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Polyamides from renewable sources as matrices of short fiber reinforced biocomposites

Summary — At present, in production of commercially available biopolyamides, castor oil is used as one of raw materials. In this paper possibilities of processing biocomposites based on biopolyamide (commercially delivered by Chinese company Suzhou Hipro Polymers) filled with 10 or 20 wt. % of glass, carbon or flax fibers were presented. Tensile properties (tensile strength, modulus of elasticity and elongation at break) of the composites in their conditioned state and after water soaking were discussed. As the results showed, a strong effect of reinforcement was obtained for all of the composites. The composites with glass and flax fibers have a great elongation and impact resistance. To evaluate fatigue stress of tested materials, simplified shortened Lehr's method was used. The results of this work can be used in the designing process of structural materials with varying stiffness and ability to dissipate energy depending on the expectations of the recipient.

Keywords: biopolyamide, biocomposite, renewable sources, flax fiber, glass fiber, carbon fiber.

POLIAMIDY ZE ŹRÓDEŁ ODNAWIALNYCH JAKO OSNOWY BIOKOMPOZYTÓW WZMACNIANYCH KRÓTKIMI WŁÓKNAMI

Streszczenie — W pracy zaprezentowano aktualne możliwości wytwarzania i modyfikacji poliamidów z surowców odnawialnych. Na przykładzie poliamidu otrzymywanego z oleju rycynowego i produkowanego na skalę przemysłową (Hiprolon 211) przedstawiono wyniki badań kompozytów z dodatkiem 10 lub 20 % mas. włókien — szklanych, węglowych lub lnianych. Porównano podstawowe właściwości wytrzymałościowe czystego biopoliamidu i jego kompozytów o różnym stopniu napełnienia (rys. 2—4). Wprowadzenie włókien we wszystkich przypadkach dało wyraźny efekt wzmocnienia. Wyznaczono wartości gęstości, temperatury mięknienia Vicata, udarności określanej metodą Charpy'ego z karbem (tabela 1), a także chłonności wody po 1, 7 i 30 dniach moczenia. Zbadano również zmiany właściwości wytrzymałościowych po 30-dniowej inkubacji w wodzie (rys. 6 i 7) czystego biopoliamidu i kompozytów z dodatkiem 10 % mas. napełniaczy. Techniką SEM wykonano fotografie powierzchni przełomów po rozciąganiu wytworzonych kompozytów. Widoczne są na nich charakterystyczne cechy struktury i morfologia różnych włókien (rys. 3). Przedstawiono rezultaty badań dynamicznych kompozytów, a w szczególności poziomy sił wymuszających, średnie wydłużenie oraz energie dyssypacji odpowiadających narastającym ilościom pętli mechanicznej histerezy (tabela 2, rys. 9). Porównano pętle histerezy kompozytów z włóknami szklanymi, węglowymi i lnianymi (rys. 8). Wyniki przedstawionych badań wykazały, że istnieją duże możliwości projektowania właściwości wytrzymałościowych kompozytów na osnowie długołańcuchowych biopoliamidów, ponieważ właściwości te zależą od rodzaju oraz ilości wprowadzanych włókien.

Słowa kluczowe: biopoliamid, biokompozyt, surowce odnawialne, włókna lniane, włókna szklane, włókna węglowe.

Biopolyamides are polyamides fully or partially derived from renewable resources. They can be synthesized in the following possible processes [1]:

— polycondensation of diamines and dicarboxylic acids with elimination of water — there are examples of bio-PA with biobased acid and petroleum-based amine (*e.g.*: PA 610, PA 410) and of biopolyamides where both components are obtained from biomass (*e.g.*: PA 1010, PA 510),

— polycondensation of amino carboxylic acids as bifunctional monomers (*e.g.*: PA 11),

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— ring-opening polymerization of lactams (*e.g.* biobased PA 6, PA 4 — production on a laboratory scale).

At present, in production of commercially available biopolyamides, castor oil is used as a renewable feedstock. Among other industrial plant oils, castor oil exhibits unique chemical structure and properties. It is built in almost 90 % of ricinoleic acid with double bond and hydroxy group, which provide possibilities for chemical modifications [2, 3]. The modifications may lead to synthesis of polyamides building blocks, such as sebacic acid, aminoundecane acid or decamethylenediamine (DMDA), which are schematically represented in Figure 1 [4].

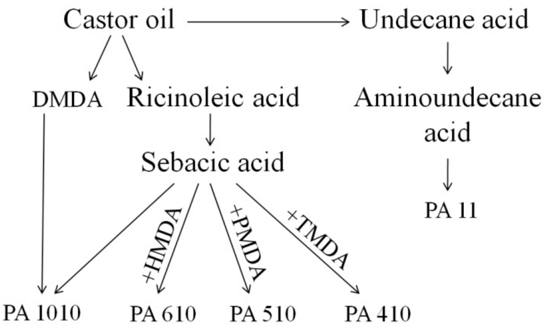

Fig. 1. Main routes of manufacturing biopolyamides from castor oil [4]; DMDA — decamethylenediamine, TMDA — tetramethylenediamine, PMDA — pentamethylenediamine, HMDA — hexamethylenediamine

Few companies have already introduced different kinds of long-chain biopolyamides into the market. Polyamide 11 (Rilsan PA 11 from Arkema) has been commercially available for over 60 years now and applied in different industry sectors (*e.g.*: automotive, textile, electronics), mainly in coatings. Polyamide 1010 (offered by DuPont, EMS-Chemie, Evonik and Suzhou Hipro) has properties similar to properties of PA 11 and may be used as its substitute. Both PA 11 and PA 1010 are fully made from castor oil. Polyamide 510 is now produced by BASF, but not yet commercially available. Its dicarboxylic acid part is synthesized from castor oil, while diamine is obtained from lysine (product of glucose fermentation). Thus PA 510 is fully biobased. Polyamide 610 (BASF, DuPont, EMS, Rhodia, Evonik, Toray, Suzhou Hipro, Akro-Plastic) with about 60 % of renewable-source content has recently met with a great interest, as it combines high mechanical properties (characteristic of PA 6) with low density and high chemical resistance (typical of PA 1010). Another partially biobased polyamides available on the market are PA 410 (up to 70 % biobased, DSM company) and PA 1012 (about 40 % bioabsed, Evonik, Suzhou Hipro).

There are also other polyamides that could be made from plant oil derivatives like, for example: PA n12, PA n4, PA 69, where „n" corresponds to the number of carbon atoms in different possible diamines [5, 6]. Moreover, some polyamides can be produced from sugars. There is a patented method of PA 6 synthesis by ring-opening polymerization of ε-caprolactam produced by glucose fermentation [7]. Likewise, glucose may be used as feedstock to produce PA 4 by fermentation to glutamate, its decarboxylation to γ-aminobutyric acid (GABA), which is then heated to produce 2-pyrrolidone and finally by ring-opening polymerization of 2-pyrrolidone [8]. However, production of biopolyamides from carbohydrates is not yet cost-effective.

In polyamides, intermolecular interactions, which determine their properties, decrease with an increasing length of polymer chain. Long-chain biopolyamides such as PA 11, PA 1010 and, to a lesser extent, PA 610 have lower density, lower water absorption but also significantly lower strength and stiffness, lower melting temperature and continuous operating temperature than polyamides like PA 6 [4]. In order to improve mechanical performance and thermal stability of long-chain biopolyamides, different fillers, both synthetic and natural, can be used. Błędzki *et al.* [9, 10] studied composites on a base of two biopolyamides PA 1010 and PA 610, filled with man-made or natural (abaca) cellulose fibers (15—30 wt. %). The fillers addition caused significant enhancement of stiffness and strength. There was also an increase of impact strength for man-made fibers and a decrease for natural filler. Liu Z. *et al.* [11] tested PA 1010/montmorillonite nanocomposites prepared by intercalating polymerization. These nanocomposites had higher modulus of elasticity and onset temperature of decomposition compared with neat biopolyamide. Similar results were obtained by Zeng *et al.* [12] for multiwalled carbon nanotubes-reinforced PA 1010 composites and by Liu T. *et al.* [13] for PA11/nanoclay composites prepared with the melt-compounding method. Rajesh and Bijwe [14] also reported major increase of mechanical properties, hardness, friction and wear properties of polyamide 11 filled with short glass fibers (20 wt. %) and copper or bronze powders (6 %), processed by extrusion followed by injection molding.

Characteristics of composites based on polyamides filled with different fibers are mainly focused on mechanical properties under static conditions [15, 16]. However, in many applications it is important to know how the material will perform under repeated loading cycles as well as what maximum stress it will bear before failure, to suitably design it. As in all engineering materials, failure often appears in plastics as a consequence of accumulated irreversible damage or growth of a fatigue flow to a critical degree. The fatigue life of a polymeric composite is controlled by a number of factors. These include: the stress or strain amplitude of the loading cycle, the mean stress of the cycle, the presence of stress concentrations or initial defects in the component, the frequency, temperature and environment of the test. To predict fatigue life of polymers and fatigue strength, Wohler curve

(S-N curve) is used. It is a graph of the magnitude of a cyclic stress (S) against the logarithmic scale of cycles to failure (N). S-N curves are derived from tests where a regular sinusoidal stress is applied to the specimen by a testing machine which also counts the number of cycles to failure. An S-N curve provides information about the amount of cycles at which the material will fail for different maximum stress levels and a unique value of stress ratio. To plot the curve, multiple tests on similar specimens with different levels of stress must be carried out. As a consequence, considerable time is needed [17—19].

For approximate determination of fatigue strength, authors used a simplified time-saving method of dynamic testing — Lehr's method [20, 21]. This method is used for metals and is based on the observation that fatigue strength is connected with local plastic strains which start to appear after exceeding fatigue strength in individual grains of metal. This method was implemented for polymers and allowed determining the fatigue properties of polyamide filled with mineral filler tuff and composites based on polyhydroxybutyrate filled with natural fibers [22, 23]. The essence of the method is to use variable loading with increasing stress amplitude and register the value of extension, dissipated energy and temperature. After exceeding the amplitude of variable stress which is close to fatigue stress in tested material, the increase of temperature, strain and dissipated energy can be observed and on that base fatigue strength can be calculated [24].

In the current paper, authors present chosen basic physical properties and dynamic tests results of commercially available biobased polyamide filled with flax, glass or carbon short fibers. The aim of the study was to examine the influence of each kind of filler on the biopolymer matrix and to determine whether the tested biocomposites possess features, that can enable them to be used as a replacement of traditional engineering, durable polyamides, like polyamide 6.

EXPERIMENTAL

Materials and specimens preparation

Three types of short fiber reinforced biocomposites have been investigated in this study. As their matrix Hiprolon 211 (Suzhou Hipro Polymers, China), a long-chain polyamide compound from castor oil has been used. The composites were made with 10 and 20 wt. % of flax, glass or carbon fibers. Untreated flax fibers, cut and low twisted roving with diameter of 10—30 μm and length of 120 μm were supplied by Safilin Sp. z o.o. Glass fibers (diameter: 10 μm, average length: 120—150 μm) and carbon fibers (diameter 8 μm, average length 80—120 μm) were provided by Zakłady Azotowe in Tarnów.

Standard dumbbell type specimens (10 × 4 × 150 mm) were produced in a two-step process. First, the Hiprolon 211 pellets and fibers were compounded on a twin-screw extruder with a gravimetric screw feeder. Thus obtained granulate was injection molded using Engel ES 200/40 HSL in Zakłady Azotowe in Tarnów. The injection process was done under the following conditions: injection temperature 210 °C, mold temperature 200 °C, injection pressure 100 MPa, cycle time 45 s (cooling time 20 s).

Methods of testing

Mechanical properties were estimated by a tensile test according to PN-EN ISO 527, with an universal testing machine (Insight 50 MTS with MTS axial extensometer) with a constant crosshead speed of 10 mm/min. Elastic modulus (E_t), tensile strength (σ_M) and strain at break (ε_B) were determined. Charpy impact strength (a_{cN}) was obtained from notched specimens using Zwick HIT5.5P tester. Materials density (ρ) was measured by hydrostatic method.

In order to observe the composite structures, scanning electron microscopy (SEM) images were made on the gold-sputtered tensile-test fracture surfaces of specimens using JEOL ISN5510LV microscope.

Absorption of water (at 20 °C) was determined after 1, 7 and 30 days of soaking, according to PN-EN ISO 62:2000. To determinate changes in properties caused by absorbed water, tensile test was performed again immediately after the 30 days of the specimens incubation.

Load-controlled cyclic fatigue tests were performed in load-tension mode at the temperature of 23 °C using hydraulic tensile tester Instron 8511.20 equipped with MTS controller and using software TestWork 4.0. Multiple applied load levels (5000 cycles on each load level) were conducted to generate hysteresis loops for the material. For all variable loading cycles performed on all specimens minimum applied load was equal to 0.1 kN. The maximum load value for the first 5000 hysteresis loops was set to 0.3 kN and in the following loops series that load level was gradually increased by 0.05 kN to ultimately achieved 0.8 kN in the last hysteresis loops. All fatigue tests were conducted at 5 Hz cyclic frequency. Hysteresis loops were registered to a file and then maximum stress at fatigue (z_z), mean elongation (l_m) and energy dissipated in each cycle (E_d) were calculated.

RESULTS AND DISCUSSION

Basic mechanical and physical properties

Table 1 presents the results of density (ρ), Vicat softening point (*VSP*) and Charpy notched impact strength (a_{cN}) measurements for 10 wt. % filled biopolyamide composites.

One of advantages of the long-chain biopolyamides and their composites is their low density. For manufacturing composites with natural fibers low processing temperature is also an advantageous feature — biopolyamide composites can be produced at temperatures lower than the lignocellulosic fiber decomposition temperature (about 210 °C).

T a b l e 1. Characteristics of biopolyamide composites filled with 10 wt. % of fibers

Matrix	Fiber type	Fiber content wt. %	Symbol of sample	ρ g/cm^3	*VSP* °C	a_{cN} kJ/m^2
Hipro-lon 211	—	—	Hpr	1.05	168	78.6
	Flax	10	Hpr/10F	1.06	172	17.5
	Glass	10	Hpr/10G	1.10	174	22.1
	Carbon	10	Hpr/10C	1.07	180	24.1

Introduction of fillers usually enables the composite to work at higher temperatures than neat polymer. As it can be seen from results listed in Table 1, addition of fillers, especially carbon or, to a lesser extent, glass fibers resulted in the noticeable increase of *VSP*.

Tensile properties like tensile strength (σ_M), modulus of elasticity (E_t) and elongation at break (ε_B) of tested materials are presented as functions of fiber weight content in Figures 2—4. These test results were highly repeatable (less than 5 % variation in most cases). Strong effect of reinforcement was obtained for all of the composites. σ_M values were above 2 and 3 times higher for samples with 10 and 20 wt. % of carbon fiber content respectively, than for neat biopolyamide. The 20 wt. % of flax fibers addition caused almost the same strength increase as 10 wt. % of glass fibers filling (about 26 %). The most positive achieved effect of fibers adding was the significant improvement of E_t of composites at low fillers content: from 2 and 4 times for 10 and 20 wt. % of flax fibers to even 18 times for 20 wt. % of carbon fibers. Thus the biocomposites modulus of elasticity values are similar or higher than for well-known neat polyamide 6 (for Tarnamid T-27, E_t = 1 100 MPa [25]). Composites filled with carbon fibers have the highest mechanical properties, but were also the most brittle. The rest of low fiber content composites preserved good ductile properties, *i.e.*, ε_B and impact strength values. All these features make the biopolyamide composites acceptable as construction materials.

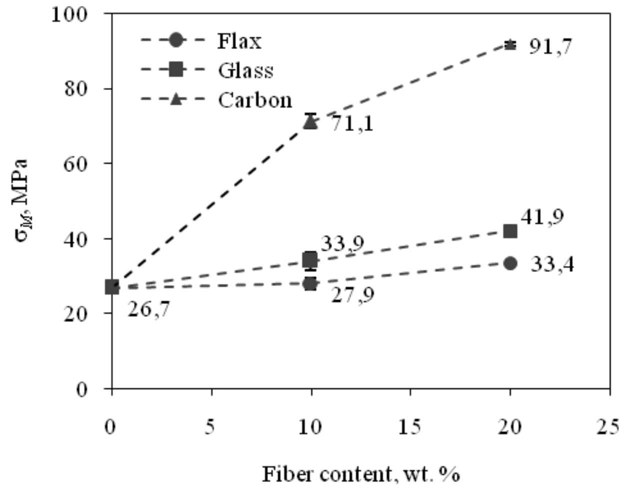

Fig. 2. Tensile strength (σ_M) of biopolyamide based composites as a function of fiber content

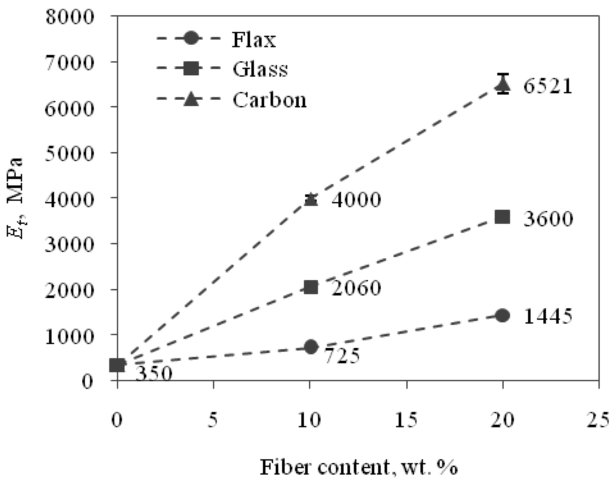

Fig. 3. Modulus of elasticity (E_t) of biopolyamide based composites as a function of fiber content

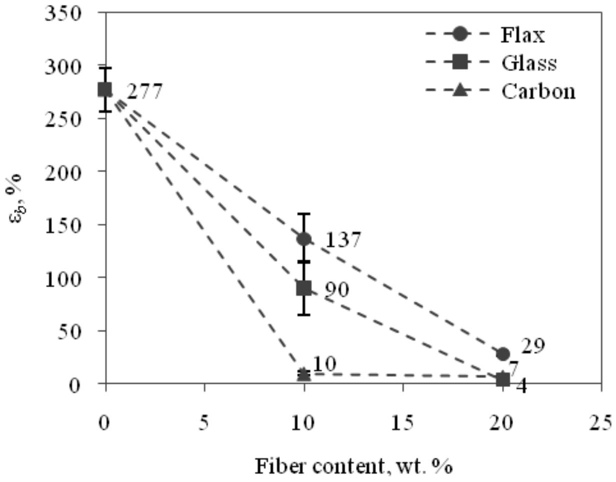

Fig. 4. Elongation at break (ε_B) of biopolyamide based composites as a function of fiber content

Scanning electron microscopic observations

After the tensile tests, the microstructures of the 10 wt. % filled composites were analyzed. In Figure 5 there are SEM images of the composites tensile test fractures. The Hpr/10F specimen was broken in liquid nitrogen in the tensile fracture zone as its highly developed fracture surface hindered the microscopic observations. Hence the images of Hpr/10F (Figs. 5a and 5b) show semicrystalline nature of biopolyamide matrix. The same images also prove that the biopolyamide composites can

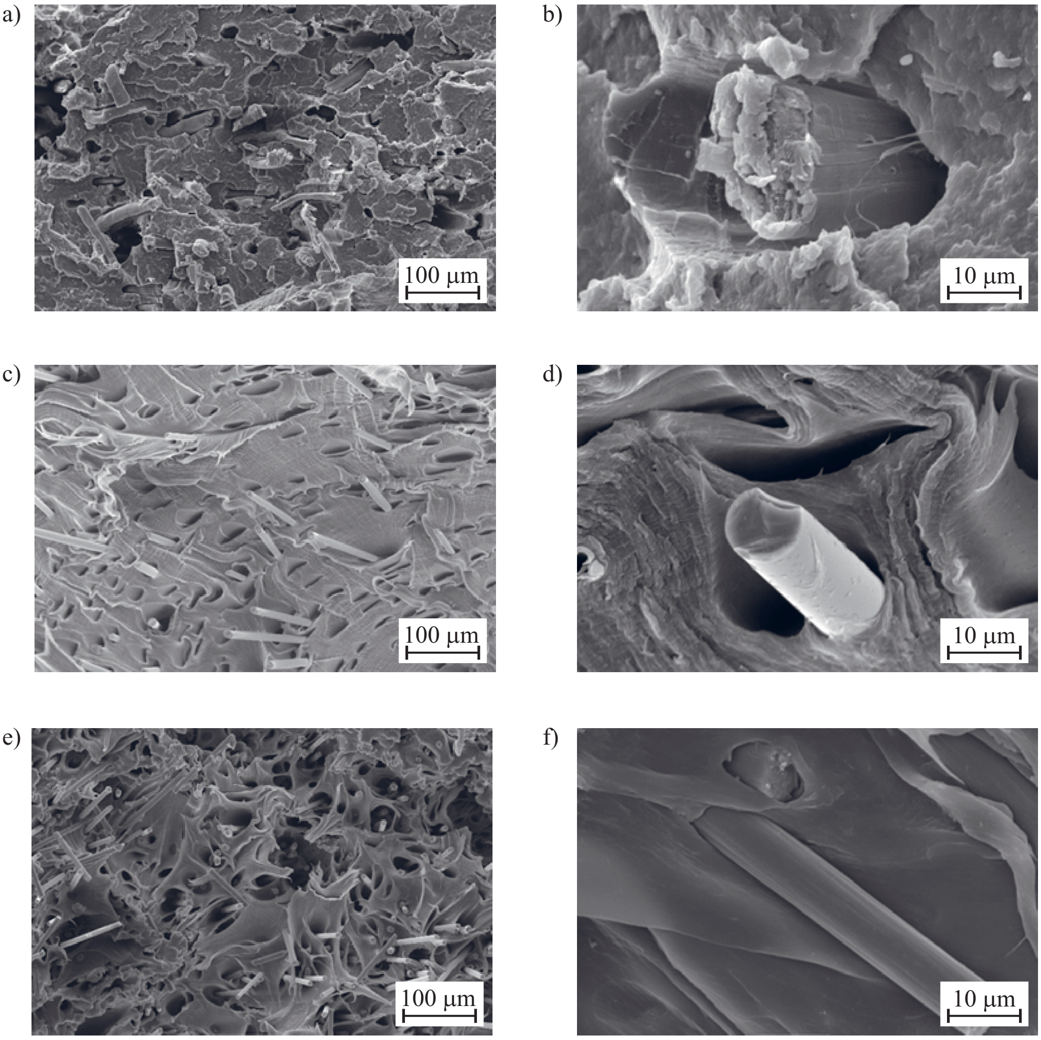

Fig. 5. SEM images of tensile fracture surfaces of the tested composites: Hpr/10L specimen broken in liquid nitrogen in the tensile fracture zone (a and b), Hpr/10G (c and d), Hpr/10G (e and f)

be successfully processed with natural fibers as there are no visible signs of thermal damage and deformations of the flax fibers in Figs 5a and 5b.

Although in the images in Fig. 5 voids left by pulled-out fibers and discontinuities at the fiber-matrix interfaces can be seen, the fiber-matrix adhesion was obtained, what can be proved by mechanical tests results (especially by the enhancement of tensile strength).

Water absorption and its influence on mechanical properties

Water absorption of neat biopolyamide and its composites calculated after 1, 7 and 30 days of soaking are shown in Figure 6 [26]. As it was expected, lignocellulosic flax fibers caused a considerable increase of absorbability, while for carbon or glass fibers there were no major changes in the absorption value. Nevertheless, the Hpr/10F composite still absorbs less water after 30 days of soaking than polyamide 6 (Tarnamid T-27, Azoty Tarnów) after 24 hours of exposure (1.8 % according to Tarnamid T-27 producer data) [25].

The influence of water soaking on the discussed materials properties is plainly visible in the graph in Figure 7,

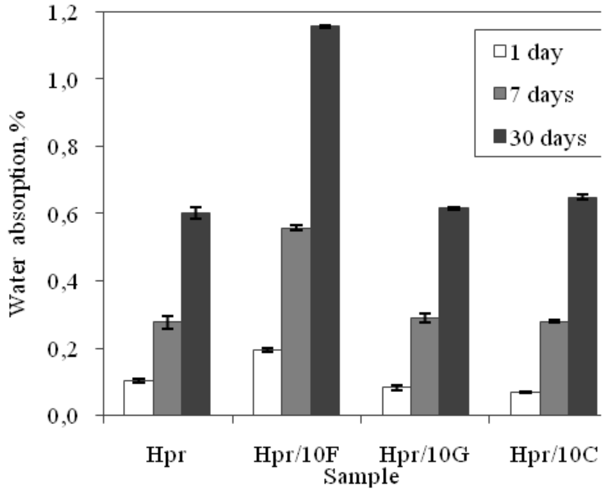

Fig. 6. Water absorption of biopolyamide and its 10 wt. % filled composites after 1, 7 and 30 days of soaking

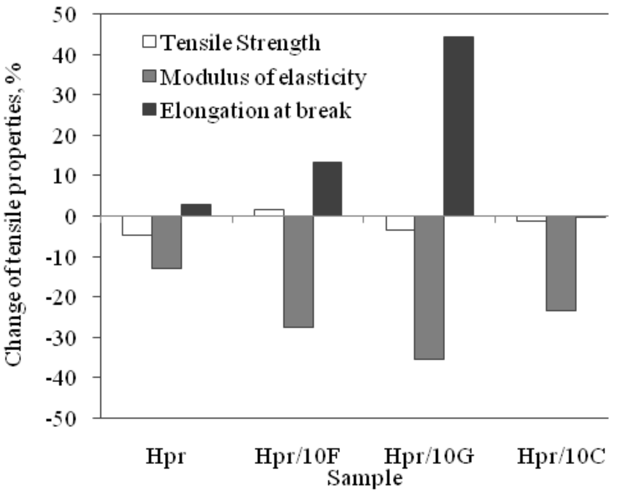

Fig. 7. Percentage changes of tensile strength, modulus of elasticity and elongation at break for tested materials after 30 days water soaking with regard to the initial state (positive values indicate an increase, negative — a decrease)

which shows tensile tests results performed after 30 days of incubation. The changes of σ_M value are the least noticeable. The most evident is a decrease of E_t of all tested materials. An interesting observation is that the flax composite (Hpr/10F) stiffness decreased after water soaking was lower than for the glass composite (Hpr/10G). It may be an effect of flax fibers expansion due to their water absorption which eventually induces residual stresses in the composite.

Dynamic tests results

Results of measurements of maximum forces in hysteresis loops for increasing number of cycles and calculation of maximum stress at fatigue (z_z), mean elongation (l_m) and energy dissipated in each cycle (E_d) for tested composites are presented in Table 2.

Maximum carried cyclic loads (corresponding to the maximum chosen load P_{max}) for tested specimens accounted for 52 % for Hpr/10G, 45 % for Hpr/10F and 21 % for Hpr/10C of their maximum tensile forces.

Adding carbon fibers to the biopolyamide matrix did not increase fatigue strength. Heating of the viscoelastic matrix could be a reason of composites failures before fibers breaking or pulling them out from matrix. Comparing to the glass, flax fibers addition to biopolyamide resulted in a 7 % lower maximum stress at fatigue and in a 20 000 lesser number of cycles to failure. Failure of composites filled with flax fibers appears to be a consequence of matrix heating and fibers breaking.

From the hysteresis loops recorded during the fatigue test, presented in Figure 8, increase of loops surface and mean elongation and stability of the slopes of hysteresis loops under increasing number of stress cycles were observed.

The surface of hysteresis loop is a measure of the energy dissipated by polymeric material during deformation. The extension of its surface means that the dissipated energy increases in the function of cycles number. The increase of mean elongation which occurred in all tested materials can be attributed to creep effects in material under cyclic loading. The main advantage of the composites filled with carbon fibers (Hpr/10C) is their great stiffness and small creep effects. What can be observed in Fig. 8 is different slope of hysteresis loops for tested fillers — the biggest for carbon and the smallest for flax fibers which corresponds with their static modulus of elasticity. Comparison of energy dissipated in each cycle (E_d) under increasing load for tested composites is shown in Figure 9.

It can be noted that at the beginning stage of fatigue process the dependence of dissipated energy versus number of cycle is linear and before cracking it rapidly increases. It is one of the assumptions of Lehr's method of approximate estimated fatigue stress (z_z).

Table 2. The level of maximum forces (P_{max}) in hysteresis loops versus increasing number of cycles and results of calculations: maximum stress at fatigue (z_z), mean elongation (l_m) and energy dissipated in each cycle (E_d) for tested composites

The chosen level of P_{max}, kN	0.3	0.35	0.40	0.45	0.5	0.55	0.6	0.65	0.7	0.75	0.8
Number of cycles	5000	10 000	15 000	20 000	25 000	30 000	35 000	40 000	45 000	50 000	55 000
Hpr/10F (σ_M = 27.9 MPa, z_z = 12.5 MPa)											
Maximum force in loop, kN	0.28	0.35	0.40	0.45	0.50	0.55	0.60	—	—	—	—
l_m, mm	1.84	2.30	2.77	3.27	3.87	4.59	5.51	—	—	—	—
E_d, J	5.4	14.6	23.8	35.5	51.9	74.4	103.0	—	—	—	—
Hpr/10C (σ_M = 77.1 MPa, z_z = 16.25 MPa)											
Maximum force in loop, kN	0.35	0.40	0.45	0.50	0.55	0.60	0.65	0.70	0.75	0.78	—
l_m, mm	0.26	0.29	0.34	0.39	0.44	0.49	0.55	0.6	0.66	0.71	—
E_d, J	2.0	2.3	3.0	4.1	5.7	7.2	9.4	11.8	14.3	17.9	—
Hpr/10G (σ_M = 33.9 MPa, z_z = 17.5 MPa)											
Maximum force in loop, kN	0.31	0.35	0.40	0.45	0.50	0.55	0.60	0.65	0.69	0.74	0.76
l_m, mm	0.74	0.84	0.95	1.06	1.18	1.34	1.50	1.74	2.17	3.47	32.7
E_d, J	3.2	3.7	5.5	7.7	10.7	15.2	21.6	32.8	56.9	124.4	541.6

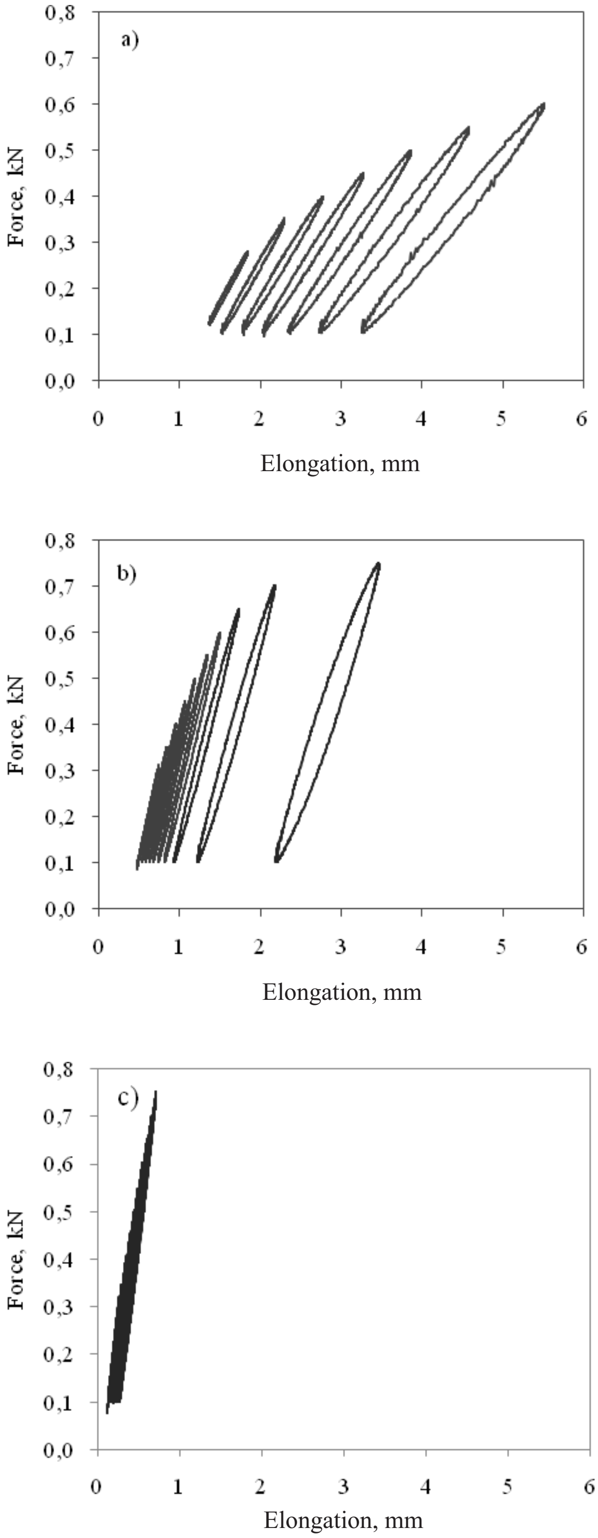

Fig. 8. Comparison of hysteresis loops for increasing values of forces for composites with biopolyamide matrix filled with 10 wt. % of fibers: a) Hpr/10F, b) Hpr/10G, c) Hpr/10C (5 Hz, 5000 cycle on one level of force)

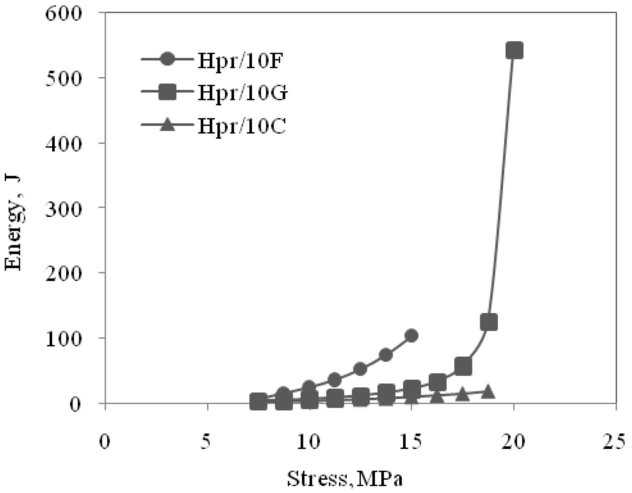

Fig. 9. Comparison of energy dissipated in a cycle (E_d) for tested composites (Hpr10F, Hpr10G, Hpr10C) for increasing values of forces (5 Hz, 5000 cycles at one level of force)

CONCLUSIONS

To conclude, composites of biobased polyamide filled with different fibers may be an interesting alternative to currently used composites based on petrochemical polyamides. Their mechanical properties are comparable and high impact resistance makes them especially suited to be used in the parts working in motion and under dynamic load. Modification with glass fibers is more advantageous both for static and dynamic properties comparing with flax fibers. Addition of flax fibers enables to produce composites derived entirely from renewable raw materials with good strength and stiffness and high toughness, without additional chemical treatment. Composites filled with carbon fibers have a great stiffness, very good static and sufficient fatigue strength.

Conducted tests confirmed the possibility of using time-saving Lehr's method for obtaining approximate value of fatigue strength of composites based on castor oil polyamides. Under the influence of cyclic loadi, strain and dissipated energy increased in the function of number of cycles and development of fatigue process. In case of composites with glass or flax fibers development of fatigue process may result from fracture of fibers and interface decohesion, but these effects have to be confirmed in further tests.

The results of this work can be used in a designing process of structural materials with varying stiffness and ability to dissipate energy depending on the expectations of the recipient. The first industrial sectors that are or could be interested in the production of parts based on biopolyamide matrix composites are the automobile industry and consumer goods (*e.g.*: electronics, sports and household applications).

In times of depletion of petrochemical feedstock and large interest in fossil CO_2 emission reduction, the renewable raw materials based composite matrices are the focus of both science and industry.

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