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MICROEXTRUSION OF FILLED POLYPROPYLENE

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Abstract

This paper presents the results of microgranulation process of polypropylene to which 1 wt % talc was introduced as a nucleating agent. The additive to increase the thermal reflectance was titanium white. The additives improving the ignition resistance were two halogen-free flame retardants: N-alkoxy hindered amine (a triazine derivative) and zinc borate. Whereas, the additives improving mechanical strength were nanofiller in the form of modified MMT montmorillonite and wood flour. An existing single-screw extruder with the symbol W25 was used for the study. A cross-flow microgranulation head with an interchangeable fillet with 18 dies and a diameter of 1 mm was designed and manufactured, together with a special connector, and a roller haul-off device with pneumatic top roller pressure and bottom roller drive, coupled to an independent rotary cutter with 9 cutting knives. The result was a random polypropylene microgranulate containing 1% nucleating agent filled with the prescribed amount of filler in the form of titanium white, halogen-free antipyrrophenes: zinc borate and a triazine derivative, and montmorillonite. The grain size of the microgranulate was 0.5 to 0.6 mm.

Keywords: extrusion, pelletizing, cold granulation, micropellets, polypropylene

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1 INTRODUCTION

In recent years, there has been growing interest in the polymer plastics processing market in the rapidly developing methods of plastic foaming, for which plastic with smaller than pellet sizes is increasingly used. For this reason, increasing attention has begun to be paid to the process of microgranulation of plastics, in which regular particles of 0.4 to 0.8 mm in size are formed [1]. This is particularly important in the manufacture of foamed polypropylene (EPP) parts for the automotive industry and thin-walled items with complex shapes. However, for this industry, foamed parts need to have more favourable mechanical and thermal characteristics, as well as increased non-flammability [2]. This makes it necessary to develop new material formulations that would ensure the efficient production of foamed parts with appropriate mechanical and thermal properties and with reduced flammability, while mastering the technique of obtaining micropellets, which is more demanding than standard extrusion techniques [3, 4] and is not computer-aided like other methods [5].

2 TEST STAND

An existing W25 single-screw extruder, located in the laboratory of the Lublin University of Technology, was used for the tests, for which a cross-head for microgranulation was designed and manufactured (Figure 1) with an interchangeable fillet with a number of openings being 18 and a diameter of 1 mm, together with a special connector. A cross section through the filier, on which the length of the dies, their diameter and its change and other processing requirement are marked, is shown in Figure 2.

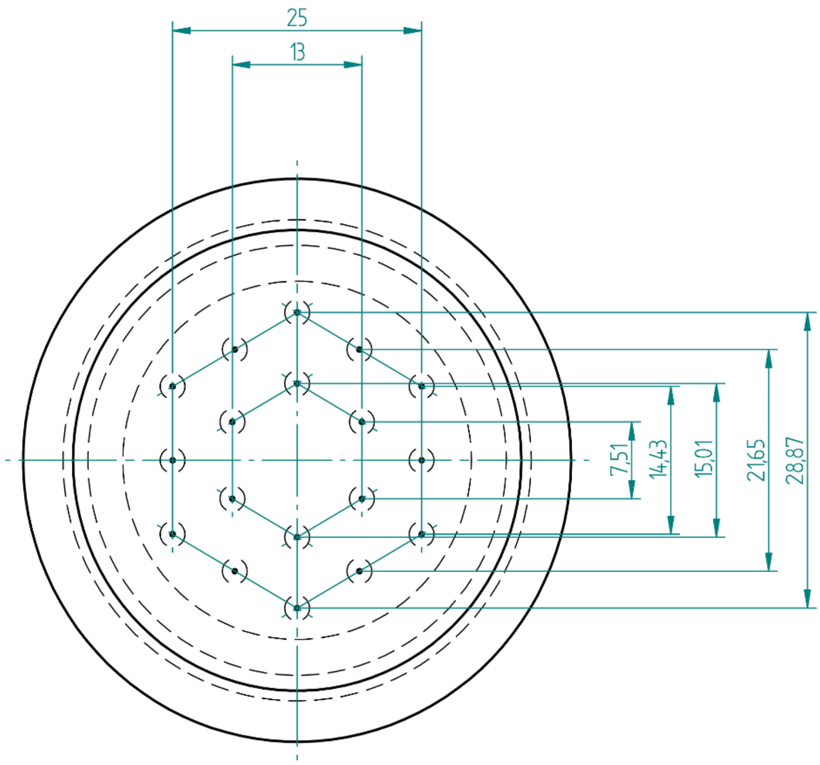

Figure 1: Front of the extrusion head with 18 dies

In addition, it was assumed that the stand would be equipped with a granulating device that would allow the extruded plastic strands to be extracted and mechanically cut into micropellets. For this purpose, a roller haul-off device with pneumatic top roller pressure and bottom roller drive, coupled to an independent rotary cutter with 9 cutting knives was designed and manufactured.

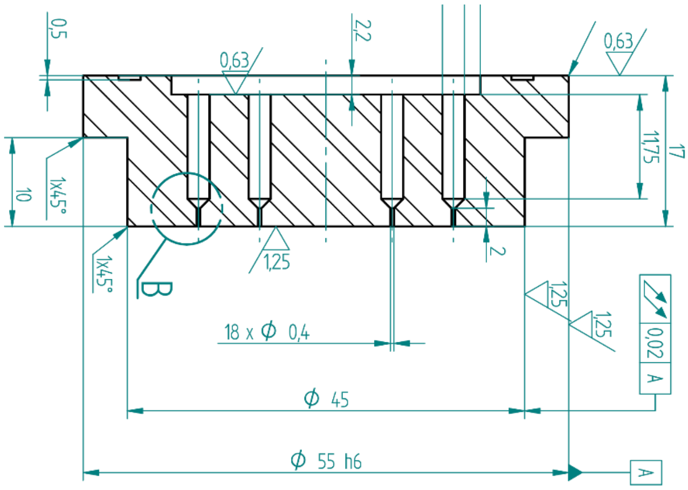

Figure 2: Cross section of the cross fillet of the microgranulation head

This solution is designed to ensure that the extruded strands are stretched, resulting in a smaller diameter and cut to any desired length.

The following technological parameters of the extrusion process with cold microgranulation were established:

- temperature distribution of the barrel along the length of the plasticizing system and extrusion head (set on the extruder): 150 °C, 190 °C, 180 °C and 180 °C,
- extruder screw speed: 30 rpm,
- linear speed of belt and roller haul-off: 11.5 m/min,
- cooling water temperature: 15 °C,
- rotational speed of the rollers of the haul-off device in the granulator: 18 rpm,
- rotational speed of the granulator cutting knife: 60 rpm.

The appearance of the cold extrusion microgranulation technological line used in the study is shown in Figure 3.

Figure 3: Technological line used in the study.

2.1 Materials used in the study

The base material used in the study was a random polypropylene to which 1% by weight of talc was introduced as a nucleating agent. The additive to increase the thermal reflectance was titanium white. Additives to improve ignition resistance were two halogen-free flame retardants: an N-alkoxy hindered amine (a triazine derivative) and zinc borate. On the other hand, additives improving mechanical strength were nanofiller in the form of MMT-modified montmorillonite and wood flour.

2.1.1 Polymer

Polypropylene is a random copolymer produced by Ineos with a medium ethylene content and no special additives. Its basic properties are shown in Table 1.

Property	Test method	Value
Mass flow rate, (230ºC; 2,16kg)	ISO1133	7
Modulus of elasticity, MPa	ISO178	900
Tensile strength at yield point, MPa	ISO527-1, 2	27
Melting point ºC	ASTM D3418	143,5

Table 1: Basic properties of PP.

2.1.2 Absorber IR

The manufacturer of the titanium white used in the study is Venator (USA), which has a representative office in Poland and produces titanium white with the trade name Altiris 550 and a grain size of 0.70 m.

2.1.3 Antipyrrophenes

The manufacturer of the flame retardant in the form of a concentrate based on PP and a triazine derivative (N-alkoxy hindered amine - NOR) used in the study is MK Kolibri Sp. z o.o., whose offered concentrate has a 20% NOR content in PP and comes in the convenient form of pellets. The agent, with trade symbol MK-120041, is designed to reduce the flammability of fibers and thin-walled products and is effective at concentrations ranging from 2.5% to 7.5%. The hydrated zinc borate used in the study was produced by Przedsiębiorstwo Przemysłowo-Handlowe "Standard" Sp. z o.o. of Lublin, whose product, with a grain size of 2 to 5 mm, was used to prepare a concentrate in the form of a pellet containing 50% by weight of PP and 50% by weight of hydrated zinc borate.

2.1.4 Reinforcing additives

For the purpose of the study, a composition containing 10% by weight of montmorillonite (MMT) and containing 30% by weight of wood flour was prepared based on a random polypropylene-ethylene copolymer.

2.2 Test metodology

All compositions for the study were obtained using a standard co-rotating twin-screw extruder and granulation head to obtain standard-size pellets, which, after being mixed with an appropriate amount of primary PP, were placed in the hopper of a W-25 extruder with a special microgranulation head, which is part of the cold microgranulation extrusion technological line shown in Figure 1.

3 RESULTS

As a result of selecting the correct operating parameters of the individual components of the cold extrusion microgranulation technological line, i.e. the extruder, cooling bath, haul-off and granulator indicated in Section 2, micropellets with dimensions ranging from 0.5 to 0.6 mm were obtained. The obtained micropellets based on the indicated PP, IR absorber, flame retardants, montmorillonite and wood flour are shown in Figure 4.

Figure 4: Micropellets with the addition of (a) titanium white, (b) MK-120041 flame retardant, (c) zinc borate, (d) montmorillonite, and (e) wood flour.

Thus, the work involved the formulation of material mixtures, which are shown in Table 2.

Additive to increase the reflectivity of the surface		
Recipe 1	Recipe 1	Recipe 1
titanium white: 2% mas.	titanium white: 2% mas.	titanium white: 2% mas.
Antipyrine: an additive to improve ignition resistance		
Recipe 4	Recipe 4	Recipe 4
Triazine derivative: 0,5% mas.	Triazine derivative: 0,5% mas.	Triazine derivative: 0,5% mas.
Recipe 7	Recipe 8	Recipe 9
zinc borate: 2,5% mas.	zinc borate: 5,0% mas.	zinc borate: 7,5% mas.
Additive to improve mechanical properties		
Recipe 10	Recipe 11	Recipe 12
wood flour 30% mas.	montmorillonite: 5% mas.	montmorillonite: 10% mas.

Table 2: Summary of recipes received.

4 CONCLUSION

As a result of the work carried out, random polypropylene micropellets containing 1% nucleating agent filled with the prescribed amount of filler in the form of titanium white, halogen-free flame retardants zinc borate and a triazine derivative, and montmorillonite were obtained. In the group of recipes containing an additive to improve mechanical properties, two types of micropellets were obtained, with grain sizes ranging from 0.5 to 0.6 mm, containing 1% by weight of talc and 5% by weight of lamellar nanofiller (montmorillonite) and 1% by weight of talc and 10% by weight of lamellar nanofiller, respectively. In the group of recipes with an additive to increase thermal reflectance, three types of micropellets were obtained with 1% wt. talc and 2% wt. titanium white, 1% wt. talc and 4% wt. titanium white, and 1% wt. talc and 6% wt. titanium white, respectively. On the other hand, in the group of recipes for improving ignition resistance, 6 types of micropellets with halogen-free flame retardant were obtained: N-alkoxy hindered amine (triazine derivative) 1% talc and 0.5% antipyrrole, 1% talc and 1.0% antipyrrole, and 1% talc 1.5% antipyrrole, respectively, and with zinc borate 1% talc and 2.5% antipyrrole, 1% talc and 5.0% antipyrrole, and 1% talc and 7.5% antipyrrole, respectively.

In the case of the filler in the form of wood flour, after repeated trials with different combinations of strand extrusion process parameters, micropellets with this filler based on polypropylene were abandoned due to the clogging of the micro-dies in the extrusion head.

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