



A study on the mechanical properties and the influence of water uptake and temperature on biocomposites based on polyethylene from renewable sources



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ABSTRACT

This work is aimed to evaluate the properties in terms of structural applications of the fully biobased composites obtained at reasonable price, without additional and costly chemical modifications. The bio-polyethylene obtained from sugarcane ethanol (Braskem, Brazil) was filled with four different fillers (25 wt.%): wood flour, ultrafine cellulose powder, kenaf chopped fibres and microparticles of mineral tuff filler. Physical, mechanical and thermal properties of the biocomposites were tested, as well as the influence of soaking in water and temperature on tensile properties. The fracture surfaces were studied using scanning electron microscope. Low density, increase in stiffness, improved resistance to deformation on heat and thermal properties stabilization within the temperatures of usage were the main advantages of the biocomposites comparing to the neat biopolyethylene.

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1. Introduction

For several years now biocomposites are gaining more and more interest among scientists and manufacturers and during that time a great number of papers have been published concerning processing, mechanical, thermal and other physical properties of such materials and the changes of the properties under various conditions. Great majority of such previous and present research focused on the composites with the matrix of biobased biodegradable polymers or traditional petrochemical plastics filled with natural fibres. Natural fibres have a lot of advantages which make them suitable for the use as a substitute of glass fibres, such as high specific strength and modulus and low environmental impact determined by using LCA (Life Cycle Assessment) methodology [1,2]. The choice of non-biodegradable polymers from renewable resources as matrices of biocomposites is a further step in producing new, light, eco-friendly materials. At present such non-biodegradable biopolymers comprise over 56% of biopolymer market (European Bioplastics data, 2012). With the advance of knowledge and technology leading to a development of the industry of green polymer synthesis (e.g. in Braskem, Evonik, BASF, DSM, EMS, Toray), we can now design fully biobased structural

composites, competitive for traditional plastics. One example of such materials are composites with biopolyamide matrix obtained from castor oil [3]. However, for structural Natural Fibre Composites (NFC) and Wood Plastic Composites (WPC) an obvious choice of the ‘green’ matrix seem to be a biobased polyolefin. Polyolefins basing on bioethanol are already present on the market (biopolyethylene) or will be launched shortly. Although there is limited number of these grades present today, their properties are similar to those of the counterparts [4].

Until now only few papers regarding biobased polyethylene composites or blends were published [4,5]. Castro et al. tested composites of HDPE from sugarcane ethanol and lignocellulosic curaua fibres in flexural, impact, DMTA, TG and DSC tests. There hydroxyl-terminated polybutadiene (LHPB) usually added as an impact modifier was also used as a compatibilizer agent. The presence of the curaua fibres enhanced some of the polyethylene properties, such as its flexural strength and storage modulus and LHPB addition was found to improve impact strength of the composites [5].

A large number of publications on WPC or NFC concern virgin or recycled petrochemical polyolefin matrix in regard to mechanical properties and the influence of different factors on the properties [6–13]. Usually, for the low filler content and for the fillers in the shape of particles rather than fibres (e.g. wood flour) the authors observed an increase in elastic modulus with no or minor improvement in strength and sudden decrease in deformability and impact

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strength [6,7,12,13]. Thermal and thermomechanical properties of such composites were also discussed in the literature – TG and DSC and DMTA test results were presented by different authors [14–17]. Thermogravimetric test results of WPC or NFC generally show that the composites have lower thermal stability than neat polymer, mainly due to the presence of hemicelluloses and lignin in the chemical composition of the fillers [16]. That hinders processing but within the operating temperature range the influence of such fillers on resistance to deformation on heat and on stabilization of mechanical properties with changing temperature may be positive.

A well-known disadvantage of natural fibres as polymer fillers for structural application is their high water absorption which in case of PE-based WPC or NFC makes non-hygroscopic material hygroscopic [9,17,19–21]. That leads to dimensional changes, accelerated ageing and it immediately affects mechanical properties [8,10,21–24]. Panthapulakkal et al. who measured flexural modulus and strength of HDPE/rice husk (65 wt.%) composite found that both of those parameters decreased significantly after about 67 days of soaking in water [23]. The authors explained that the decrease may be due to internal stress developed inside the composite because of the water-swollen filler, degradation in the interfacial adhesion formed between the filler and the matrix, and degradation of the filler as a result of long-term water absorption. The conclusions were similar in many other research, e.g. in Espert et al. work where injection moulded polypropylene with 10–30 wt.% lignocellulosic fillers were tested [24]. It is expected that the strength properties of NFC in general would decrease with increased water uptake because of the reasons mentioned, however the modulus of elasticity may also change in another direction (see: Results and discussion Section 3.2).

Most of the studies on petrochemical polyolefine-based biocomposites conducted during the last two decades, including those already cited in the text, were focused on the hydrophilic fibre – hydrophobic matrix interactions and the strategies to improve interfacial adhesion in this system [11,14,16–18,24–27]. However, thinking of potential industrial applications, those methods that lead to an enhancement of the composites properties (e.g. silanes, maleated polyolefins, mercerization) have serious drawbacks, as they require additional and costly operations and may not always be ecologically acceptable [28]. In practice, the producers are likely to order ready-to-use filler modified for the use with polyolefins but, depending on the application of a composite part, availability and price of the filler, it can be more economic to use unmodified one.

In the present work mechanical properties and the influence of water absorption and temperature on different biocomposites on biobased polyethylene matrix filled with lignocellulosic or mineral fillers are presented. The aim of the research was to evaluate the properties in terms of structural applications for the fully biobased composites obtained at reasonable price with ready-to-use lignocellulosic fillers for thermoplastics or with fibres supplied without additional and costly chemical modifications. As an addition to lignocellulosic fillers, natural inorganic filler in the form of crushed and milled tuff rock was also used in the study and presented as a potential interesting filler of a polyolefin matrix.

2. Materials and methods

2.1. Materials

The materials presented in this study are the composites with biobased and non-biodegradable matrix. Green PE SHC7260 a high density biopolyethylene produced from sugarcane-based ethanol was supplied by Braskem, Brazil. Green polyethylene (HDPE, LDPE and LLDPE) from Braskem is produced from December 2010 and in

2012 it comprised of 14.3% of the whole biopolymers market (European Bioplastics data). Presently it is used mainly in packaging applications.

There were four different fillers (25 wt.%) used to modify the properties of biopolyethylene matrix: wood flour, kenaf fibres, cellulose powder and tuff particles. Wood flour Lignocel BK 40/90 from soft wood (spruce) with particle size 300–500 μm and ultra-fine cellulose ARBOCEL UFC 100 in a form of powder (approx. 8 μm diameter) were supplied by J. Rettenmaier & Söhne (JRS) company, Germany. The fillers were prepared for processing with thermoplastic materials by JRS. Chopped kenaf fibres were prepared in the Institute of Natural Fibres, Poznan, Poland and they were not chemically modified. Tuff particles of particles size 15–50 μm were prepared in the Institute of Materials Engineering of Cracow University of Technology [29]. Filipowice tuff was mined in Poland and then crushed, milled and calcined at 800 °C for 2 h.

Standard dumbbell type specimens (10 × 4 × 150 mm) were produced in Grupa Azoty in Tarnow, Poland, in a two step process. First, composite pellets were obtained by compounding extrusion using two-screw extruder MARIS TM 30VI with a gravimetric twin screw feeder (cylinder temperature: 100 °C – zone 1, 130 °C – zones 2–10, screw rotation: 60 rpm) and then injection moulded using Engel ES 200/40 HSL. The parameters of the injection moulding process are shown in Table 1. All tested materials are characterized in Table 1 with their acronyms used further in the text.

2.2. Methods

Mechanical properties were estimated by a tensile test (EN ISO 527) and a three-point flexural test (EN ISO 178), with an universal testing machine Insight 50 MTS with MTS axial extensometer with a constant crosshead speed of 10 mm/min. Modulus of elasticity (E_t), tensile strength (σ_M), strain at break (ϵ_B) as well as flexural modulus (E_f) and stress at 3.5% strain (σ_f) were determined. Tensile tests were carried out under standard conditions and at –23 °C and 80 °C using Instron thermal chamber. These values of lowered and elevated temperatures were taken from low and high limits of standard temperatures at which HDPE and its composites work in structural applications (e.g.: plastic pallets, car panels).

SEM images were acquired on the gold-sputtered tensile-test fracture surfaces of specimens using JEOL JSN5510LV.

Charpy impact strength of notched specimens (a_{cN}) was measured using Zwick HIT5.5P under standard conditions.

Materials density (ρ) was measured by hydrostatic method. Vicat softening temperature (VST) was measured according to ISO 306 under 50 N loading and with 50 °C/h heating rate using CEAST machine. Absorption of water (20 °C) was calculated after 1, 7, 30, 240 days of soaking, according to PN-EN ISO 62:2000. To determine the influence of water uptake on mechanical properties and on the surface quality, tensile test was performed again after the 7, 30 and 240 days of incubation and the surface roughness average (R_a) was measured using profilometer Mitutoyo SJ-301.

DSC tests were performed at Poznan University of Technology, Faculty of Chemical Technology, Poland using NETZSCH model DSC-200 with computer software for test analysis. The measurements were made on the samples of 7–7.5 mg obtained from a central part of the injection moulded standard dumbbell-shape specimens in the temperature range between 40 and 190 °C under argon atmosphere. All measurements were taken according to the following program: heating between 40 and 190 °C at a scanning rate of 10 °C/min and cooling between 190 and 40 °C at a scanning rate of 5 °C/min. The whole process was carried out twice to analyse processing memory/history of the materials (the first heating–cooling cycle) and the thermal properties of the composites (the second heating–cooling cycle). An empty pan was used as a reference.

Table 1
Injection moulding parameters.

Material	Filler (wt.%)	Temperature (°C)			Time (s)				Pressure (bar)		
		Cylinder	Die	Mould	Holding	Cooling	Injection	Plastification	Injection	Holding	Plastification
BPE	-Neat biobased HDPE	160	160	40	30	20	0.83	8.2	110	110	6
BPEM	Wood flour, 25	160–170	175	40	30	20	1.87	7.1	100	100	6
BPEC	Ultrafine cellulose powder, 25	160–170	175	40	30	20	1.55	7.5	100	100	6
BPEK	Kenaf fibres, 25	160–175	180	40	30	20	1.95	7.0	100	100	6
BPET	Tuff microparticles, 25	160–175	180	60	30	20	1.30	7.7	100	100	6

Table 2
Basic physico-mechanical properties of the materials.

Index	ρ (g/cm ³)	E_t (MPa)	σ_M (MPa)	ε_B (%)	E_f (MPa)	σ_f (MPa)	a_{cN} (kJ/m ²)	VST (°C)
BPE	0.942	1140 ± 6	23.8 ± 1.2	500 ± 30	1078 ± 19	20.4 ± 0.3	4.9 ± 0.1	122
BPEM	1.022	2740 ± 219	23.8 ± 0.1	6.5 ± 0.2	2484 ± 77	35.6 ± 0.3	5.2 ± 0.2	124.3
BPEC	1.006	2250 ± 79	23.5 ± 0.3	18 ± 0.7	1942 ± 72	31.6 ± 0.1	3.2 ± 0.1	123.2
BPEK	1.025	2630 ± 171	22.6 ± 0.8	5.8 ± 0.3	2667 ± 15	36.3 ± 0.2	4.3 ± 0.1	124.0
BPET	1.119	1720 ± 67	21.8 ± 0.5	48 ± 3.4	1728 ± 12	28.6 ± 0.1	3.0 ± 0.1	123.8

3. Results and discussion

3.1. Mechanical properties and structure

Tensile properties, results of bending test and impact strength of tested materials are shown in Table 2. Tensile modulus increased when adding fillers, especially for wood flour and kenaf fibre (approx. 2.4-fold) comparing to the neat biopolyethylene. There was a similar effect of enhancement of flexural modulus for the composites. Tensile strength of all tested materials remained at similar level while bending stress at 3.5% strain increased with the addition of fillers of approx. 170%. Wood flour and kenaf fibre addition caused significant decrease in strain at break, what was expected because of the shape and size of the fillers. High deformation ability was preserved for the composites with microparticles of tuff and ultrafine cellulose powder. In notched Charpy impact test results, though the decrease in the impact strength could be noticed in most cases, the change of the property was not very significant for the composites comparing to neat biopolyethylene, which was a positive result. An increase in impact strength was observed for the composite with wood flour.

In Fig. 1 fracture surfaces of the tested composites are shown. Each SEM image shows the morphology of the fillers (different magnifications) and gives some information about the filler–matrix interactions. There is no visible evidence of pull-outs and voids between the matrix and short lignocellulosic fillers. The observations of BPET composite were done on the specimens broken in liquid N₂ because the fracture surface of BPET tested at room temperature was highly irregular and developed and so it was difficult to acquire any useful image.

3.2. Water absorption influence and surface quality

The results of water absorption after 1, 7, 30, 240 days of soaking for the tested materials are presented in Fig. 2. Hygroscopic lignocellulosic fillers caused noticeable increase in the parameter value. Introductions of untreated kenaf fibres resulted in the highest water uptake (5 wt.% after 240 days). There was no statistic difference between the neat biopolyethylene and its composite filled with mineral tuff filler.

High water absorption of lignocellulosic fillers resulted in an increase in surface roughness of the composites which can be seen in Fig. 3 showing the results of surface roughness average at initial

state and after 240 days of soaking. The surface quality was most significantly changed for the composites filled with wood flour (large particles of irregular shape). The smallest changes of the surface roughness were observed for the composites filled with tuff.

Changes in tensile properties due to long-lasting soaking in water were not large, but can be seen with some regularity. Reduction in tensile strength was noticed for pure polyethylene (approx. 2 wt.% after 240 days of soaking) and for its composites (the highest decrease of approx. 6 wt.% was observed for wood flour composite BPEM). The modulus of elasticity (Fig. 4) of neat polyethylene decreased by 15% after 240 days of soaking in water but the addition of fibres and particles caused the increase in E_t of about 10% for its composites. This phenomenon can be explained due to development of internal stress caused by the swelling of the filler, which is in agreement with [23]. Only for the addition of unmodified kenaf fibre, modulus of elasticity was reduced after 240 days of soaking and there was also the highest change in tensile strength by nearly 11%.

3.3. Changes in mechanical properties under temperature

The results of tensile test at –23 °C and at 80 °C were compared with the properties measured at room temperature and presented as percent changes in tensile strength and in tensile modulus (percent increase at lowered temperature and decrease at elevated temperature) in Figs. 5 and 6. The highest change in modulus of elasticity was observed for neat biopolyethylene both in high and low temperatures (140% at –23 °C and 75% at 80 °C). Addition of lignocellulose fillers resulted in stabilization of the property. Small microparticles of the fillers like ultrafine cellulose or tuff affected the stabilization of E_t or σ_M to a lesser extent than wood flour or kenaf fibres. For the composites with lignocellulosic fillers, especially for wood flour composite (BPEM) the value of the modulus of elasticity at 80 °C was on the similar level as for the neat BPE tested at room temperature (1140 MPa). The differences in the tensile strength between the composites and neat biopolyethylene were not significant.

Additionally, to compare the ability of the materials to perform at elevated temperatures Vicat softening point was measured and the results are presented in Table 2. With the introduction of cellulose fibres the enhancement of VST value was noticed to be higher than in the case of the addition of tuff filler comparing to neat biopolyethylene.

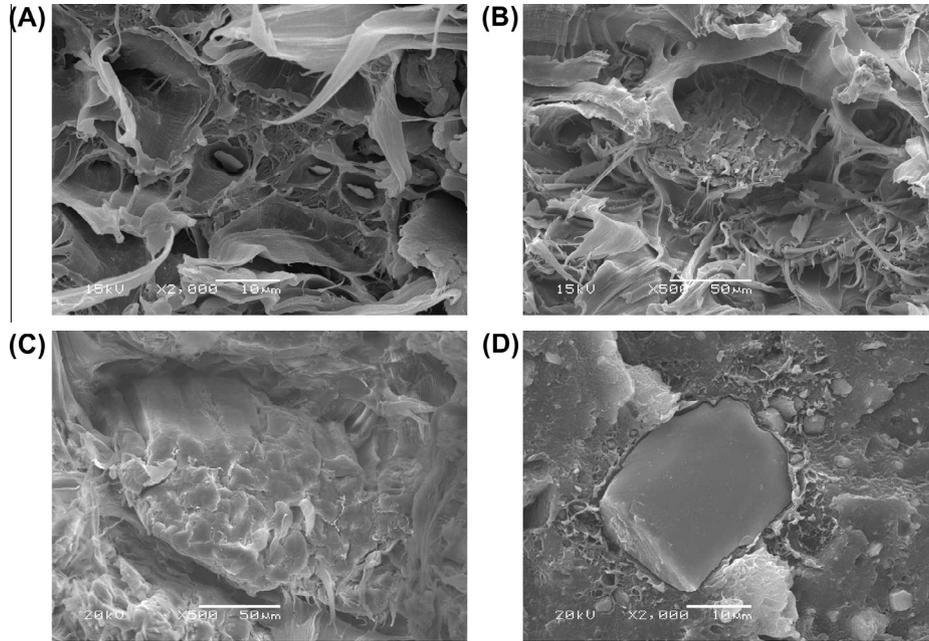


Fig. 1. SEM images of tensile test fracture surfaces of on BPE filled with 25 wt.% of: (A) ultrafine cellulose, (B) kenaf fibres, (C) wood flour, (D) tuff microparticles.

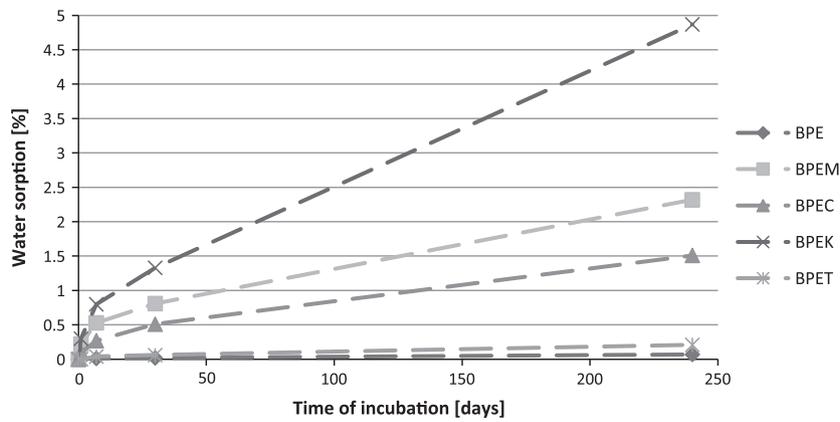


Fig. 2. Comparison of absorption of water for BPE and tested composites.

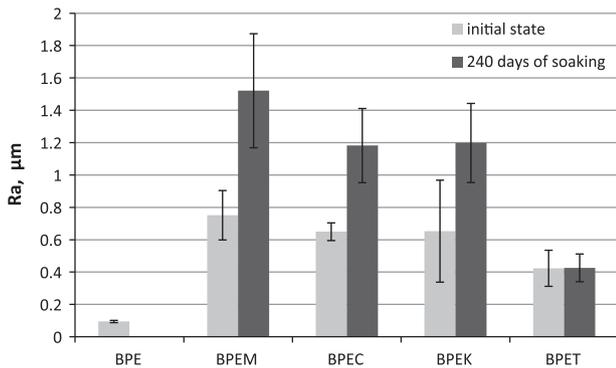


Fig. 3. Average surface roughness of the tested materials in initial state and after 7, 30 and 240 days of soaking in water.

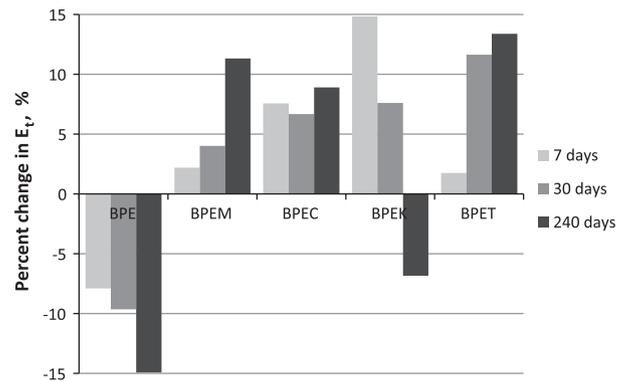


Fig. 4. Percent change in tensile modulus for increasing time of soaking in water.

3.4. DSC analysis

The values of the melting and crystallization point for biopolyethylene (reference material) and the composites recorded in both

heating–cooling cycles are shown in Table 3. The DSC curves obtained for the tested materials in the second heating–cooling cycle are shown in Fig. 7. Analysis of the results showed that the introduction of the cellulose to the biopolyethylene matrix did

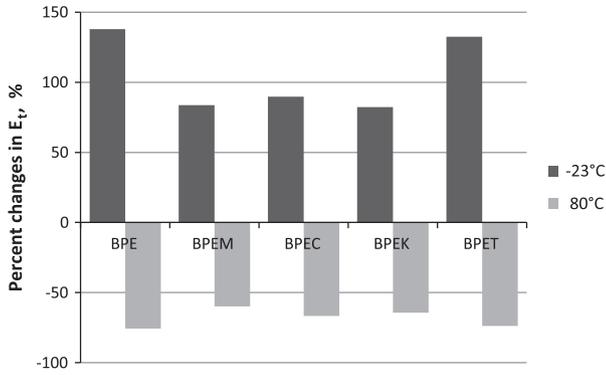


Fig. 5. Percent change in tensile modulus at $-23\text{ }^{\circ}\text{C}$ and $80\text{ }^{\circ}\text{C}$ compared to E_t at room temperature.

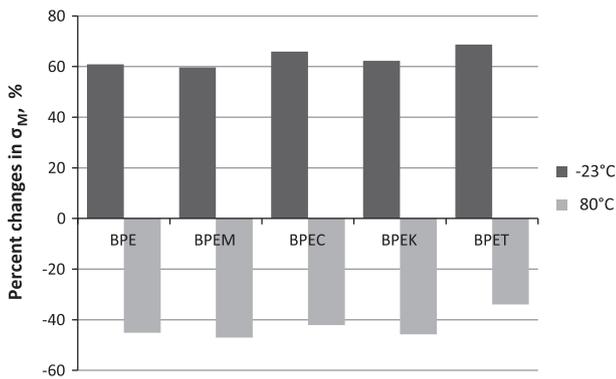


Fig. 6. Percent change in tensile modulus at $-23\text{ }^{\circ}\text{C}$ and $80\text{ }^{\circ}\text{C}$ compared to E_t at room temperature.

Table 3

Temperatures of melting (T_m) and crystallization (T_c) and values of ΔT for tested materials (1 – first heating–cooling cycle, 2 – second heating–cooling cycle).

Material	T_{m1} ($^{\circ}\text{C}$)	T_{m2} ($^{\circ}\text{C}$)	T_{c1} ($^{\circ}\text{C}$)	T_{c2} ($^{\circ}\text{C}$)	ΔT_1	ΔT_2
BPE	138.4	137.9	117.6	117.5	20.8	20.4
BPEM	136.4	135.9	117.3	117.2	19.1	18.7
BPEC	138.6	137.4	117.1	117.0	21.5	20.4
BPEK	135.3	136.0	117.4	117.4	17.9	18.6
BPET	135.0	135.4	118.9	118.6	16.1	16.8

not change the melting temperature and the crystallisation of the composite comparing to the reference sample in any significant way. The three other fillers: wood flour, kenaf and tuff caused a slight decrease in the melting point (T_m) of the composites in comparison to BPE. These changes, however, were minor and in the range of $1.9\text{--}3.4\text{ }^{\circ}\text{C}$. It was noticed during analysing the crystallization temperatures (T_c) of the composites that only in the case of tuff filler there was a noticeable increase in the value of T_c comparing to the reference material (of 1.3 and $1.1\text{ }^{\circ}\text{C}$).

Studying melting and crystallization points and their impact on the performance of the injection moulding process it was noted that the introduction of fillers into the BPE matrix, despite the small changes of T_m and T_c , affected the value of ΔT , defined as the difference between the melting point and crystallization ($\Delta T = T_m - T_c$). The smaller is the difference the faster the crystallization process occurs during processing, which in turn contributes to the shortening the manufacturing process. The values of ΔT for the tested materials are summarized in Table 3. It can be seen that use of cellulose powder did not change the ΔT value in reference to BPE. In the case of the other three fillers, these changes were more noticeable. The use of wood flour and kenaf resulted in a reduction of the ΔT of 1.7 and $2.9\text{ }^{\circ}\text{C}$ respectively. The most promising results were obtained for the composite with the tuff filler (BPET). Here the values of ΔT were lower than for BPE of as much as $4.7\text{ }^{\circ}\text{C}$ and $3.6\text{ }^{\circ}\text{C}$ for the first and second heating–cooling cycle respectively. The results therefore suggest that the use of such fillers as wood flour, kenaf and tuff as biopolyethylene fillers is justified not only in terms of the performance of the composites, but also in terms of economics of the manufacturing process.

4. Conclusions

For the tested composites of biobased HDPE with low filler content (25 wt.%) produced by compounding extrusion followed by injection moulding, an advantageous effect of the increase in stiffness, flexural stress at 3.5% strain, Vicat softening temperature, thermal properties stabilization, small changes in the density, and some promising result of DSC analysis suggesting the decrease in time of crystallization process should be emphasized. Those enhancements are important advantages that predispose these materials for structural applications of limited contact with moisture and water environment. The effects of reduction in elongation at break or high water absorption causing deterioration in surface quality were expected because of the fillers shape and dimensions and their hydrophilic and hygroscopic nature which is difficult to modify.

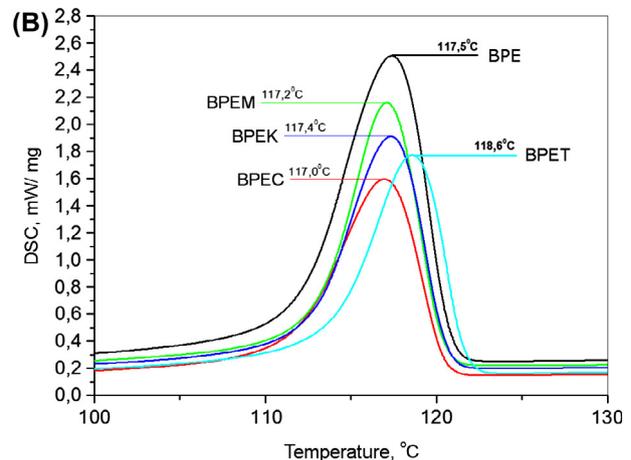
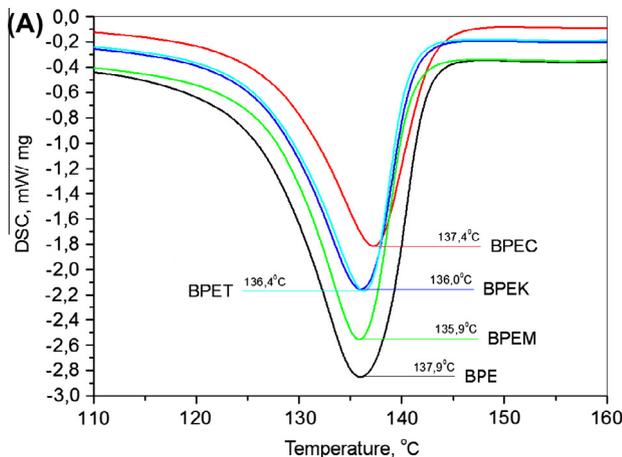


Fig. 7. DSC heating curves (A) and cooling curves (B) for the tested materials in the second heating–cooling cycle.

Among the materials tested, the composition with wood flour addition seems advantageous in respect to mechanical properties (increase in stiffness, good impact properties) and stabilization of the properties at lowered and elevated temperatures, as well as in resistance to deformation on heat. Unfortunately, at the same time the composition is the most prone to dimensional changes caused by water uptake of all tested materials. The influence of wood flour and kenaf on the properties of biobased HDPE is similar while wood flour is a cheaper solution. However, in applications where lower increase in stiffness is acceptable, fine cellulose microparticles or tuff microparticles addition to biobased HDPE may be also beneficial. The compositions preserve high elongation at break, lower water uptake and they are easier to process than the composites with wood flour or kenaf fibres (easier feeding, more homogeneous mixing and for tuff filler – faster crystallisation process).

Addition of natural fillers to biobased polyethylene allows us to produce light-weight, structural, eco-friendly products with improved stiffness at relatively low price. Employing both matrix and reinforcing materials obtained from renewable plant sources and replacing traditional petrochemical plastics and materials which hinder utilization process such as glass fibres, is an important environmental benefit.

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