores, but this increase is not proportional. Additionally, growth is influenced by the position of micropores relative to each other. Where agglomerations of micropores are visible, their dimensions are extreme relative to each other, which is visible when the content of microspheres is highest. In samples with a lower content of microspheres, their growth is more free and results

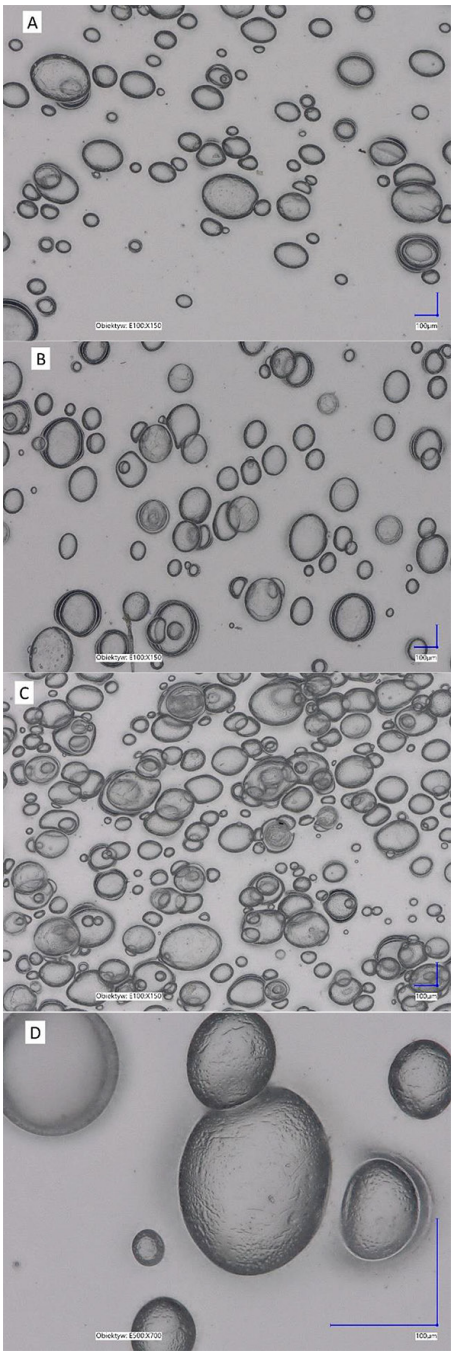

Figure 11. Example view of the microscopic structure of LDPE film obtained at a receiving rollers rate of 0.25 s^{-1} containing microspheres in quantity: A – 0.5%, B – 1.0%, C – 1.5%, D - example view of individual microspheres (magnification: 150x for A, B, C and 700x for D)

Table 3. Results of microscopic analysis

Mocrospheres dosage, %	Total micropores area*, μm²	Micropores perimeter*, μm	Min. micropores diameter*, μm	Max. micropores diameter*, μm	Feret diameter*, μm	Circularity*
0.5	625553.37	29.32	5.25	8.26	6.93	2.19
1.0	806739.08	36.52	6.05	10.55	10.26	2.17
1.5	1688438.34	41.97	6.89	10.62	10.17	2.14

***Note:** the results presented are average values.

in micropores of similar diameter, especially where individual cells are not closely adjacent to each other. Similar observations were presented in [31], where the cell size tended to decrease depending on the foaming temperature, but was not necessarily proportional. Non-adjacent cells had increasing cell size with increasing foaming temperature. However, in the case of foamed samples in which the foam cells were densely located and close to each other, their growth interfered. The obtained cell sizes showed higher values, which resulted from the type of dosed blowing agent in the form of compressed gas introduced at high pressure.

CONCLUSIONS

The foaming process of thin-walled products by blown film extrusion with the use of chemical modifying agents is difficult to carry out due to the possibility of loss of continuity of the produced film. During the process, micropores are created throughout the entire cross-section of the extrudate and on its surface, which significantly changes the mechanical, tribological and structural properties of the film.

The conducted research has shown that the mechanical and tribological properties of thin-walled porous products are closely related to the obtained microporous structure and should take into account the effects of several factors, such as density, pore size and their distribution in the material structure.

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