Analysis of the influence of the geometric features of the filler on the thermal properties and structure of the composites in the polypropylene matrix

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Abstract. The article presents the results of research on polymer composites based on polypropylene filled with various fillers. The physical and thermal properties of the composites are the result of the used polymer matrix as well as the properties and geometric features of the used filler. The geometric shape of the filler is particularly important in the processing of plastics in which the flow is forced, and high shearing tension occurs, which determines the high macromolecular orientation and specific arrangement of the filler particles. Thermal analysis (STA) was used in the research and photographs were taken using a scanning electron microscope (SEM) of fractures of polymer composites. The following fillers were used: talc, fibreglass, glass beads, and a halogen-free nitrogen-phosphorus flame retardant. The test material was obtained by extrusion. Shapes for strength tests, which were subjected to scanning microscopy tests after a static tensile test, were obtained by injection. The carried-out tests allowed us to determine the influence of the type and shape of individual fillers on structural changes in the structure of polypropylene composites and the degree of sample weight loss in a specific temperature range, depending on the used filler.

Key words: polypropylene-based composites; mechanical properties; thermal properties; fillers.

1. INTRODUCTION

Polymer materials have specific properties and are characterized, in most cases, by the ease of processing and obtaining a large batch of products at the same time. Therefore, they have found applications in many industry branches [1]. They are used on a large scale in the packaging industry, construction, automotive industry, and many others. The wide range of applications of polymer materials means that it is needed to research solutions to improve the physical, mechanical, thermal, and combustible properties of plastics [2–5]. Changing their properties allows for a wider range of their usage. An example is the usage of polymeric materials to produce composite reinforcing bars, which are made in the pultrusion process by embedding fibreglass in a polymer matrix based on epoxy resin [6–8]. These bars have better functional and mechanical properties, and most importantly, significantly higher corrosion resistance compared to traditional steel bars. They are lighter, which facilitates transport and subsequent application, and puts less strain on the load-bearing elements of the buildings. Another example of the usage of composites based on polymeric materials is the automotive industry, where traditional materials have been replaced with elements based on polymers for many years [9]. However, it is noteworthy that they are more commonly used in the engine compartment, ergo, in areas with an increased temperature. Of course, before using the material in

these spaces, first, they must be subjected to appropriate modification and long-term tests allowing for conscious application in given environmental conditions and trouble-free operation for a long time.

To modify polymeric materials, diverse types of fillers are used that affect the physical, functional, and structural properties of the final products based on polymer composites [10–16]. The purpose of modifying plastics is to change diverse types of functional, physical, mechanical, thermal, and combustible properties. However, one should not forget that an important motive for using diverse types of fillers to modify plastics is to reduce the price of the final product. Commonly used diverse types of fillers, more or less significantly, change the above-mentioned properties and make the composites based on the polymer matrix have reduced flammability, better thermal resistance, thermal conductivity, shape durability, and higher or lower coefficient of friction [17–19]. There are several classifications of fillers in the available literature [20]. One of them divides the materials used to modify the plastic into active and inert fillers. The first of them, otherwise known as amplifiers, cause the previously mentioned changes in diverse types of properties – improving them. The latter, called fillers, are used to increase the mass and volume of the polymer composite – usually at the expense of losing certain properties.

Another well-known division of polymers classifies them according to their shape and is as follows:
- Mineral fillers (nuggets and flakes):
  - Ground chalk, limestone, marble, precipitated calcium carbonate (< 0.7 mm).
  - Quartz flour, kaolin, feldspar, aluminum hydroxide, silica.

- Spherical fillers (improve the flow of plastics, reduce shrinkage, increase shape durability):
  - Full glass beads (< 50 μm) – hardness.
  - Hollow fly ash balls (5–250 μm) – high resistance to compression, reduction of product weight.
- Fibrous fillers (limit the flow of plastics, reduce shrinkage, increase stiffness and strength).

An interesting group of fillers are flame retardants of polymer composites called flame retardants. Flame retardants introduced into the structure of the polymer composite make it more resistant to burning. For example, the value of the oxygen index (OI) increases [21–23]. If the OI value for a given material rises above 21, there should be no combustion that is self-sustaining after removal of the pilot energy stimulus under test conditions, or in natural conditions after elimination of exposure to flame and heat.

The mechanism of action of flame retardants depends on their structure and can be both chemical and physical. The chemical mechanism consists of the fact that during the exposure of the polymeric material to an energy stimulus (e.g. flame), free radicals that support the combustion reaction in the gas phase are deactivated. In addition, in the solid phase, a layer of porous coke is formed on the surface of the material, which isolates the unburned part of the material from the flame. However, in the case of the physical mechanism, in the gas phase, the combustible gas-air mixture is diluted, and heat is rapidly absorbed from the material. Inert gases (e.g. nitrogen and argon) used to extinguish fires work in an analogous way. Introduced into the combustion zone, they displace the air, which significantly reduces the oxygen concentration to below 12.5%. In such an atmosphere, there is a disproportion between the concentration of the oxidant and the combustible gas, and the combustion reaction ceases to take place. It should be emphasized that not only the type of filler used, but also its shape both have an impact on the change of its properties.

The work aims to examine the effect of the type of filler on the thermal and mechanical properties of composites based on polypropylene. In addition, the composition of the composite based on polypropylene with the addition of a flame retardant and talc was proposed in the paper, ensuring the appropriate value of the oxygen index.

2. MATERIALS AND METHODS

In order to modify the materials, the homopolymer granulate was mixed with a properly weighed number of individual fillers and then, using a co-rotating twin-screw extruder, it was subjected to plasticization and homogenization processes. After extrusion and cooling, the filament went to the granulator. As a result, composite granules were obtained in a polypropylene matrix with a variable content of the fillers presented above. The produced samples were divided into four series depending on the used filler. The processing process was conducted using a co-rotating twin-screw extruder equipped with a zone of degassing and side dosing of fillers. Depending on the type of filler, dosing was done from the side feeder closer to the main hopper or closer to the extrusion head (temperature range from 190°C to 230°C).

The first of them are composites in which the Luzenac OXO filler was used. It is a white talc with a lamellar structure and a density of 2.78 g/cm^3, mainly used to obtain an aesthetic surface. Another filler used in the tests was glass beads (full GB and empty GBL), which are characterized by the following properties that improve the processing and flow in the mass of the injected material: smooth surface, the smallest possible ratio of surface to volume, high load capacity, excellent form of flow, uniform dispersion. Materials filled with glass beads improve mechanical properties (surface hardness, strength, durability, high modulus of elasticity), excellent chemical resistance, shrinkage and low warping, and flow. A-Glass 3000 glass beads with an average particle size between 30 and 50 μm and a density of 2.5 g/cm^3 were used as the filler.

Polypropylene composites filled with glass fiber were also tested. These materials, in addition to being used in the household appliances, automotive, and electrical industries, are also an alternative to PA 6. The materials were filled with glass fiber ECS13-4.5-508A, type E glass, cut CS, fiber diameter 13 μm, cutting length 4.5 mm.

The last group of polymer composites tested were composites based on polypropylene with fire retardant and talc. To modify the above Dolomite talc was used, as well as ADK STAB FP2100JC, a halogen-free nitrogen-phosphorus flame retardant with good fire resistance while maintaining mechanical properties. The used flame retardant shows high processing stability when combined with thermoplastics. It is recommended for usage in combination with polypropylene (homo- and copolymer) and high- and low-density polyethylene and in ethylene-vinyl acetate copolymers. The selection of composition No. 7 was conducted based on our own research and the assumption of reducing the appropriate value of the oxygen index. For this purpose, a composition consisting of a higher proportion of a cheap filler, i.e. talc, and a minimum amount of flame retardants was proposed to ensure the appropriate value of the oxygen index. Table 1 summarizes the characteristics of the produced composites.

The TGA test made it possible to determine the mass loss of individual polypropylene composites when heated in a protective nitrogen atmosphere. Samples weighing about 10 mg, placed in ceramic crucibles, were heated in the temperature range from 45°C to 850°C with a heating rate of 30°C/min. To perform the TGA test, pellets of individual composites in a polypropylene matrix were used. The weight of the sample was checked using a laboratory balance. This test was conducted in accordance with the PN-EN ISO 11358-1:2014-09 standard using the STA 449 F5 Jupiter Netzsch device.

As a result of the test, graphs of the dependence of mass loss as a function of the temperature to which the test chamber was heated were obtained. Based on the obtained results, the mass loss start and end temperatures, the percentage of residue, and the temperature at the maximum decomposition rate determined from the TGA first derivative curve were defined.

The tests of the static tensile were conducted using the universal testing machine Inspekt Desk 20 by Hegewald & Peschke with a measuring head up to 20 kN and a tensile speed of 50 mm/min. Five repetitions were made for each of the sam-

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Table 1
Characteristics of the produced samples of composites based on polypropylene

No.	Characteristics	Sample Name
1	Polypropylene – reference	PP
2	Composite in a polypropylene matrix with 30% volume of hollow microspheres	PP GBL 30
3	Composite in a polypropylene matrix with 30% volume of solid microspheres	PP GB 30
4	Composite in a polypropylene matrix with 30% volume of fibreglass	PP GF 30
5	Composite in a polypropylene matrix with 20% volume of talc	PP T 30
6	Composite in a polypropylene matrix with 30% volume of ADK STAB FP-2100JC	PP WH HF
7	Composite in a polypropylene matrix with 20% volume of dolomite talc and 6% volume of ADK STAB FP-2100JC	PP T 20 V0

ples, while the figure shows exemplary runs of individual samples. They show the nature of the tensile test and the nature of the behaviour of the material in the static tensile test.

Injection molded samples were used. The granulate, which was obtained by the extrusion process described above, was dried at 65 degrees Celsius for two hours before the injection process. A Shini CD9 Cabinet laboratory dryer was used for drying the granulate. A KraussMaffei KM 65 160 C4 injection molding machine was used in the injection process. They were produced using standard injection parameters. The samples were then conditioned for 48 hours.

For fractures of samples subjected to a static tensile test, microscopic examinations were conducted. The microstructure of the composites was examined using a HITACHI S-4700 scanning electron microscope (SEM) at the Institute of Geological Sciences, Faculty of Geography and Geology, Jagiellonian University in Krakow.

For SEM tests, the samples were cut to a height of approx. 10 mm so that they fit under the microscope objective. They were then glued to the microscope stage using double-sided carbon tape. Four samples could fit on the microscope stage at the same time. The samples prepared in this way were additionally sputtered with gold (due to the lack of electrical conductivity of the samples produced). In two cases, samples that were initially frozen in liquid nitrogen and then subjected to breaking were used for the tests. This concerned polypropylene composites with glass fiber (PP GF 30) and solid glass beads (PP GB 30). In the first case, the surface, after breaking the sample, was so irregular that it was difficult to make a test sample of the appropriate size. If such a sample was obtained, the microscopic images were blurred due to the enormous difference in the height of the fracture. However, in the second case, the oars obtained as a result of injection had blisters, which also made it impossible to conduct a reliable static tensile test for this group of composites.

3. RESULTS

Figure 1 shows the TGA curves measured for the produced composite samples.
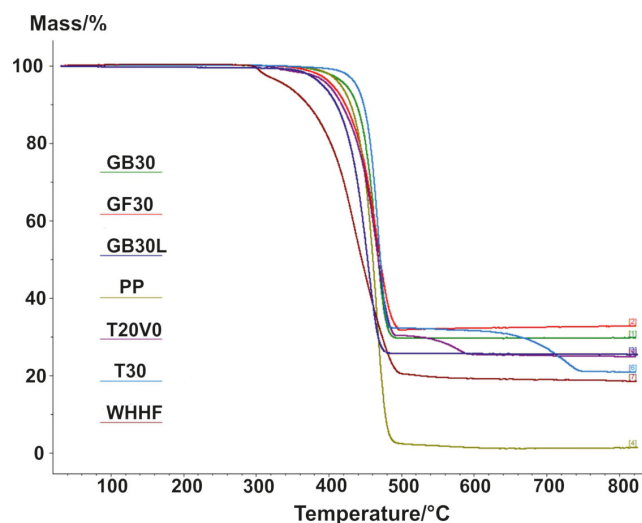

Fig. 1. TGA curves for the tested materials

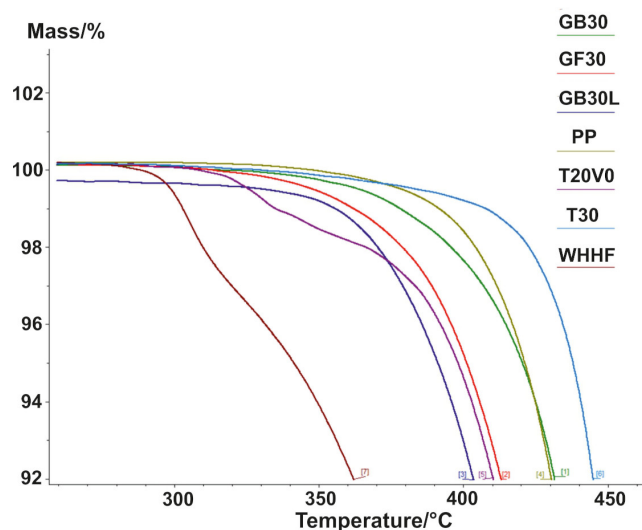Figure 2 shows a section of the TGA curves at the beginning of the thermal degradation of the tested materials.

Fig. 2. Approximation of TGA curves in terms of the onset of thermal degradation of the tested materials

Based on the course of the curves, the percentage weight loss was determined, divided into stages, and the temperatures at which this phenomenon occurred were determined. The results are presented in Table 2.

The first column of Table 2 presents the degrees of mass loss for individual transformations that took place in the tested polymer materials. The second column contains the temperature ranges in which the greatest mass losses occurred. The third column represents the temperature at which mass changes are most intense. This is the extreme of the first derivative of the mass change as a function of the temperature change.

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Table 2
Thermogravimetry analysis results

Material	Mass change, %	Temperature range, °C	DTG peak, °C	Residual mass at 850°C, %
PP	97.91	343.4÷541.7	466.4	1.43
GB30L	73.53	327.4÷490.1	453.9	25.48
GB30	70.26	323.8÷500.7	463.8	29.78
GF30	68.18	312.4÷507.3	467.5	32.83
T30	67.50 10.83	360.4÷502.8 583.7÷822.3	467.6 728.5	20.91
WHHF	3.63 76.24	276.4÷324.4 324.4÷507.8	305.7 434.5	18.53
T20V0	1.78 67.91 4.95	304.2÷360.1 360.1÷501.1 501.1÷609.2	329.9 466.8 577.7	24.88

The last column contains information about the ash or filler residue after the firing process at 850 degrees Celsius. In the analysis and presentation of the results, the first, small mass loss, corresponding to the moisture content in the tested material, was not considered.

The analysis of the results showed that the addition of any filler, except for talc, reduced the thermal stability of the tested materials. However, considering the first derivative, i.e. the dynamics of these changes, it can be noticed that their nature is different. The combination of the polymer matrix with any additive causes changes at the beginning of the thermal degradation of the tested materials. The additives affect both the mass and volume of the base material, which has a substantial impact on the degradation of the polymer matrix.

Based on the data contained in the table, it can be seen that the plastics containing the filler (T20V0 and WHHF composition) have a much lower temperature at the beginning of the degradation process than the polypropylene matrix from which the tested composites were made. For T20 V0, the beginning of the degradation process is observed at 304°C, while for WHHF it is already at 276°C. This is essential information from the point of view of usage and processing. In processing, one should not approach the temperature at which the plastic degradation processes occur, not only during plasticization but also during the flow of the material in the mold, where the temperature also increases due to friction. In other materials, i.e. composites with solid glass beads (first mass loss at 323°C), microspheres (first mass loss at 327°C), and fiberglass (first mass loss at 312°C), mass loss also occurs earlier, and so the beginning of the degradation process is comparable. Based on the obtained results, it should be stated that the mass loss process proceeded differently for individual groups of polymer composites based on polypropylene. In the case of a sample based on unfilled material and polypropylene composites with fillers in the form of glass beads and fiberglass, the process was conducted in one stage. In the case of the remaining polypropylene matrix composites, i.e. composites with talc and a flame retar-

dant, the mass loss process was conducted in several stages. It should be emphasized, however, that the mass loss extreme oscillated in a similar temperature range. This value for PP was 466.4°C, while the largest shift in this range was recorded for the sample with 30% flame retardant content, where the temperature at which the fastest weight loss occurs was 434.5°C. In the case of polypropylene composites with fillers in the form of glass beads and fiberglass, only the polymer matrix underwent thermal degradation, as evidenced by the value of the sample weight remaining after the test. However, in the case of composites with talc, thermal degradation of polypropylene occurred in the first phase, because it was at this stage that the loss was the largest, and at higher temperatures, talc was also partially degraded. On the other hand, in the samples containing the flame retardant, the weight loss in the first stage was small and amounted to approx. 3.63%. At this stage, the flame retardant was oxidized with the simultaneous release of gaseous products, which, according to the mechanism of its operation, are intended to deactivate free radicals, thus inhibiting the thermal decomposition (combustion) reaction. In addition, charred layers are formed on the surface of the material, which are designed to hinder the contact of the unburned part with the oxidant. Considering the large mass loss of 76.24% in the second stage, the polypropylene matrix and, to a lesser extent, the flame retardant degraded.

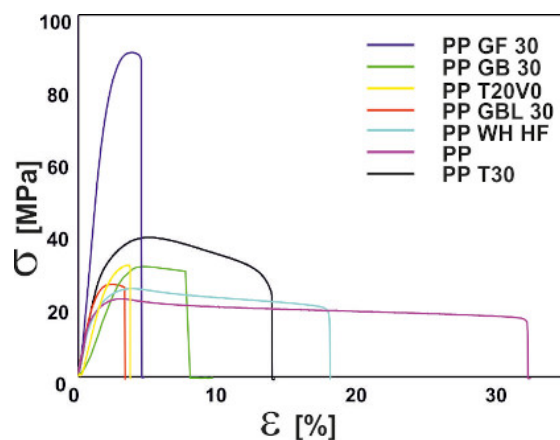Figure 3 shows an exemplary course of a static tensile test for a sample.

Fig. 3. Static tensile test diagram for tested samples

A series of five measurements was conducted for each composite. Figure 4 shows the cumulative results of the maximum stress and strain for the tested samples (maximum stress and maximum strain).

The stretch curves vary depending on the filler added. In the case of maximum stress, as expected, the highest values were recorded for PP reinforced with glass fiber. The lowest values were obtained for PP with the addition of talc (T30) and flame retardant (WHHF). Similar results were obtained for the remaining samples. In the case of deformation, the highest values (again as expected) were obtained for pure PP. For PP with the addition of talc or flame retardant, higher values were recorded than for the remaining samples.

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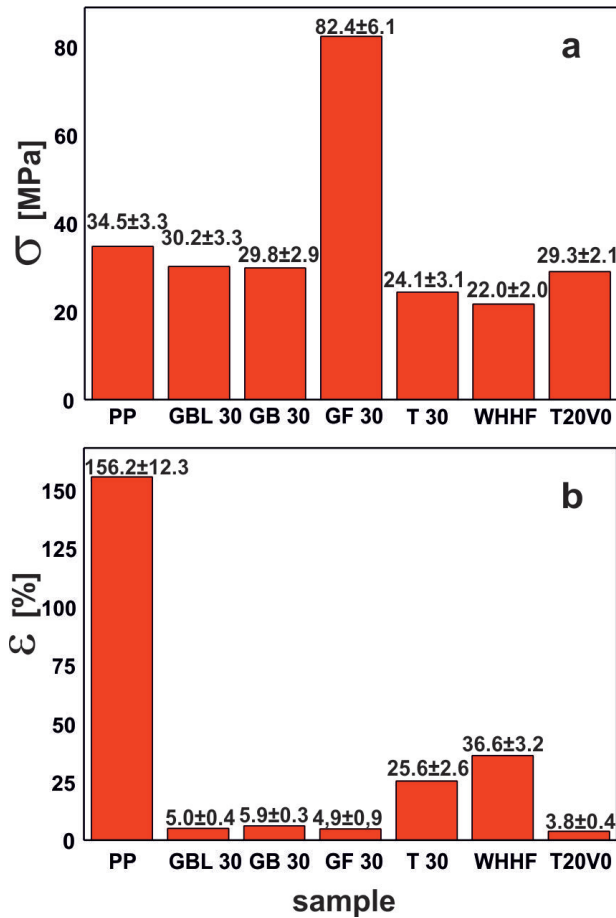Fig. 4. Average stress and strain values for the tested composite samples

of the fiber, there were also cracks in individual fibers of the filler itself. However, it should be noted that on the surface of the fracture, between the filler and the polymer matrix, there is a large adhesion. Single fibers adhere well to the polymer matrix, they are even "glued" with it. Thus, when comparing the fiberglass used with glass beads in this type of polymer com-
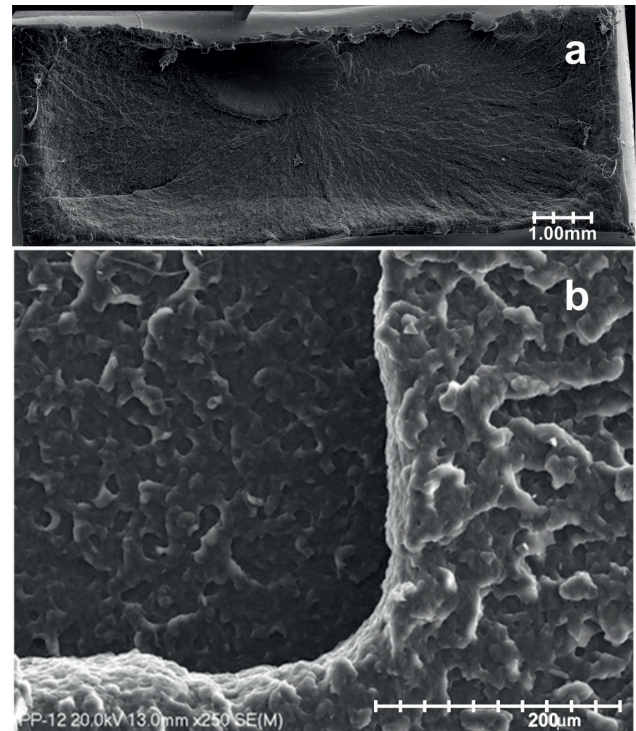

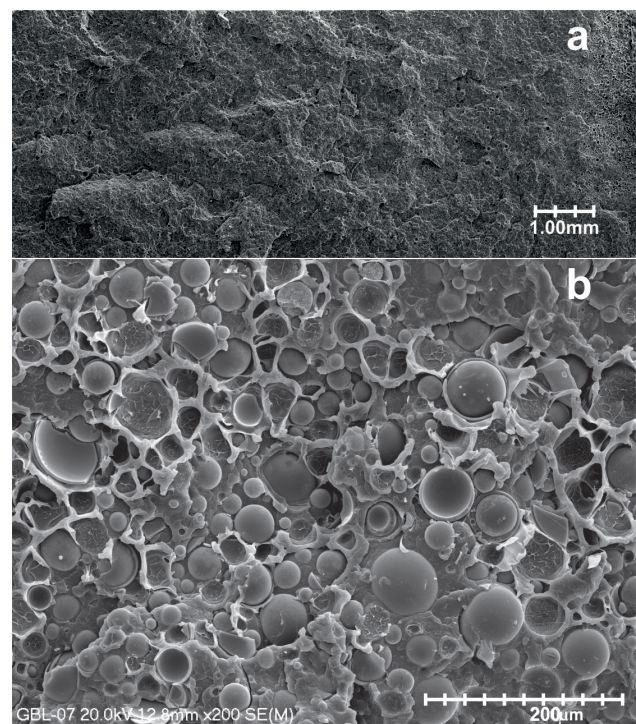Fig. 5. SEM image for a sample made of polypropylene

Figures 5–11 show SEM images for fractures of the tested samples.

The obtained images of the scanning microscope show that as a result of polypropylene modification with fillers in the form of glass beads, fiberglass, talc, and flame retardant, the obtained polypropylene composites present various structural changes, mainly related to the homogenization of the material with the filler and adhesion, i.e. one of the most desirable phenomena occurring in polymer composites. In the case of unmodified material, the surface of the fracture was smooth, only on the edge of the fracture, small "burrs" could be seen. In the case of polypropylene composites with solid glass beads and hollow microspheres, it should be noted that in most of the fracture area, ball "pockets" were formed, and the balls themselves were partially or completely detached from the polymer matrix. This proves the low adhesion between the polymer matrix and the filler. As shown in [24], weak bonding strength affects the macroscopic properties of samples. This can cause, among other things, a reduction in mechanical properties. Adhesion can be improved, among others, by modifying the physico-chemical properties of the filler or by modifying its surface. In addition, there were cracks in the glass beads, although more cracks can be seen in hollow microspheres. The situation is different for polypropylene composites with fiberglass. In the case

Fig. 6. SEM image for PP GBL 30 sample

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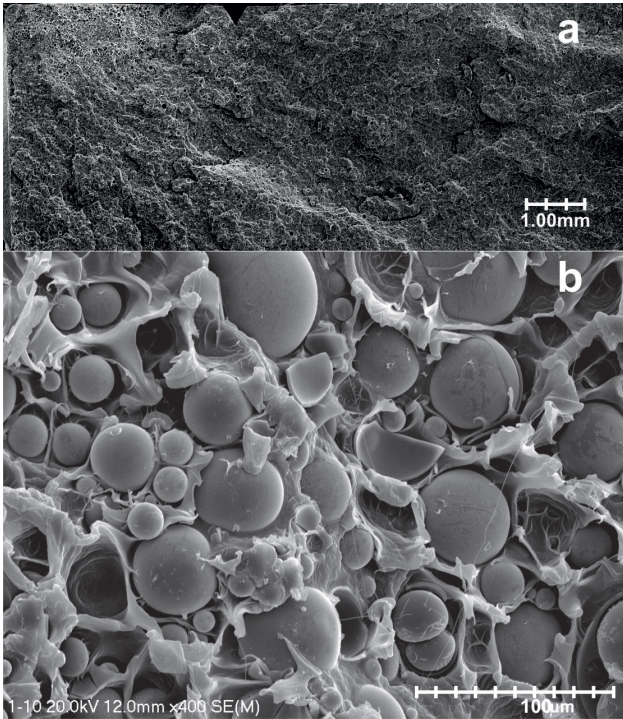

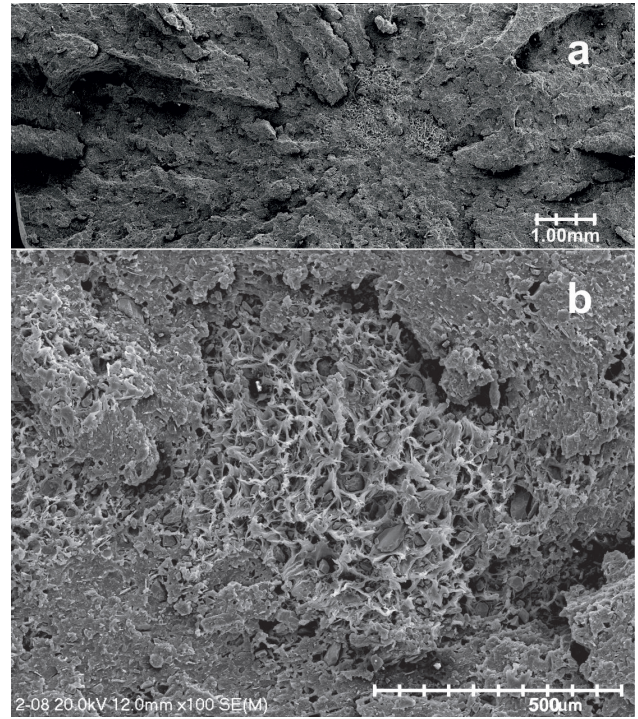**Fig. 7.** SEM image for PP GB 30 sample
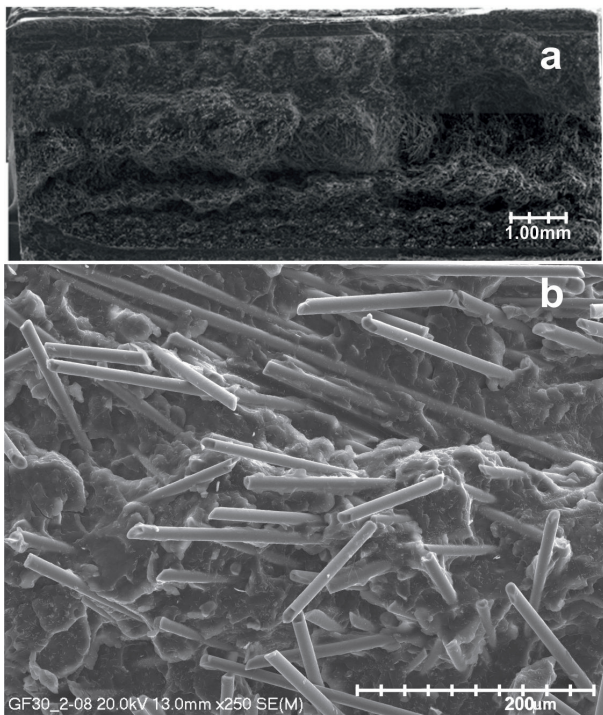

Fig. 9. SEM image for PP T 30 sample

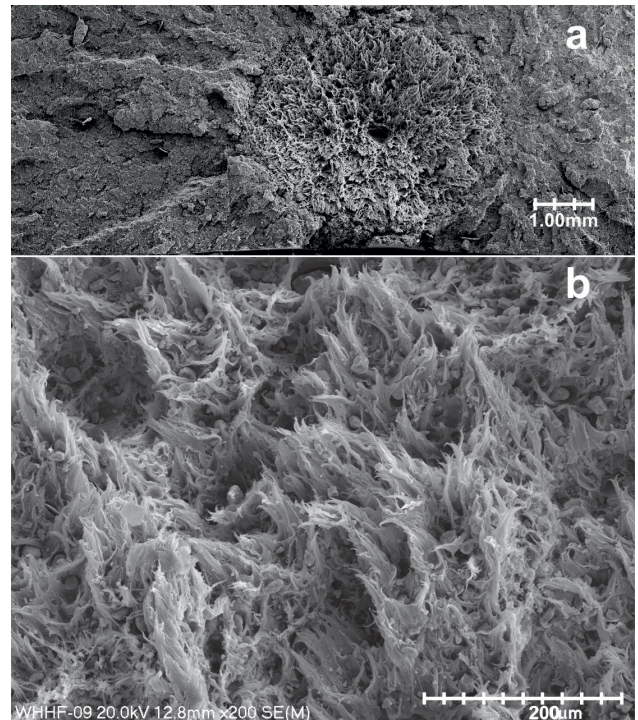**Fig. 8.** SEM image for the PP GF 30 sample

Fig. 10. SEM image of the WH HF sample

posite, it should be noted that in this case the structure of the composite in terms of homogenization of both substrates is determined by the shape of the filler, not its type and amount. In the case of composites with talc, a sample with 30% filler content was selected as a reference. The SEM images clearly show that the breakthrough is fragile. The mostly lumpy talc particles do not adhere to the fracture surface, in some areas even pockets of lumps are visible. In these areas, the material was most likely less brittle. It can therefore be concluded that the homogenization of the material did not occur uniformly within the entire compact. Adhesion in this case, as in the case of glass beads, was not high. Breakthroughs of composites with 30% flame re-
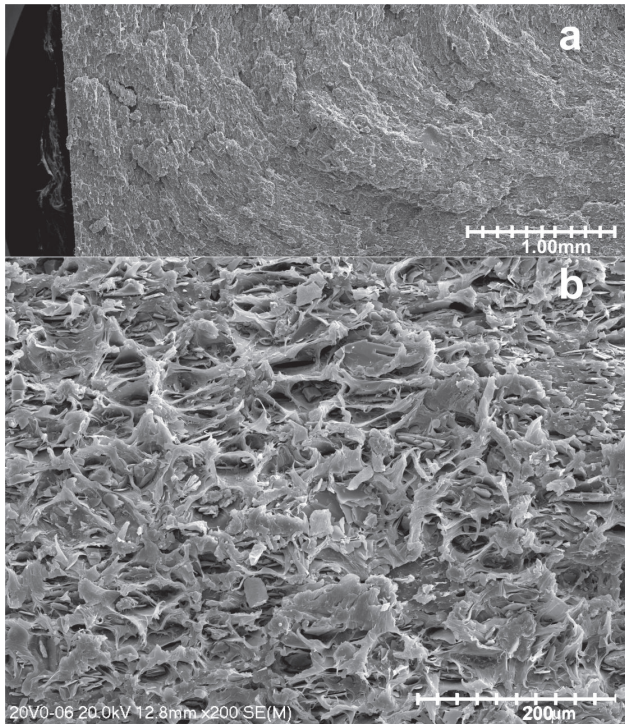
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Fig. 11. SEM image of the T20V0 sample

tardant content of lump and partly lamellar structure look similar to talc. In this case, too, the fracture is mostly brittle, but there is an area where the material is more flexible. However, unlike the talc composites, where less brittle areas were distributed irregularly on the fracture surface, in this case, they were concentrated in the centre of the sample. Also, in the case of these polypropylene composites, the adhesion between the polypropylene matrix and the filler was not particularly strong. The material also did not homogenize well with the filler, which was also seen during the static tensile test. The fractures were very irregular and jagged.

4. CONCLUSIONS

The research aimed to demonstrate the influence of the type of filler on the thermal and mechanical properties of composites based on polypropylene. Several types of filler were used for the tests. The results were related to the results obtained for pure polypropylene. One of the prominent features of this type of composite is its thermal resistance. For this purpose, fillers are added to the polypropylene volume to increase their thermal resistance, for example by reducing flammability. The obvious addition is a flame retardant. On the other hand, it is important to maintain the mechanical properties at an appropriate level. For this reason, the samples were subjected to a static tensile test and thermogravimetric tests. The obvious result is that the tensile strength value is several times higher for the samples with the addition of fiberglass. For samples with the addition of glass beads, the results were similar to those of pure polypropylene. Comparable results were obtained for the following series of samples: with the addition of talc, flame retardant, and a mix-

ture of talc and flame retardant. The tests showed that the type (material) of the filler alone had a relatively small effect on the mechanical properties of composites based on polypropylene (this effect was observed only for fiberglass, which is an obvious effect related to the shape of the reinforcement). The results of mechanical properties tests were supplemented with SEM images. The analysis of the images taken for the fractures confirmed the influence of the shape of the filler on the mechanical properties of the fiberglass ("adhesion" of the polypropylene matrix to the fibers is visible). For the remaining samples, no increase in adhesion was found after the addition of fillers. The introduced fillers in the shape of spheres (glass beads), nuggets (talc) as well as nuggets and plates (flame retardant) did not significantly change the mechanical properties of the composites.

On the other hand, thermogravimetric tests showed significant differences in relation to the filler used. By far, the best thermal properties were obtained for the sample with the addition of a flame retardant. In this case, the flame retardant oxidized at a temperature of about 365°C, which resulted in the release of gaseous products and inhibition of the thermal decomposition reaction. In the further stage of the experiment, the polypropylene matrix and the flame retardant remaining in it were degraded. A similar effect was obtained for a composite sample containing 20% talc and 6% flame retardant. In this case, the weight loss mechanism was similar. The oxygen index was calculated for the tested samples. As is known, for an index above 21, the combustion process of the material should not occur after eliminating exposure to flame and heat. In the case of the tested samples, OI index values above 21 were achieved for samples with 30% flame retardant content (PP WHHF), and 6% flame retardant content with 20% talc content (PP T20V0) [2]. Hence the conclusion is that it is possible to improve the thermal properties without a significant deterioration of the mechanical properties by a relatively small addition of an expensive flame retardant (6%) and a cheap filler such as talc (20%).

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