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The Influence of the Aging Process on the Change of Selected Strength Properties of Polypropylene Compositions with Mineral Fillers

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ABSTRACT

The goal is to understand the influence of minerals fillers on the course and performance of process of injection molding polypropylene compounds as well as on the mechanical properties of the obtained products. Three types of mineral fillers, derived from post-production waste, were used for testing. It was aluminosilicate (zeolite), fly ash and gypsum powder, all in powder form. The minerals fillers were introduced into the tested PP in a mechanical mixing process prior to the processing. During the injection molding process, inorganic fillers are subject to the same steps as plastic processing, compression, homogenization, transport. Organic fillers used in the injection process were introduced into the processed PP in the amount of 30% by weight. The test stand consists of a screw injection molding machine, Arburg AllRounder 320C. The research on the structure of manufactured materials, mechanical strength, impact resistance and hardness are presented. The laboratory tests of accelerated aging were conducted using an aging chamber. The aging temperature in the heat chamber was set to even amount of 63 °C and irradiance 0.51 W/m^2. According to the standard, the aging time has been applied accordingly: 120, 240, 360 h, which conform to degradation at room temperature for 4 month, 8 month and one year. It was found that the type of mineral fillers used did not have a significant influence on the hardness of the surface of the moldings. The changes in hardness shown in the figures are primarily influenced by the properties and type of polymers used during the injection process. During the tests, differences in the mechanical strength of composites for injection molded parts made of PP with mineral fillers were observed. Filling PP with zeolite in the tested value causes a decrease in mechanical strength by an average of 10% from 24 to 21.6 MPa. Different mechanical interactions are shown by fly ash and gypsum powder fillers, increasing mechanical resistance of the composition. Fly ash increases mechanical strength by 30% on average, from 24 to 31.2 MPa. In case of gypsum powder application the resistance of PP composition increases analogically, but on average 20%, to 29.5 MPa.

Keywords: polymer compositions, polypropylene, zeolite, fly ash, Gypsum Powder, accelerated aging, strength properties

INTRODUCTION

The significant development of plastics processing in recent years has resulted in an increase in the amount of technological and post-use waste. European legislation requires all members of the European Union to strictly follow the rules which aim, is to develop modern waste-free technologies, limit the consumption of natural resources and energy and minimize the amount of waste collected in landfills. This increases interest in recycling of waste mineral products and recycled products made of plastics [1]. This is also due to economic and legislative reasons and to the reduction of the negative impact of mineral waste on the environment. Such mineral wastes include gypsum powder, waste ash, natural zeolites. Modifying polymers is aimed at giving the products

the desired properties or facilitating their processing [3]. The modification process is characterized by a change in the technological conditions of the production process, as well as by adding auxiliary materials to the polymers including fillers or plasticizers. Aids are split into two groups such as functional and processing [11, 12, 24]. The results presented in the studies [10, 12, 20] show that the mechanical properties of the fillers simplify the flow of the polymer during its processing. However, they do not measure whether the fillers have any effect on the efficiency or effectiveness of the process. The authors [8, 9] dealt with the extrusion process in terms of decomposition, mechanical properties of High Density Polyethylene (HDPE) / Polypropylene (PP)and Polyvinyl chloride (PVC) composite products [25].

Gypsum powder, of which calcium sulphate is the main component, is waste from the manufacture of phosphoric acid. The growing demand for minerals containing phosphorus causes constant formation of massive amounts of gypsum powder - a by-product, which is an integral part of the production of phosphoric acid and is very problematic in terms of its management. Despite the high content of dihydrogen gypsum in gypsum powder (reaching about 95%), Gypsum powder is not used as a substitute for natural gypsum in the building materials industry. [4, 21]. The analysis of the acquired knowledge allows to state that calcium sulfate in the form of gypsum powder can be used as a filler and modifier of thermoplastic polymer composites [20, 21]. Calcium sulfate introduced into the material, similarly to mineral fillers (e.g. talc), increases strength, hardness, abrasion resistance, reduces flammability and reduces processing shrinkage of the obtained polymeric composites [1]. According to the authors, the addition of gypsum powder to LDPE causes an important increase in the material bending strength and decrease in elongation at break [19, 21, 24]. Although economic analyses of the use of new polymeric compositions with the use of recycled materials, such as gypsum powder, indicate that the obtained compositions and products are not cheaper and better at the same time, they are often characterized by unique properties (porosity, vibration damping, biodegradability) justifying their use [4, 19, 20, 21].

Zeolites are a mineral group with a specific internal structure (pores and channels) in which water particles are present. As a result of adding zeolite to mineral mix together with asphalt, the water contained in the pores of those minerals is released. As a result of zeolite water evaporation, volumetric expansion of the binder occurs, the effect of which is asphalt foaming and decreasing in its viscosity [18, 29]. Thus the workability of mix asphalt and asphalt adhesion to aggregate at lower temperatures increase. The introduction of zeolite into the material, similarly to talc, increases the strength, hardness, abrasion resistance and reduces processing shrinkage of the polymeric composites obtained [31]. The addition of gypsum powder to HDPE causes an increase in material tensile strength and significant decrease in elongation at break [3, 21, 22].

Waste fly ash, most often perlite ash is cumbersome are expensive to store [7]. At present, this waste is very little recycled. The nominal grain size of waste perlite dust is similar to that of quartz flours, most often used as microfillers in concrete and resin compositions, polymeric building composites, which is a prerequisite for the attempts to use perlite dust as a component of building composites [8]. The number of literature information on this subject is small [9-11]; mineral additives of this kind were considered for a long time to be poorly compatible with polymeric binders [12, 23].

The research objective of the work is to understand the influence of the mineral filler on selected mechanical properties in modified polypropylene compositions subjected to the thermal aging process. The application purpose of the work is to determine the possibility of using an appropriate type of mineral waste for the production of products from a polypropylene composition with appropriate mechanical properties. Three different types of mineral fillers from production waste were used in the research.

EXPERIMENTAL

During the tests, composites plastic, polypropylene (PP) Propolder FPP2040, manufactured by TWO H Chem's Company, was used. According to the manufacturer's data, PP in the form of powder characterized by a white color. It has the following properties: MFR (230^{o}C, 2.16kg): 10-25 g/10 min; tensile strength: 30-33 MPa; yield point: 33-36 MPa; Young's modulus: 1200-1300 MPa; elongation: 10-15%. PP powders are available in a wide range of average particles sizes from 5-300 μm.

According to the research programme, three types of mineral fillers from waste were used. These were aluminosilicate - zeolite (ZL), fly ash (FA) and gypsum powder (PG), in a powder form.

In this study the synthetic zeolite NaP1 produced from fly ash in the process of hydrothermal synthesis was used. The quantitative content of zeolite minerals in NaP1 material was about 80% by volume. The mineral composition of zeolite fillers is supplemented by small amounts of mullite, quartz and aluminosilicate glaze [31]. Zeolite NaP1 represents the distribution of zeolite particles to a maximum of 25 μm.

Another mineral filler is silica fly ash, which is a by-product of hard coal combustion [28]. Fly ash according to PN-EN 450-1 is a fine-grained dust, consisting mainly of spherical glazed grains of particle size (on average 20-40 μm) and very low bulk density (90-100 kg/m^3) obtained when burning coal dust by electrostatic or mechanical separation of dusty particles from power plant waste gases. Gypsum powder used was a by-product from phosphorous fertilizers' industry and was treated by method elaborated in Poland [22].

The main component of, according to the data of the distributor, Permedia S.A., is calcium sulfate ($CaSO_4$) formed in the reaction of sulphuric acid with phosphorites or apatites. It contains numerous admixtures of elements, mainly phosphorus (P), sodium (Na), fluorine (F), aluminium (Al), iron (Fe), silicon (Si) as filler causes related changes of mechanical properties [16, 17, 27].

The minerals fillers were introduced into the tested PP in a mechanical mixing process prior to the processing. During the injection molding process, inorganic fillers are subject to the same steps as plastic processing, compression, homogenization, transport. The inorganic fillers set in the injection molding process were introduced into PP processed in an amount 30% by mass [32].

The research stand contain a screw injection molding machine, Arburg 320C. The machine consists of a single-screw plasticizing system, with diameter of 30 mm. In the tests, the temperatures of the compositions tested were inspected were positioned in the particular heating zones of the plasticizing system: in zone I -180°C, II – 190°C, III – 200°C, IV – 210°C, V – 230°C. The following injection molding process parameters were adopted: injection and crimping time = 2-3 s; mold cooling time = 30 s, thermostated mold temperature = 19 ± 1°C.

Observations of the obtained mouldings were made in transmitted light on a Nikon ECLIPSE metallographic microscope, which enables direct viewing of the image on a computer screen. As part of the research program, tensile strength and elongation at break were performed. Mechanical properties were tested on the Zwick / Roel Z010 machine in accordance with the PN-EN ISO 527-1:1998 standard [33]. The strength properties of moldings subjected to static stretching were determined on a Zwick Z010 testing machine. The measurements were performed at a tensile speed of 10 mm / min. The test parameter were as follows: test speed 50 mm/min., 10 measurements were made for each samples. Impact tests were carried out with a Charpy hammer. The hammer has an basic energy of 5 J. The tests were conducted in agreement with ISO 179-1:2010 [34]. The Shore hardness tests were carried out using the hardness tester Affri ART13 – Shore'a D method. This choice resulted from the method used to determine the hardness of stable polymers materials [35]. The shape and dimensions of the samples used were in accordance with the relevant standard. The thickness of the sample corresponded to the thickness of the part and was measured and recorded each time together with the width of the measuring section before the tests.

Laboratory tests of accelerated aging were carried out in an aging chamber of the HD-E711 type. According to the PN-EN ISO 4891:2013 standard, the aging temperature in the thermal chamber was 63 °C, and the irradiance was 0.51 W/m^2 [5, 6, 13, 26, 36]. Aging tests were performed for the following steps: 120, 240 and 360 hours.

RESULTS AND DISCUSSION

The photographs of the cross section of the composite research samples were taken on the stand for image analysis of cellular plastics that consisted of metallographic microscope equipped with a digital camera and a personal computer with suitable software.

During the research, a clear influence of the type of mineral fillers and the characteristics of the influence on the obtained morphology of porous moldings was observed. The PP+30%ZL (Fig.1a.) composition has a homogeneous structure in which spherical zeolite grains predominate. Zeolite, having grains with a diameter of 20-50 μm, forms a composition with PP, with

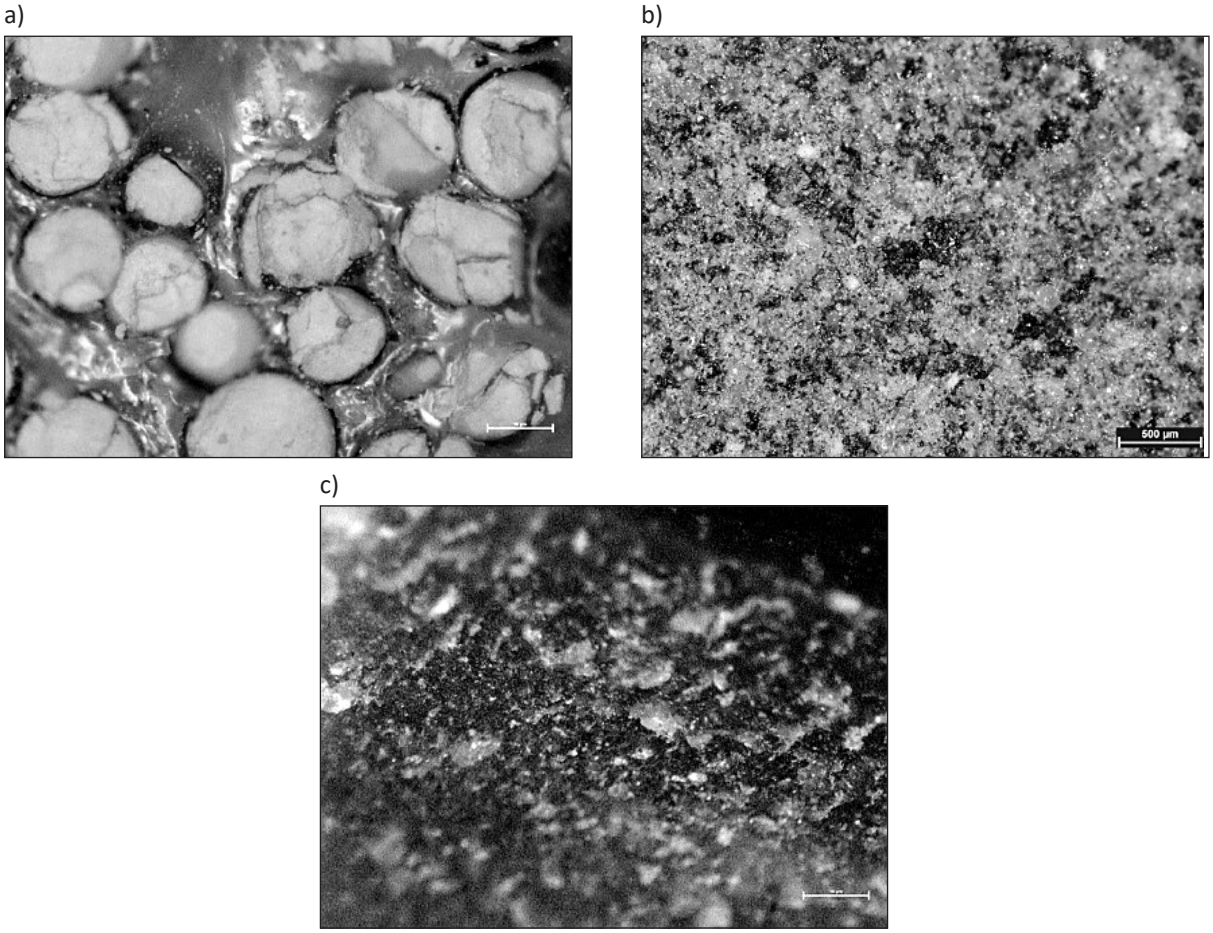

Fig. 1. Fragment of the cross section of the a) PP + 30% ZL (zeolite), b) PP + 30% FA (fly ash), c) PP + 30%PG (gypsum powder)

adjacent grains (Fig.1b). PP+30% FA composition has a structure with visible, linearly distributed spherical and oval layers of FA grains, black, 2-4 µm in size.

The compositions of PP + 30% PG have a structure with visible flakes of gypsum powder, distributed unevenly over the entire cross-section of the tested sample gypsum powder flakes are 2 to 10 µm long (Fig.1c). The obtained results of the determination of selected mechanical properties of PP composite moldings, gained with different contents of mineral fillers in the processed polymer materials, are shown in Figures 2-6 and Table 1-2. The direct parameters were measured 10 times for each sample type. Overall, the results were largely the same or similar.

Obtained results of the hardness of the moldings are shown in Figure 2. The type of mineral fillers used does not change the surface hardness of the moldings. The changes in hardness presented in the figures largely depend on the type and properties of polymer materials used in the injection process. The change in zeolite hardness and PP fly ash is 1-3°Sh and is related to the type of PP used and technological quality of the process, and not to the type of mineral fillers.

Only in the case of PP+30% PG compositions the surface curing after thermal aging was found. The increase in hardness is, on average, 5% (5°Sh), and this is, however, a different tendency from the other mineral fillers used.

The applied test method was the ball indentation method, in accordance with the PN-EN ISO 2039-1:2004 standard. The HPK 841 hardness tester was used for the test. Measurements were carried out under constant ambient temperature conditions:, $T_o = 25 \pm 3$ °C air humidity 50 ± 10%. The hardness measurement method using the ball indent principle is based on the measurement of the plastic deformation depth measured under the influence of the acting load. The ball pressing method performed on the Brinell apparatus is originally a type of measurement dedicated to the hardness measurements of metals. It was also adopted after measuring the hardness of a group of materials

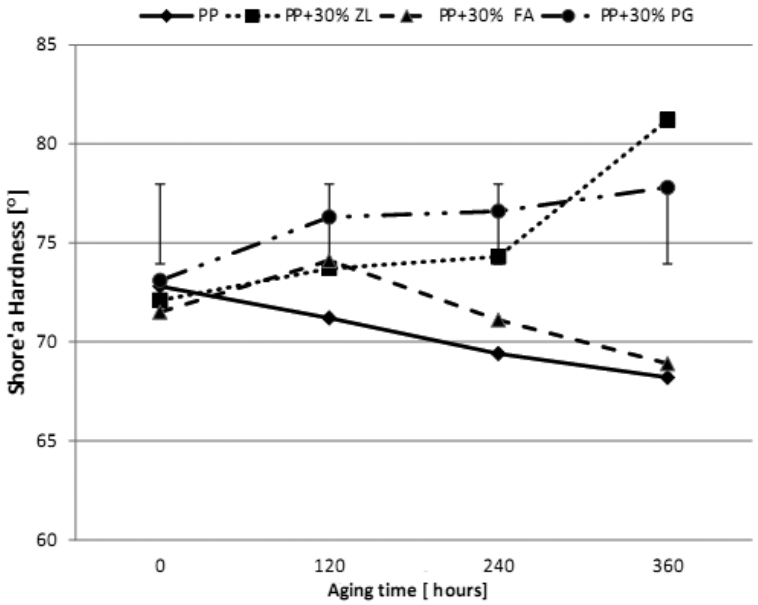

Fig. 2. Dependence of hardness of injection molded part examinated with Shore's method on the aging time and type of polymer composition

such as ceramics and polymer materials. The essence of the method is to slowly force an indenter (5 mm) with high hardness into the tested material, with an appropriate measuring load, in the case of the tests presented in the article the load was 357 N.

It was investigated that the aging process reduces the mechanical resistance of the surfaces of the tested compositions. It was also noticed that the time of the aging process changed the resistance of the composition in a non-linear, non-harmonious way. This is most noticeable in PP compositions filled with fly ash (PP+30% FA) and PP compositions with gypsum powder (PP+30% PG), where the aging process initially reduces the surface resistance, but then, after a longer time of thermal aging, increases the resistance. mechanical surface by 18 and 14%, respectively. Perhaps, as a result of thermal aging, the surface hardens, but at the same time the brittleness of the tested compositions increases, but it was not the subject of the conducted research and requires further research. Analyzing the obtained results, it was found that the hardness measurements carried out by the ball indentation method reflect the changes in the surface hardness of the tested polymer compositions in a different way. The use of a ball-shaped indenter in this method results in a more accurate determination of changes in hardness caused by the type of tested material and the method used to determine the aging of the materials.

To determine the preferred mechanical properties PP composites, tensile strength tests, yield strength, young modulus and strain at maximum stress were carried out in compatibility with the relevant standards (ISO 868:2003 and ISO 527-1:2010). The thickness of the sample corresponded to the thickness of the injection molded part and was measured each time along with the width of the measurement length. The results of determination of selected mechanical properties of PP composites parts, obtained at different minerals fillers in the processed polymers, are presented in Table 1 and Figures 3-6.

Considering the obtained results of the σ_m value, it was found that the addition of mineral fillers and the aging process had an impact on the tensile strength of the tested samples. A significant decrease in σm of PP samples subjected to aging tests in the UV chamber was found. After 360 h, the decrease was 46% relative to the unaged sample. In the case of PP samples mixed with zeolite, the decrease was not as high and amounted to 29%. The addition of post-production waste in the form of fly ash (FA) caused a decrease σ_m of from 31.2 MPa to 16.9 MPa for the samples staying 360 h in the aging chamber, which is equal to a 46% decrease as in the case of PP samples. The aging process also had a negative effect on the mechanical strength of the samples with the addition of gypsum powder (PG), after 360 h in the UV chamber a decrease in σm by nearly 14 MPa was shown. On the basis of these tests, it can also

Table 1. Research results of the mechanical properties of the injection molded parts of PP composites, before and after aging

Materials type	Time of aging, h	Ultimate strength σ_m, MPa	Tensile strength σ_B, MPa	Yield strength σ_y, MPa	Youngs modulus E, MPa	Strain at maximum stress ε_m, %
PP	0	24	21.8	24.5	1290	7.6
	120	21.2	18.8	19.4	1270	7.8
	240	17.3	16.3	14.5	1240	8.3
	360	12.9	11.7	13.1	1200	7.6
PP + 30% zeolite	0	21.6	21.5	23.4	1660	3.3
	120	18.6	18.3	18.7	1660	4.1
	240	17.8	15.9	16.1	1680	3.4
	360	15.4	10.9	12.3	1610	3.3
PP + 30% fly ash	0	31.2	29.4	31.4	1470	7.8
	120	26.2	23.4	23.5	1490	7.9
	240	21.3	18.8	19.2	1450	6.5
	360	16.9	14.1	15.9	1380	6.3
PP + 30% gypsum powder	0	29.5	27.9	29.4	1620	4.7
	120	24.6	21.6	21.2	1610	4.9
	240	20.5	17.4	18.2	1600	4.6
	360	15.8	14.7	15.7	1560	4.3

be clearly stated that the addition of mineral fillers to the material increases their tensile strength. These samples, both before and after the aging process, showed higher σm than PP without additives (Fig. 3).

The use as a filler of the zeolite results in a slight change between the mechanical strength determined by ultimate strength and yield strength, immediately after the production of test samples of about 0.1 MPa, from 21.5 to 21.6 MPa. However, aging after 360 h results in a decrease in yield strength by 11%, from 12.3 to about 11 MPa. When used as a fly ash filler, the situation is similar. Immediately after the production of test samples, the difference between ultimate strength and yield strength is only 2 MPa. After aging, after 360 h, yield strength is reduced by 12%, from 15.9 to about 14 MPa. A similar character of the change in ultimate strength and yield strength occurs with gypsum powder. However, gypsum powder fillings cause, after 360 h aging, a decrease in yield strength of PP + 30%

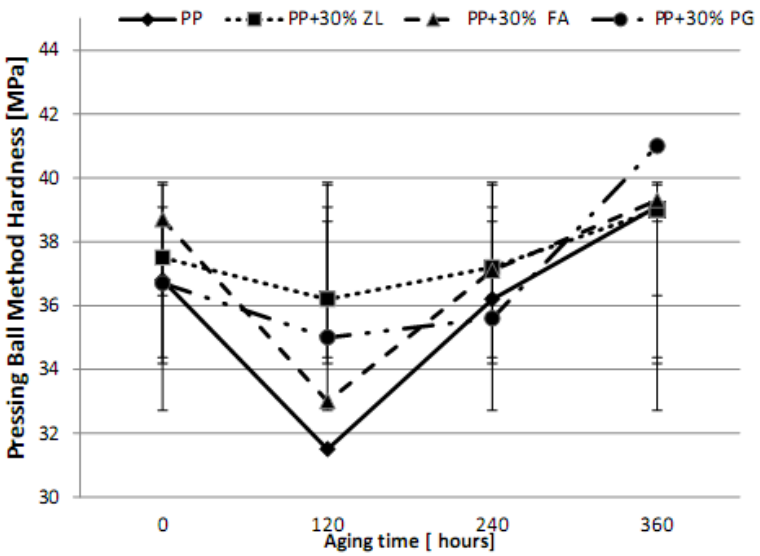

Fig. 3. Dependence of hardness of injection molded parts examined with Pressing Ball method on the aging time and type of polymer composition

PG composition, almost by 15 MP, i.e. by 50%. A similar relationship exists for ultimate strength, which is reduced by about 43% (Fig. 4).

The influence of mineral additives and the aging process were also analyzed for the Young's Modulus (Fig. 5). The E value of the non-aged PP sample was 1290 MPa and it decreased by nearly 90 MPa in relation to the samples aged 360 h. Much higher values were found for the samples with the addition of zeolite. The values ranged from 1660 MPa (without aging) to 1610 MPa after 360 h, which is a decrease of only 4%. However, it is worth noting that these values did not increase. The greatest change in E was shown by the tested samples with the addition of fly ash, their value in relation to the samples not subjected to aging tests decreased by 90 MPa. As in the case of other samples, the value of Young's Modulus for samples with the addition of PG decreased. From the value of 1620 MPa to 1560 MPa.

The results of the Charpy impact tests are presented in Table 2, and to better illustrate the changes in Figure 6, as the connection between the impact strength and the type of fillers tested before and after aging. After the aging process, the toughness of the samples increases significantly.

CONCLUSIONS

It was found that mineral fillers contained in PP composites cause a significant decrease in the

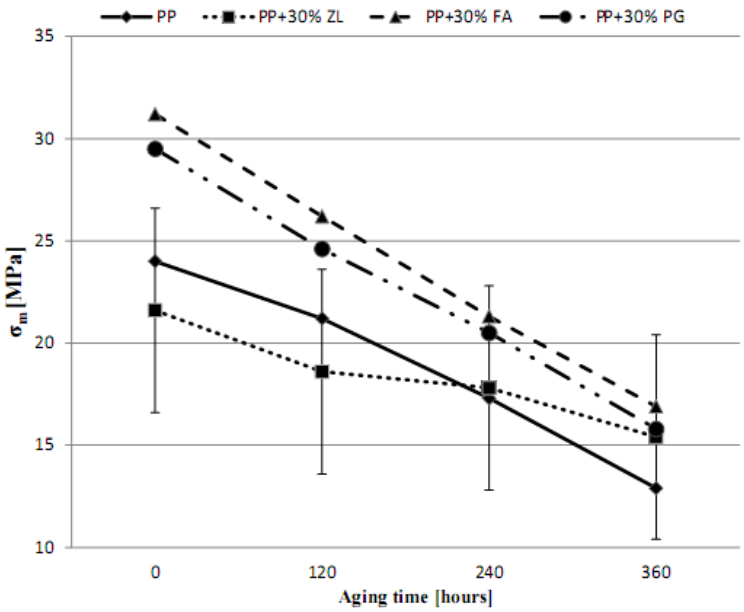

Fig. 4. Impact of aging time on tensile strength of tested composites

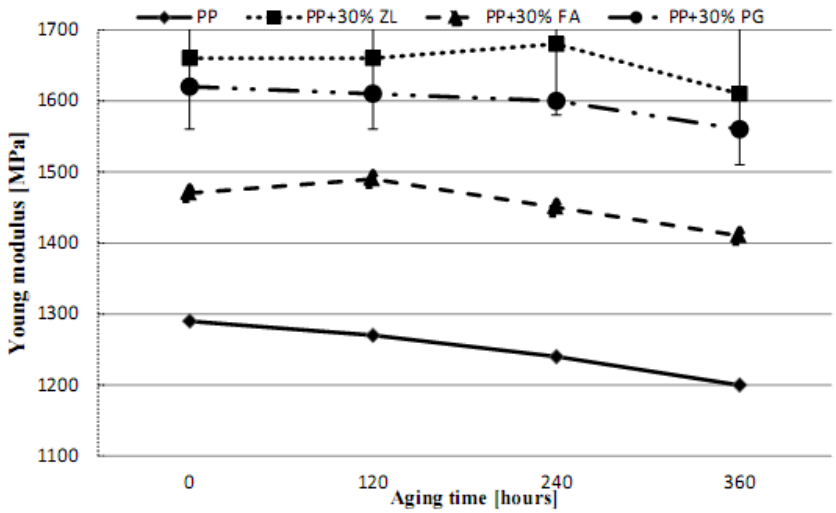

Fig. 5. Impact of aging time on the Young's modulus of tested composites

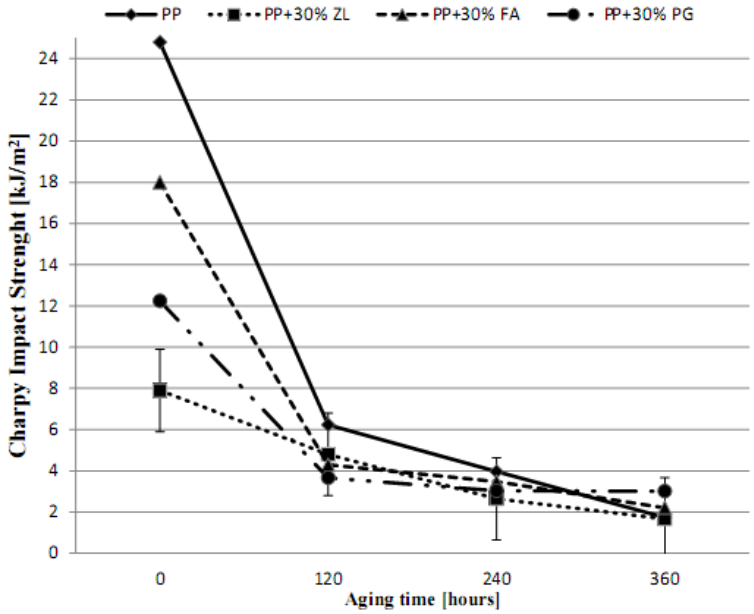

Fig. 6. Dependence of impact strength parts of PP composites on the aging time and type of menerals fillers

Table 2. Research results of the Charpy impact strength parts of PP composites, before and after aging

Materials type / Time of aging, h	Charpy impact strength, kJ/m²			
	0	120	240	360
PP	24.8	6.23	3.96	1.73
PP + 30% zeolite	7.90	2.47	1.64	1.67
PP + 30% fly ash	18.00	2.63	3.47	5.32
PP + 30% gypsum powder	12.24	3.66	3.03	3.01

impact resistance of the material. This change ranges from 25% (fly ash), 50% (gypsum powder) to 67% (zeolite). Aging of the tested composites in the range 120-360 h causes a rapid decrease in impact resistance of the composition. The resistance of PP decreases even 8 times, from 24.8 to 1.73 kJ/m^2. Resistance of minerals fillers also decreases rapidly to 1.67–3.01 kJ/m^2, i.e. by 80% zeolite, 87% fly ash and 75% gypsum powder respectively.

The mechanical strength of the PP filled mineral fillers presented in the paper depends to a large extent on the type of filler used. A significant effect of aging process on the properties of the PP compositions studied was also found. The type of mineral fillers used does not significantly affect the surface hardness of the moldings. The variation in hardness depends mainly on the properties and type of the tested polymer materials. The change in the hardness of zeolite and fly ash PP is related to the type of PP used and technological conditions of the process, and not to the type of mineral fillers.

Important differences in mechanical strength of PP injection molded parts of composites, with minerals fillers, can be observed. For example, filling PP with zeolite, in the tested value, results in an average 10% reduction in mechanical strength. Different mechanical interactions are shown by fly ash and gypsum powder fillers, increasing mechanical resistance of the composition. Fly ash increases mechanical strength of PP composition by 30% on average, while filling PP gypsum powder increases mechanical strength by 20% on average.

Zeolite causes a slight change between the mechanical strength determined by ultimate strength and yield strength. However, aging after 360 h results in a yield strength reduction of 11%. When used as a fly ash filler, the situation is analogous, after 360 h aging, the yield strength is reduced by 12%. A similar character of the change in ultimate strength and yield strength occurs with gypsum powder. However, after 360 h aging, the yield strength of PP compositions filled with gypsum powder decreases by almost 50%.

A similar correlation occurs for ultimate strength, which is reduced by approx 43%.

It was proved that mineral fillers contained in PP composites cause a significant decrease in the impact resistance of the material. This change ranges from 25% (fly ash), 50% (gypsum powder) to 67% (zeolite). Aging of the tested composites in the range 120–360 h causes a rapid decrease of impact resistance of PP composites even 8 times. Resistance of the compositions filled with minerals fillers also decreases by 80% (zeolite), 87% (fly ash) and 75% (gypsum powder) respectively. The strength properties discussed in the article largely depend on the properties of the mineral fillers used. This is mainly due to the thermal properties of the polymer material used and the effect of mineral fillers on the polymer material used in the process. However, this connection has not yet been thoroughly considered and therefore will be the topic of further research.

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