

PA 10.10 and PA 6 composites with glass or basalt fiber: mechanical properties in dry and wet state

Paulina Romańska^{1),*} (ORCID ID: 0000-0002-7133-8522), Stanisław Kuciel¹⁾ (0000-0001-9244-7499)

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Abstract: The effect of basalt and glass fibers and moisture on the impact strength, tensile mechanical properties and heat resistance of PA 10.10 and PA 6 was examined. It was shown that PA 10.10 composites can compete with PA 6 composites in terms of mechanical properties, in a humid environment. Their heat resistance was lower than that of PA 6 composites. Regardless of the type of polyamide, composites with glass fiber had better properties than those with basalt fiber.

Keywords: biopolyamide, short fibers, water absorption, mechanical properties, heat resistance.

Kompozyty PA 10.10 i PA 6 z włóknem szklanym lub bazaltowym: właściwości mechaniczne w stanie suchym i mokrym

Streszczenie: Zbadano wpływ włókna bazaltowego i szklanego oraz wilgoci na udurowość, właściwości mechaniczne przy rozciąganiu i odporność cieplną PA 10.10 i PA 6. Wykazano, że kompozyty PA 10.10 mogą konkurować z kompozytami PA 6 pod względem właściwości mechanicznych, głównie w środowisku wilgotnym. Ich odporność cieplna była niższa niż w przypadku kompozytów PA 6. Niezależnie od rodzaju poliamidu, kompozyty z włóknem szklanym miały na ogół lepsze właściwości niż z włóknem bazaltowym.

Słowa kluczowe: biopoliamid, krótkie włókna, chłonność wody, właściwości mechaniczne, odporność cieplna.

In polyamides (PA) produced on an industrial scale from derivatives of fossil fuels, short-chain ones predominate, PA 6 and PA 6.6. Petrochemical long-chain PA 12 and PA 12.12 are also well-established on the market. Nonetheless, there is a property gap between PA 6.6 and PA 12. In recent years, the gap is being filled by partially or completely bio-based, long-chain polyamides, such as: PA 5.10, PA 6.10, PA 10.10, PA 11, PA 10.12 (and others). Among them, PA 10.10 outstands as it is fully biobased and produced in accordance with green chemistry principles.

A substantial number of scientific papers is devoted to PA composites but only a small part of it concerns composites with fully biobased matrix. Despite the small number of publications, research on PA 10.10 or PA 11 composites covers various thematic areas and concerns several types of reinforcements, including: nanocomposites, natural fiber composites, composites with chopped synthetic fibers and hybrid composites [1–19].

Nanocomposites of PA 10.10 and PA 11 investigated by different researchers exhibited significant increase in mechanical properties and enhanced thermal stability or barrier properties and aging resistance comparing

to unfilled matrix [1–4]. For low nanofillers content the authors observed a nucleating effect upon the polymers. By obtaining an exfoliated structure for PA 11 composites with montmorillonite content of less than 4 wt%, Liu *et al.* improved the thermal stability of the composite by 20°C compared to the unfilled matrix, and the main positive result of the modification was an increase in tensile strength by approximately 40% and an increase in the elastic modulus by over 80% [1].

Other research groups addressed the usefulness of the biopolyamides as matrices for natural fiber composites [5–10]. Although the processing of plant fibers with PAs is problematic because of their melt temperatures, successful attempts have been made to produce excellent quality biocomposites. Research by Błędzki *et al.* concerned composites based on PA 10.10 and PA 6.10 filled with regenerated cellulose fiber or abaca fiber (15–30 wt%). The addition of fibers resulted in a significant improvement in the static mechanical properties of biopolyamides, as well as an increase in the impact strength for semi-synthetic cellulose fibers [5, 6]. Such beneficial effects were achieved due to the use of a modified method of producing composite pellets, which involved feeding strands of cellulose or abaca roving to a special extruder head and coating them with a PA matrix, similarly to the production of cables.

¹⁾ Cracow University of Technology, Department of Materials Engineering, al. Jana Pawła II 37, 31-864 Kraków, Poland.

*) Author for correspondence: paulina.romanska@pk.edu.pl

Recent investigation on PA 10.10 and PA 6.10 composites reinforced with regenerated cellulose fibers conducted by Wolff *et al.* also yielded beneficial results [7]. High values of tensile modulus, tensile strength and impact resistance were obtained. The possibility of using the tested composites in automotive interiors was suggested.

Hirsch *et al.* made a not extensive but an interesting comparative study on PA 10.10 and PA 11 composites containing 30% wt% of wood flour [8]. A superior increase in tensile strength and an improvement in thermo-mechanical properties were found for the PA 10.10 composites which was attributed to the higher number of amino groups per repeating unit in PA 10.10 compared to PA 11.

Several studies focused on tribological properties of biopolyamide composites, including the composites with short carbon fibers [11, 12]. Rajesh *et al.* also evaluated sliding properties and wear resistance of PA 11 filled with short glass fibers (20 wt%) and copper or bronze powder (6 wt%) [13]. Particularly satisfactory results were achieved by introducing copper powder into the composite, which resulted in a significant reduction in the coefficient of friction and the rate of abrasive wear. Recently, Pereira *et al.* obtained promising wear performance results of PA11 with titanate nanotubes (TTNT) [14]. Maximum wear resistance was reached in functionalized nanocomposites with low TTNT loading (0.5 wt%) and high sodium content.

Feldman evaluated fully or partially biobased polyamides (PA 10.10, PA 6.10, and PA 4.10) reinforced with 30 wt% recycled carbon fibers. The composites had outstanding mechanical properties and particularly high tensile modulus. The tensile strength was slightly higher compared to glass fiber reinforced composites and approx. 30% lower compared to composites with virgin carbon fibers [15]. Plasticized and unplasticized PA 10.10 with carbon fibers were also investigated previously by Kuciel *et al.* [16]. Low water uptake, low density, high specific modulus, and high biobased content were the most beneficial features of the composites.

Valuable work in the field of PA 10.10 matrix composites for technical applications was also undertaken by Rinberg *et al.* who investigated composites with various carbon or glass fibers content prepared via compounding and injection moulding [17]. The researchers focused on the reinforcing effect of the fibers and on the effect of fiber breakage during processing on the properties of the composites. They determined specific strength and stiffness of the composites as well as the impact strength and heat deflection temperature. They have found that carbon fiber reinforced PA 10.10 composites were advantageous in stiffness-dominated structures, while composites with glass fibers could be used in strength-dominated structures with the same efficiency as carbon fibers at a significantly lower cost. They qualified their composites for the use in thermally stressed components e.g. in the engine compartment of vehicles.

Another research on PA 10.10 with glass or carbon fibers was conducted by Nikiforov *et al.* but here hybrid composites of cellulose and carbon fibers were analysed [18]. The combination of cellulose and carbon fibers resulted in intermediate properties of the composites comparing with glass and carbon reinforced materials. The authors showed them as an attractive material from the standpoint of reducing the product weight and cost.

In an analogous manner, Armioun *et al.* focused on the PA 11 hybrid composites with wood fibers and carbon fibers. However, the composites were prepared via compounding and injection moulding at low processing temperatures (up to 210°C) and the resulting reinforcing effect of the combined fibers was not significant [19].

Hybrid composites of PA 10.10 with basalt and aramid fibers were also evaluated by Bazan *et al.* [20]. The authors showed that the addition of the fibers increased stiffness and strength properties in a moderate way and resulted in an increase in mechanical energy dissipation. For most of the assessed parameters, hybrid composites had intermediate properties between aramid and basalt fiber reinforced composites.

Bednarowski *et al.* investigated mechanical properties of composites with basalt and glass fibers [21]. Although the research concerned partially biobased PA 4.10 and not PA 10.10, the results proved the usefulness of basalt fibers as a replacement for glass fibers in PA composites. Tensile strength and tensile modulus of basalt reinforced composites were higher than for glass fiber reinforced composites, regardless of the fiber fraction (15, 30 or 50 wt%). It is important to note, that in this research basalt fibers underwent less fragmentation during the compounding process compared to glass fibers.

The cited studies indicate a wide application potential of composites based on completely bio-based PAs. Nonetheless, the literature on the subject lacks comparative studies of such materials with traditional PA composites, which have an established position on the market and are widely used in many industries. This publication is a response to this need.

The goal of the research was to compare biobased PA 10.10 short-fiber reinforced composites with analogous PA 6 composites. The choice of reinforcing fibers was not random. There are similarities between glass and basalt fibers in terms of their chemical composition and physical properties. Their production process is also similar. However, it does not require the introduction of additives or ingredients harmful to health and the environment, such as boron and fluorine oxides, present in most E-glass fibers [22]. While glass fibers are an obvious choice to reinforce PAs, basalt fibers have proven to be an alternative of added value [20–23]. In this study chopped glass or basalt fibers of similar nominal diameter were introduced into PA matrices in an amount of 15, 30 and 45 wt%. The properties determining the use of polyamides as engineering materials were analysed, including mechanical properties in dry, moisture condi-

Table 1. Samples characteristic

Sample	Polymer	Fiber	Fiber content, wt%	Density, g/cm ³
VT	PA 10.10	–	0	1.056±0.001
VT/15B		Basalt	15	1.156±0.002
VT/30B			30	1.274±0.020
VT/45B			45	1.391±0.004
VT/15G		Glass	15	1.197±0.001
VT/30G			30	1.271±0.004
VT/45G			45	1.417±0.003
TA	PA 6	–	0	1.140±0.005
TA/15B		Basalt	15	1.241±0.006
TA/30B			30	1.358±0.019
TA/45B			45	1.424±0.018
TA/15G		Glass	15	1.282±0.003
TA/30G			30	1.341±0.013
TA/45G			45	1.489±0.016

tioned and wet state, as well as their impact and thermal resistance. The mechanical properties were correlated with the microstructure of the composites.

EXPERIMENTAL PART

Materials

Long-chain polyamide 10.10 (viscosity number 160 cm³/g), Vestamid Terra DS 16 was provided by Evonik Industries (Geesthacht, Germany). Short-chain polyamide 6 produced by Grupa Azoty SA (Tarnow, Poland), under the trade name Tarnamid T-27, natural, with medium viscosity. Chopped glass and basalt fibers with a nominal monofilament diameter of 13 μm were used to reinforce the PA matrixes. Basalt fibers BSC 13-3.2 with a cutting length of 3.2 mm were supplied by Basaltex (Wevelgem, Belgium). These fibers were not available with a surface preparation for PAs, nor were they subjected to additional preparation as part of this research. CS EC 13 672 type E glass fibers with silane preparation (fiber length: 2-5 mm, diameter: 12.4-13.6 mm) were supplied by Johns Manville (Denver, CO, USA). Both PA matrices were reinforced by introducing the fibers in an amount of 15, 30 and 45% by weight. The tested materials are listed in Table 1.

Specimens preparation

Composite pellets were produced using a compounding line with a MARIS TM30 (Turin, Italy) co-rotating twin-screw extruder ($D = 30$ mm, $L = 36D$). The process was carried out at 260°C and screw speed was set to 80 rpm. In the case of glass fiber composites, the classic melt-compounding method was used. Chopped fibers were introduced into the plasticized matrix using gravimetric feeder. Due to low bulk density of chopped basalt fibers, an attempt to dose them classically during com-

pounding ended in failure. Therefore, PA pellets and basalt fibers were dry blended in an adequate proportion and compounded thereafter. The use of different compounding methods for basalt fiber composites, although necessary, was associated with undesirable effects. As described in the scientific literature, the average fiber length in the compound is significantly reduced with increasing dwell time in the extruder [21].

Standard type ISO 527 1A specimens were injection moulded on an Engel ES 200/40 HSL (Schwartzberg, Austria) injection moulding machine. The cylinder zones temperatures were set at 260°C for PA 10.10 and its composites, 250°C for PA 6, and 280°C for PA 6-based composites. Mould temperature for all the tested materials was 80°C.

Methods of testing

Density was measured by hydrostatic method using Radwag WAS 220/X (Radom, Poland). Water absorption was determined according to the ISO 62 standard. The percent increase in weight of the specimens was obtained after incubation periods of 1, 7, 30 and 90 days of water exposure at 23±2°C. Static tensile tests were performed using universal testing machine MTS Criterion 45 (Eden Prairie, MN, USA) (30 kN force capacity) with MTS axial extensometer with a constant crosshead speed of 5 mm/min. Modulus of elasticity, tensile strength and strain at break were determined. Tensile tests were conducted under conditions: dry as moulded materials, materials conditioned in air at 23°C and 50% relative humidity, wet materials after 90 days of incubation in water at room temperature. A JEOL JSM5510LV (Tokio, Japan) scanning electron microscope (SEM) was used for the microstructure analysis of the conditioned composites. SEM images were acquired on the gold-sputtered tensile fracture surfaces. Charpy impact tests were con-

ducted in accordance with the ISO 179 standard using the following methods: with notch (1eA method) and without notch (1eU method). The fracture energy was measured using a Zwick/Roell HIT5.5P (Ulm, Germany) device with a measurement range of up to 5 J. The tests were conducted on conditioned specimens. The heat deflection temperature (HDT) was determined according to the ISO 75 standard, method A (bending stress: 1.8 MPa). The test was performed on the Zwick/Roell HDT/Vicat A apparatus-B6-6300ALL.001 (Ulm, Germany) device. HDT was determined as a comparative measure of the thermal resistance of the tested composites (resistance to deformation under the influence of heat).

RESULTS AND DISCUSSION

Density and water absorption

Density and water absorption are the two main basic physical properties that characterise PA-based materials and are listed in technical data sheets of commercially available PA grades. The results of density measurements presented in Table 1 show the differences between composites based on long-chain bio-based PA 10.10 and materials based on short-chain PA 6. The density of the tested materials based on PA 6 is 6–8% higher than that of analogous materials based on PA 10.10. This is a significant difference from the point of view of the possibility of using such composites in the automotive industry and other industries where the weight reduction of products is a crucial issue. Basalt fibers have higher density than glass fibers, which is 2.55 g/cm³ and 2.67 g/cm³, respectively. Comparison of the density values of the composites allowed for the conclusion that the mass fractions of fibers in some of the composites (VT/45B, VT/15G, TA/45B, TA/15G) differed to some extent from the desired values. This must have been the result of irregularities in the dosing of fibers in the compounding process. On the other hand, the repeatability of the test results proves the excellent quality and repeatability of the test specimens.

A characteristic feature of PAs is their high absorption of water and other polar liquids. For short-chain polyamides, in which the methylene to amide ratio (CH₂/CONH) is low, water absorption is particularly high [22]. This is clearly visible in Fig. 1, which shows the water absorption of the tested PA matrices and composites. After 90 days of incubation, water absorption reaches 9.3% for unfilled PA 6. In long-chain polyamides the value is significantly lower and for unreinforced PA 10.10 after 90 days of incubation it is 1.3%.

To assess the effect of fiber content on the water uptake of composites correctly, the volume fraction of fibers in composites was calculated based on the density method (rule of mixtures for the density). The measured density of the tested materials and the densities of the fibers obtained from the producer's data were used for the calculations. As the volume fraction of fillers increased, the

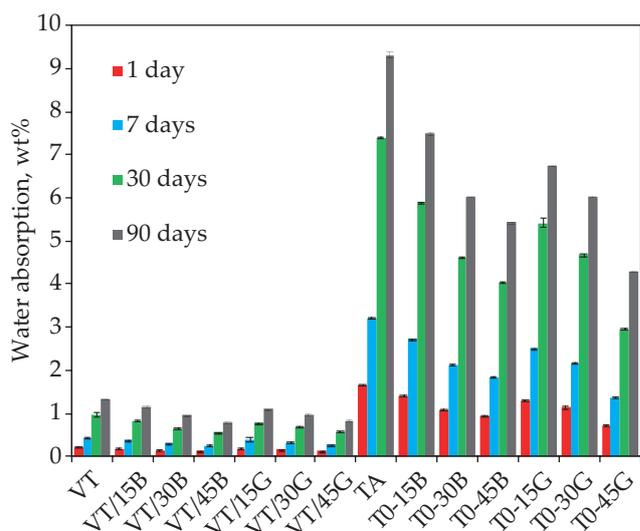


Fig. 1. Water absorption of the composites after different incubation time

water absorption decreased linearly, as shown for the water absorption after 30 days of incubation in Fig. 2. For both types of the fibers, a similar effect of their volume fraction on the reduction of water uptake was observed. Differences in the length of glass or basalt fibers and the presence or absence of surface preparation appeared to have no influence on the amount of water absorbed by the tested composites.

Mechanical properties

It is a well-known phenomenon that on the exposure to water or moisture at room temperature, water has a plasticizing effect on PAs or PA matrix composites, and this is a reversible process. It causes a decrease in the glass transition temperature, which in turn results in a decrease in strength parameters, but at the same time the deformability and impact strength of such materials increases. These statements can be found in many publications, including the paper of Hassan *et al.* [23].

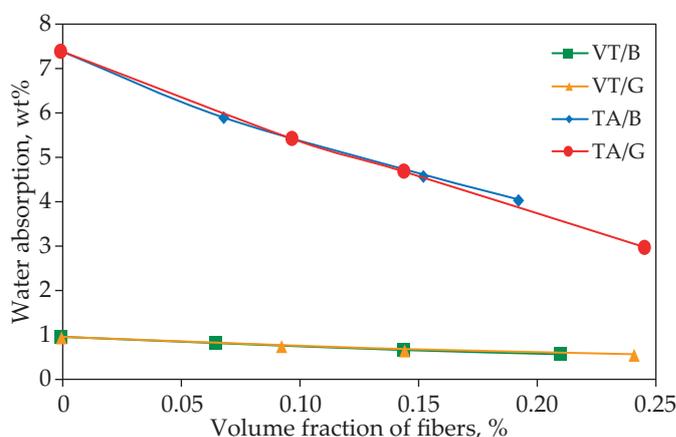


Fig. 2. Water absorption of composites vs fiber content after 30 days of incubation

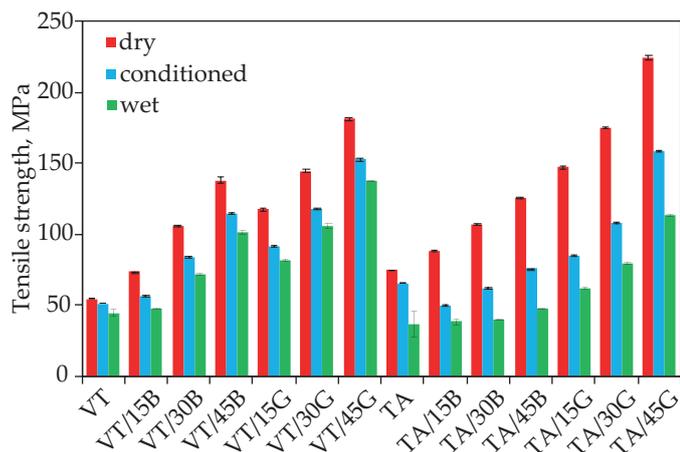


Fig. 3. Tensile strength of the composites in dry, conditioned, and wet state

The properties determined in the static tensile test for PA10.10 or PA 6 and the composites assessed in the dry, conditioned, and wet state are shown in Figs. 3–5. They highlight certain advantages of biopolyamide materials over traditional ones. In the dry state, PA 6 and its glass fiber filled composites had approximately 30% higher tensile strength and elastic modulus than PA 10.10-based materials. Nevertheless, it should be emphasized that in the moisture conditioned state, all the materials based on PA 10.10 showed higher strength parameters than those analogous based on PA 6 matrix. The effect was even more pronounced in wet conditions, and it was clear that PA 10.10 and its composites exhibited more stable mechanical properties with varying moisture or water content than PA 6-based materials.

Composites reinforced with basalt fibers had lower tensile strength and elastic modulus than composites with glass fibers. Better reinforcing effect was obtained by introducing basalt fibers into the PA 10.10, while the addition of basalt fibers into PA6 was of little benefit. The opposite effect was observed for glass fiber composites.

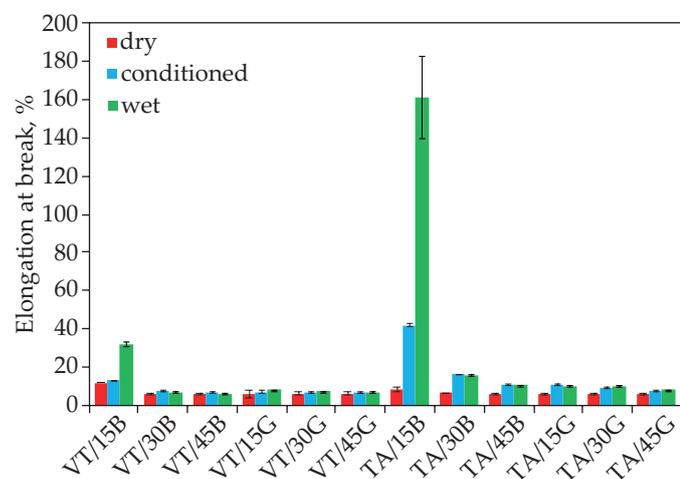


Fig. 5. Elongation at break of the composites in dry, conditioned, and wet state. The elongation at break for pure polyamides in the dry state is over 100% for PA 10.10 and over 200% for PA 6

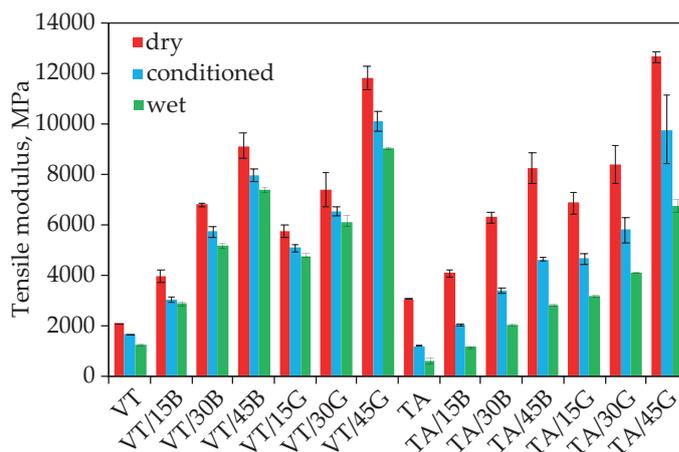


Fig. 4. Tensile modulus of the composites in dry, conditioned, and wet state

Moreover, composites with basalt fibers showed greater deformation at break, especially those based on PA 6 matrix. This may indicate intensive fiber pulling out from the matrix and low ability of the fibers to block intermolecular slip in the matrix. Based on microscopic observations (Fig. 6), it can be concluded that in the case of composites with basalt fibers, especially those based on PA 6 (Fig. 6a and c), a large number of voids left by pulled out fibers is visible. Further evidence is provided in Fig. 7 where fragments of basalt fibers with a smooth surface are clearly visible (Fig. 7a – TA/15B), while glass fibers are coated with a layer of the matrix (Fig. 7b – VT/15G). This indicates that the length of the basalt fibers was lower than the critical fiber length which was the effect of the severe compounding conditions and weak interactions at the fiber-matrix interface. On the other hand, the ductile fractures of PA 10.10 and PA 6 with glass fibers (Fig. 6b and d) indicate that the crack occurred by connecting areas of delamination at the ends of the fibers or areas of fiber cracking. The effect of glass fibers pull out is least distinct for PA 6 composites. This proves good adhesion of glass fibers to the PA matrices, especially for PA 6, which is consistent with mechanical test results.

Impact resistance

Pure PA 10.10 and its composites showed high impact strength in the conditioned state (Fig. 8). The highest unnotched Charpy impact strength was demonstrated by unreinforced PA 6. PA 10.10 composites showed impact strength comparable to that of PA 6 composites. The unnotched impact strength of the composites was lower than that of PA 6 or PA 10.10. However, most composites based on the PA 10.10 matrix showed higher notched impact strength than the unreinforced matrix. In most cases, as the fiber content increased, the impact strength of glass or basalt fiber composites increased too. Composites based on PA 10.10 showed similar or even slightly lower notch sensitivity compared to composites based on PA 6 matrix. The impact strength of composites

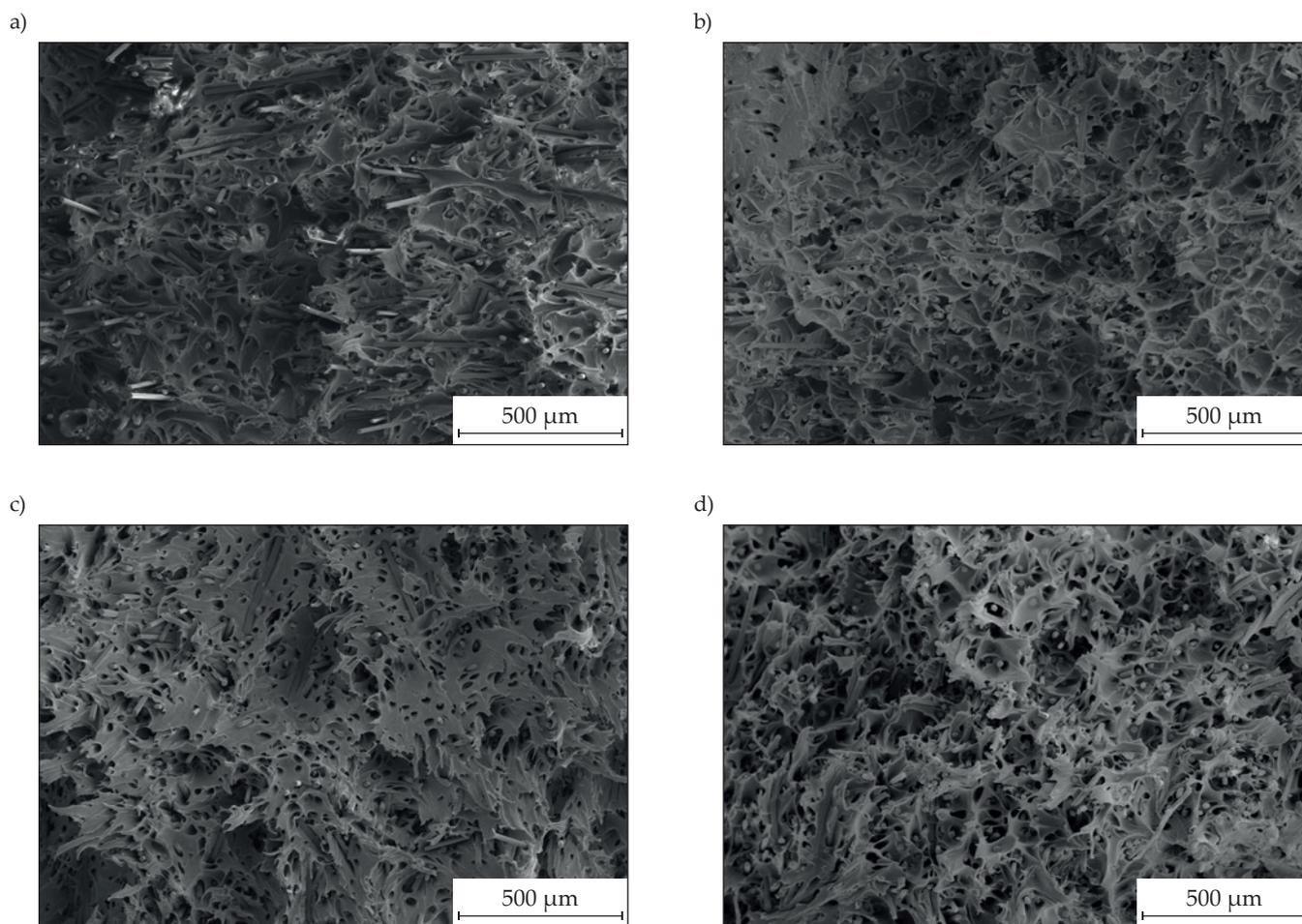


Fig. 6. Ductile fracture surfaces of the composites after static tensile test: a) VT/15B, b) VT/15G, c) TA/15B, d) TA/15G

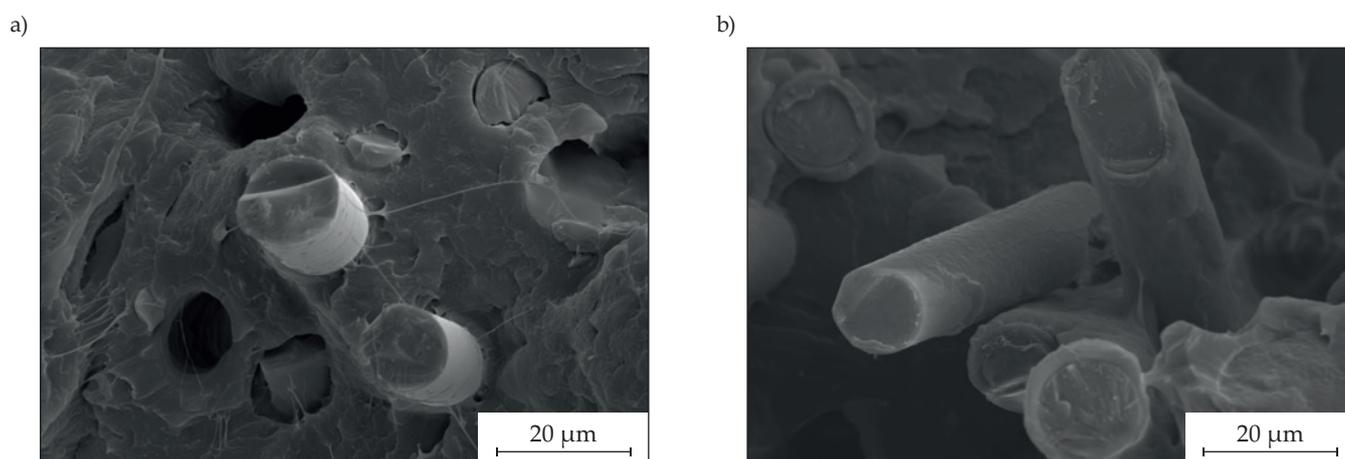


Fig. 7. Brittle fracture surfaces of the composites after static tensile test: a) TA/15B – surfaces of the fibers with no matrix layer (adhesive delamination effect), b) VT/15G – surfaces of the fibers covered with a thin matrix layer (cohesive delamination effect)

with basalt fibers, regardless of the matrix used and the measurement method, was lower, and the notch sensitivity was slightly higher than in the case of composites with glass fibers. Once again, the severe fragmentation of the basalt fibers during compounding process might have been a decisive factor here.

Thermal resistance

PAs and their composites are often exposed to elevated temperatures. Since the biopolyamide composites analysed in this work were assessed for the possibility of using them as structural materials competitive with

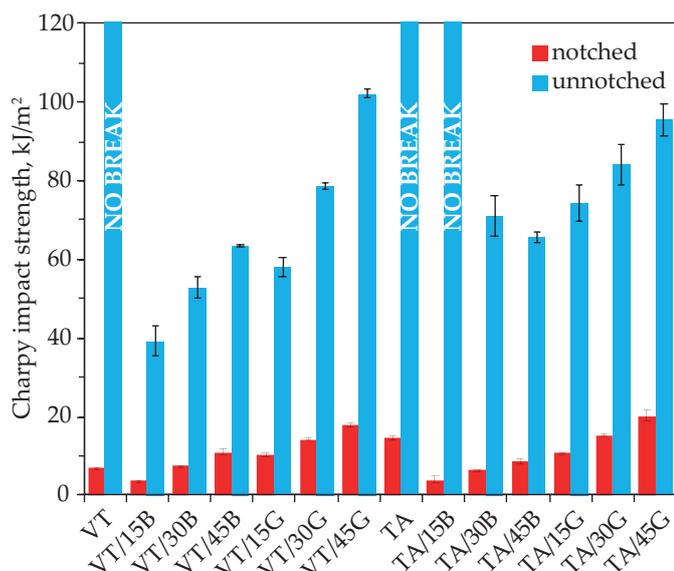


Fig. 8. Notched and unnotched Charpy impact strength of the composites

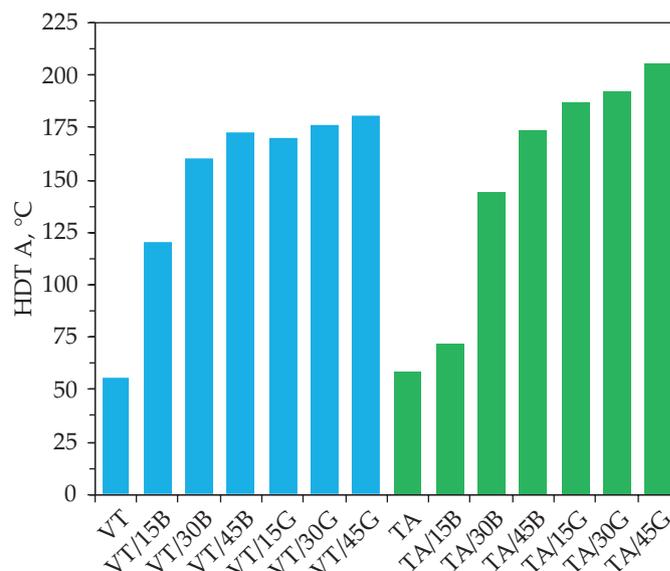


Fig. 9. Heat deflection temperature of the composites

PA 6-based composites, it was important to investigate their heat resistance. In the case of polymer composites, the parameter commonly used for this purpose is the heat deflection temperature (HDT). The results of measurements performed for PA 10.10 and PA 6 and their composites are presented in Fig. 9.

Pure PAs showed a similar HDT value (56–58°C) in dry state. As in the case of other engineering semi-crystalline materials, the introduction of reinforcing fibers led to a significant increase in heat resistance of both PA 6 and PA 10.10. PA 6 composites exhibited higher thermal resistance comparing to analogous PA 10.10 composites. High values of HDT A exceeding 170°C were obtained for all the glass reinforced composites and for basalt fiber composites with 45 wt% of the fibers. The values exceeding 180°C were reached for all the PA6/glass fiber composites and for PA 10.10 composite with 45 wt% of glass fibers. PA 6 composites with basalt fibers were characterized by the lowest heat resistance (the lowest reinforcement efficiency at elevated temperatures). Here, the effect of the fiber type had more significant impact on the HDT value than the fiber content, on the contrary to the results of Rinberg et al study where glass or carbon fiber reinforced PA 10.10 were compared [16]. This indicates that the fiber length is more crucial for such composite properties like impact resistance or heat resistance measured at bending conditions (HDT) than the chemical composition of the reinforcing fibers.

CONCLUSIONS

Taking into account the results of all performed tests, the type of the matrix used (PA 10.10 and PA 6) had a very significant, greater impact than the type of fibers used (basalt and glass fibers) on parameters such as density, water absorption and changes in mechanical properties along with increasing water uptake. PA 10.10 was supe-

rior to the traditional PA 6 in these respects. Composites based on the PA 10.10 matrix were also characterized by a similar Charpy notched impact strength to the composites based on the PA 6 matrix.

The best reinforcing effect and the highest thermal resistance were achieved for PA 6 composites with glass fibers. This should not be surprising, as glass fibers have been developed for years to reinforce short-chain PAs. This results in good adhesion at the fiber-matrix interface and facilitates the production of composites using the compounding and injection moulding methods.

Composites with glass fibers produced using the classic compounding method were characterized by better mechanical parameters and higher heat resistance than composites with basalt fibers obtained by extruding a dry mixture of matrix pellets and fibers. Most probably, the degree of fragmentation of basalt and glass fibers and their surface preparation had a fundamental impact on the mechanical properties and varied mechanisms of destruction of PA composites under static loads. This was confirmed by the fractographic observations. Unfavourable microstructural features resulted in lower reinforcement efficiency for composites with basalt fiber than for composites with glass fibers. To improve the mechanical and thermomechanical parameters when using basalt fibers, it is necessary to use fibers with appropriate surface preparation. Currently, the availability of basalt fibers with the desired surface preparation and sufficiently high bulk density is much greater than at the time of the research described here. However, a large diameters of basalt fibers available on the market (10–20 μm) remains a limitation compared to chopped glass fibers intended for reinforcing thermoplastics (6–13 μm).

Author contributions

P.R. – conceptualization, methodology, validation, investigation, writing-original draft, writing-review and edit-

ing, visualization; S.K. – methodology, investigation, writing-original draft, supervision.

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Conflict of interest

The authors declare no conflict of interest.

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