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ORIGINAL PAPER

Relationship Between the Shape of Giant Reed-Based Fillers and Thermal Properties of Polyethylene Composites: Structural Related Thermal Expansion and Diffusivity Studies

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Abstract

This paper describes the effect of two different fillers derived from giant reed (*Arundo donax* L.), namely fibers and shredded aerial parts of the plant, on the thermal properties of polyethylene-based composites, as the analysis of dimensional stability of lignocellulose-based composites, and its relationship with their thermal diffusivity, has not yet been assessed in the literature. It has been found that the introduction of such materials resulted in a significant reduction of the coefficient of thermal expansion, particularly more important in the case of fibers, due to their higher aspect ratio; in particular, this coefficient is reduced to less than half for fibrous composites (from $1.6 \cdot 10^{-4}$ K^{-1} to $6.1 \cdot 10^{-5}$ K^{-1} or $3.5 \cdot 10^{-5}$ K^{-1} for 20 and 40% loadings). This parameter also influences the thermal diffusivity of the final parts; the diffusivity of composites increases with the ratio of lignocellulosic filler used, particularly when using fibers, due to a better orientation of these fibers than the shredded material, which does not exhibit a fibrous shape. Composites with 20% share of the filler exhibited a thermal diffusivity increased by about 15% compared to neat polyethylene, while 40% loadings resulted in a 25% and 60% increase for ground and fibrous materials, respectively. These results provide additional features to lignocellulose-composites characterization, providing properties not usually mentioned in the literature to expand the knowledge about such composite materials beyond mechanical properties, providing a broader range of properties to offer a wider application area of such composites.

Statement of Novelty

Arundo donax L. is of great interest to biorefineries due to its fast growth and resistance to adverse environmental conditions. Most research on this plant species focuses on obtaining energy products or valuable chemicals, while very few are related to composite production, particularly on thermoplastic materials. The work found in the literature so far does not provide insights into the relationships between the types of filler (and their aspect ratio) and their thermal properties. Therefore, this work expands the knowledge on the thermal behavior of lignocellulose-polymer composites, supplementing the research, usually focused only on mechanical properties, in their characterization by correlative analysis of thermal influenced dimensional change with structure and thermal diffusivity. Determining the coefficient of thermal expansion (CTE) is a relevant parameter to assess the possibilities of using a material at high or low temperatures and evaluate the dimensional stability of a product during its service lifetime. On the other hand, thermal diffusivity brings together the capacity of a material to store thermal energy and distribute it throughout the material; that is, it relates heat capacity and thermal conductivity, which are also essential in using materials in market applications. Therefore, the work not only

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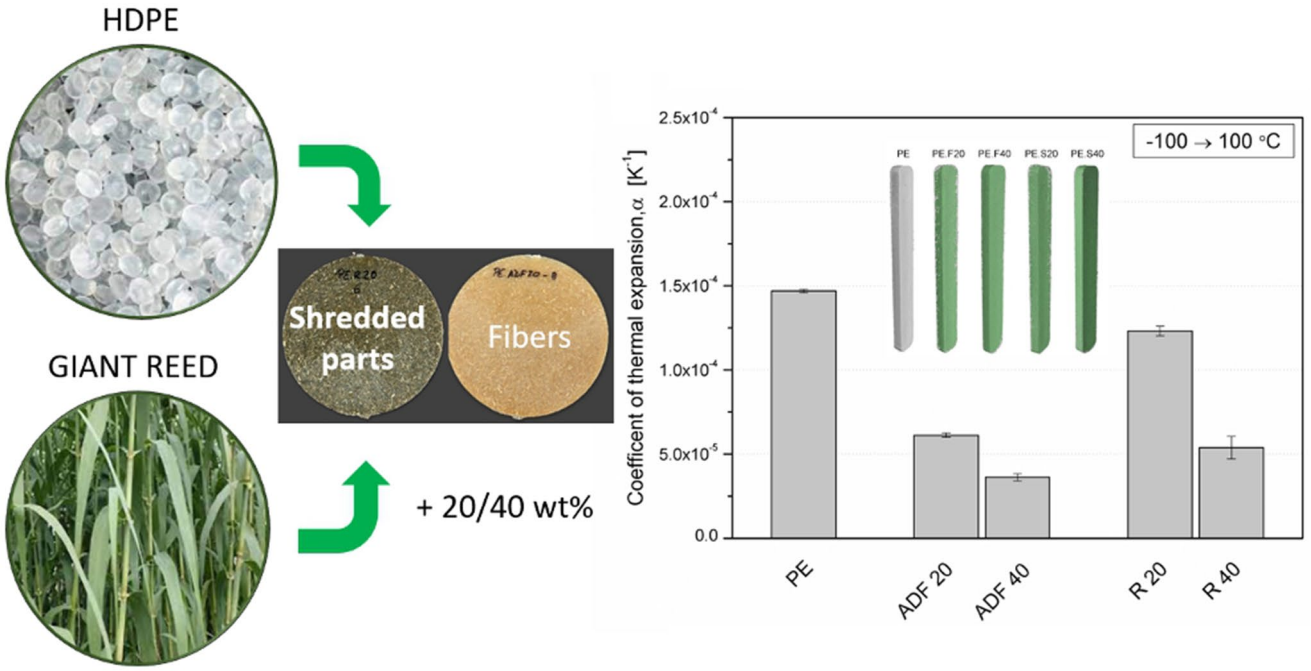
provides the results of thermal diffusivity and CTE of thermoplastic-reed composites but also correlates both parameters as a way to widen the range of application of plant-based composites in areas where dimensional stability (i.e., low thermal expansion) is required.

Graphical Abstract



Keywords Giant reed · Natural filler · Thermal diffusivity · Thermal expansion · Polyethylene

Introduction

A pivotal focus on environmental responsibility and recycling principles has gained prominence in the quest for sustainable composite materials. Initiatives driven by a shared commitment to environmental stewardship have marked notable progress in utilizing plant-based fillers and the development of composites with reduced polymer content [1]. As researchers worldwide intensify efforts towards innovative solutions for the introduction of waste streams, this paper contributes to the scientific discourse by exploring the thermal behavior of polymer composites containing lignocellulose fibers as a way to widen the knowledge about such materials and increase their uptake in commercial applications. Such composites are commonly referred to as natural fiber composites (NFC) or wood polymer composites (WPC) [2–4]. Besides the often used long natural fibers, mainly in the implementation with thermoset polymers [5, 6], many studies focused on composites reinforced with plant particle-shaped fillers or short fibers [1, 7, 8]. Current research activities increasingly focus on attempts to completely valorize plant parts and give each waste-based filler a dedicated function depending on its specific physicochemical, mechanical, or chemical characteristics [4, 9].

Apart from the understanding of the effect of lignocellulose fibers within polymer matrices in the mechanical behavior or durability of materials, the determination of the coefficient of thermal expansion (CTE) is a relevant parameter to determine the possibilities of using a material at high or low temperatures, as the dimensional change resulting from the thermal expansion phenomenon can be undesirable. On the other hand, thermal diffusivity is related to the capacity of a material to store thermal energy and distribute it throughout the material; that is, it relates heat capacity and thermal conductivity. It is then advisable to correlate thermal expansion in detail with the ability to accumulate and distribute heat due to the high requirements for polymeric and composite products regarding dimensional stability during exploitation. In most cases of injection-molded products, minimizing the thermal expansivity of final products is crucial [10]. This phenomenon is related to the polymer structure's heterogeneity and phase transitions [11]. As shown in previous studies, the crystallinity and the introduction of fillers result in significant changes in the thermal expansion of products made of thermoplastic polymers

[11–13]. The most effective and straightforward method for obtaining good dimensional stability in a polymeric composite is introducing a filler with high-temperature conductivity [14]. While the relationships between the role of the filler orientation in the polymer matrix and the interfacial bonding have been described for inorganic fillers [15, 16], these studies are strongly limited in the case of composites with plant-based fillers. The influence of the shape factor of natural plant fillers on the mechanical properties and structure of composites based on thermoplastic polymers has been studied previously [17, 18]; however, the correlations between their thermal properties and temperature-assessed dimensional stability should still be explained, considering broad aspects of natural fillers' structure.

Biodiversification and the diversity of plant structure, influenced by species characteristics and environmental conditions accompanying growth, is often a significant objection to using plant-derived fillers [19–21]. It is advisable to correlate the chemical composition of plant-based fillers with the final properties of their composites and consider the impact of grinding processes and their shape [22]. Therefore, it becomes purposeful and justified to conduct research in the field of pre-testing the broadest possible range of composites reinforced with natural-based fillers to define the crucial aspects determining their role in modifying polymeric composites' thermal behavior.

Giant reed (*Arundo donax* L.) is considered a promising crop for obtaining bioenergy and bioproducts, including natural fibers for composites production [23–27], following a biorefinery scheme. The interest in using this plant relies on its fast growth and resistance to adverse environmental conditions, such as water scarcity or poor quality. Most research on this plant species focuses on the obtaining of energy products (ethanol or biogas) or valuable chemicals (levulinic acid or oligosaccharides), while very few are related to the obtaining of composites, particularly on thermoplastic materials [27–32]. This plant can be grown in marginal or polluted lands, with low-quality waters and low fertilizers or pesticides use, which could imply a vital step within the establishment of an industrial crop to produce valuable products without replacing food crops or using valuable inputs (water, land) for their growing, thus contributing to the achievement of the sustainable development goals (SDG). This research work compares the behavior of the neat matrix (high-density polyethylene, HDPE) with composites loaded at 20 and 40% in weight of two different fillers: fibers obtained from the culms of the giant reed through a chemo-mechanical process and ground stems and leaves, as a more inexpensive material with almost no processing, as described in our previous works [32, 33]. This work allows expanding the knowledge on the thermal behavior of polymer composites containing lignocellulose fibers, and particularly, it constitutes a supplement realized research in the manufacturing and characterizing *Arundo donax*-based composites by correlative analysis of thermal influenced dimensional change with structure and thermal diffusivity.

Materials and Methods

Materials and Sample Preparation

A high-density polyethylene (HDPE) purchased from Total (Antwerpen, Belgium) with the trade name HD6081 was used as a polymeric matrix. This HDPE is characterized by a 0.960 g/cm^3 density and 8 g/10 min (190 °C/2.16 kg) melt flow index (MFI).

Natural fillers used for manufacturing the composites were produced from the same plant part, i.e., *Arundo donax* L. culms (from Gran Canaria island, Spain). The fillers used are fibers and shredded material. Shredded filler was obtained by grinding aerial plant parts (leaves, culms) and washing them with water. Fibers were obtained after soaking culms in a NaOH (from Sigma Aldrich, Missouri, USA) solution for around one week, with further processing by a series of rolling mills to separate the fibers from the softer material. A full description of the manufacturing procedure was previously given [33].

The samples were named appropriately in the work regarding their material composition and filler concentration, as follows: PE for unmodified polyethylene, PE.F20 and PE.F40 for composites with 20 and 40 wt% fibers, and PE.S20 and PE.S40 for composites with shredded *Arundo*.

Lignocellulosic fibers were dried overnight at 105 °C before compounding, while HDPE was dried overnight at 60 °C before melt processing. Composites were melt-mixed in a ThermoScientific Process11 (Massachusetts, USA) co-rotating twin-screw extruder with a temperature profile set up 170-175-175-185-185-175-165–165 °C and screw rotation speed of 100 rpm, cooled in a water bath and pelletized. Samples were formed by injection molding through an Arburg 320 S hydraulic injection molding machine (Lossburg, Germany). Prior to processing, pellets were dried at 60 °C overnight with dry air (dewpoint of -40 °C). From the back to the nozzle, the following temperature profile was used: 175-180-185-185-190 °C, while the mold temperature was 30 °C, and the cooling time was 15 s. The back pressure was 5 MPa, and the holding pressure was 50 MPa. The aspect ratio of both fillers was determined by optical means [34] for the injection molded composites; it was found to be about 1 for composites with shredded material and over 4 for those with fibers, with sizes of 0.65 mm length for the fibers and 0.23 mm for the shredded material [35].

Methods

The composite samples' structure was examined using a measuring X-ray tomography, model v|tome|x s240 (Waygate Technologies / GE Sensing & Inspection Technologies GmbH, Pennsylvania, USA). Micro-computed tomography (µCT) was used to evaluate the filler distribution in injection-molded samples and their porosity. The following parameters were used during the measurements: X microfocus x-ray tube (voltage 150 kV/current 200 µA), exposure time of 150 ms per picture, and voxel size of 123 µm.

The thermal diffusivity analysis was performed using a modified Ångström method with a Maximus (Poznan, Poland) apparatus. Previous works by Prociak, Jakubowska, and collaborators [36, 37] described the experimental setup in detail. In short, this method is characterized by a convenient preparation of samples and short analysis time; one end of the sample is heated, and the temperature of the sample at two points is measured using resistance temperature detectors [36], as shown in Fig. 1.

During investigations, the micro heater was charged with 23 V to heat the samples for 400 s. Thermal diffusivity (D) is

Fig. 1 Experimental setup for the measurement of thermal diffusivity

dependent on thermal conductivity (λ), specific heat capacity at constant pressure (c_p), and density (ρ) [37], defined by the following formula:

$$D = \frac{\lambda}{c_p \cdot \rho} \quad (1)$$

The specific heat capacity determination was realized according to the DIN 51,007 standard with differential scanning calorimetry (DSC) measurements, conducted using a Netzsch DSC 214 Nevio apparatus (Selb, Germany) with aluminum crucibles and 20 ± 0.1 mg samples under nitrogen flow. All samples were heated with a constant heating rate of 10 °C/min from − 50 to 200 °C and held molten for five minutes. For the test, sapphire references have been used.

Thermal expansion analyses were performed on the Netzsch TMA 402 F1 Hyperion apparatus (Selb, Germany). The measurements were carried out in a heating cycle with a 2 °C/min heating rate with a flow of 100 ml/min protective argon atmosphere. The measurements were conducted in the temperature range of -100 to 100 °C, with an applied force of 0.01 N. The test results were analyzed using the Netzsch Proteus software. Due to changes in the linear coefficient of thermal expansion (CTE) in the tested temperature range, this coefficient was determined for the entire range and sub-ranges, respectively − 100–25 °C and 25–100 °C. These ranges were selected considering the potential applicability of composites in different service conditions, i.e., under room temperature or frozen environments and over room temperature, considering sectors such as packaging or urban furniture.

Results and Discussion

Figures 2 and 3 show the structural analysis results of 3D computed tomography of tested materials. Figure 2a shows the distribution of fibers in the volume of the injection molded samples, while Fig. 2b presents a void distribution. In the case of both fillers used, for 20 wt%, uneven filling of the volume of the composite product with a visible skin layer is visible. A more heterogeneous structure was noted for the fibrous filler (PE.F), which may be related to the more directional arrangement of the filler particles in the core of the sample. This effect is also observable in Fig. 3f. Complete measurement data associated with the presence of pores in the structure of the samples are presented in Fig. 3a-e. The observed points allowed for determining the size and volume of pores observed for a selected sample from each series. The calculated porosity in the samples was as follows: PE 0.09%; PE.F20 0.29%; PE.F40 40 0.86%; PE.S20 0.36%; PE.S40 1.26%. Composite series containing

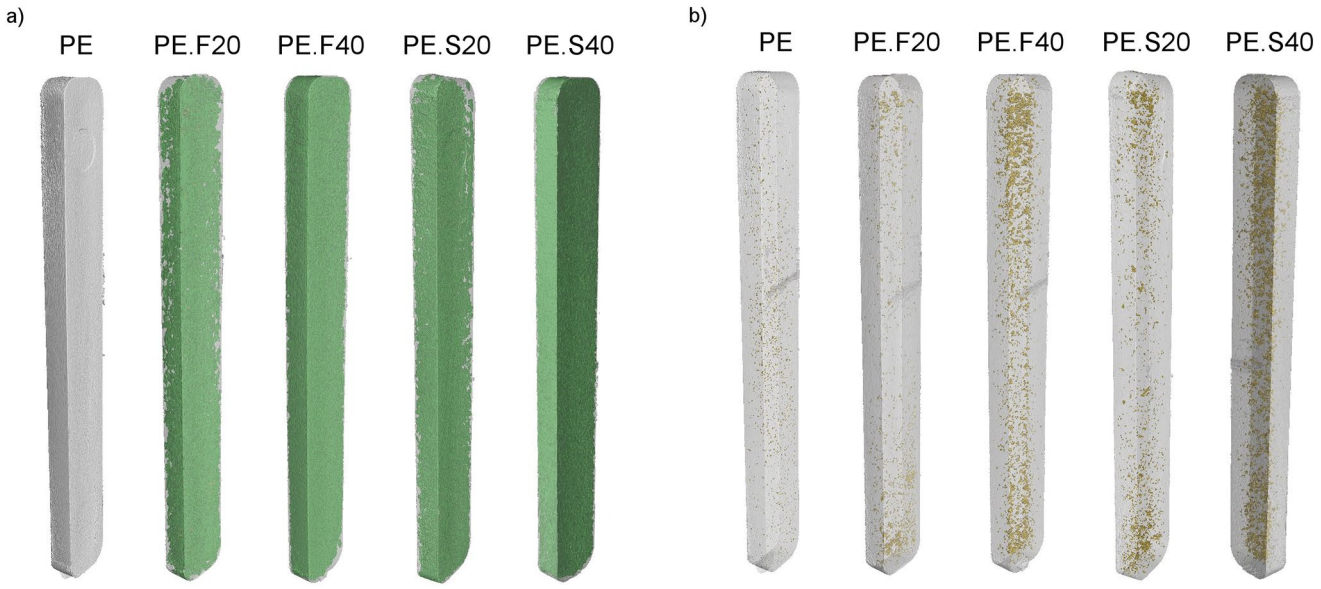


Fig. 2 3D computed tomography images presenting filler arrangement (**a**); porosity (**b**)

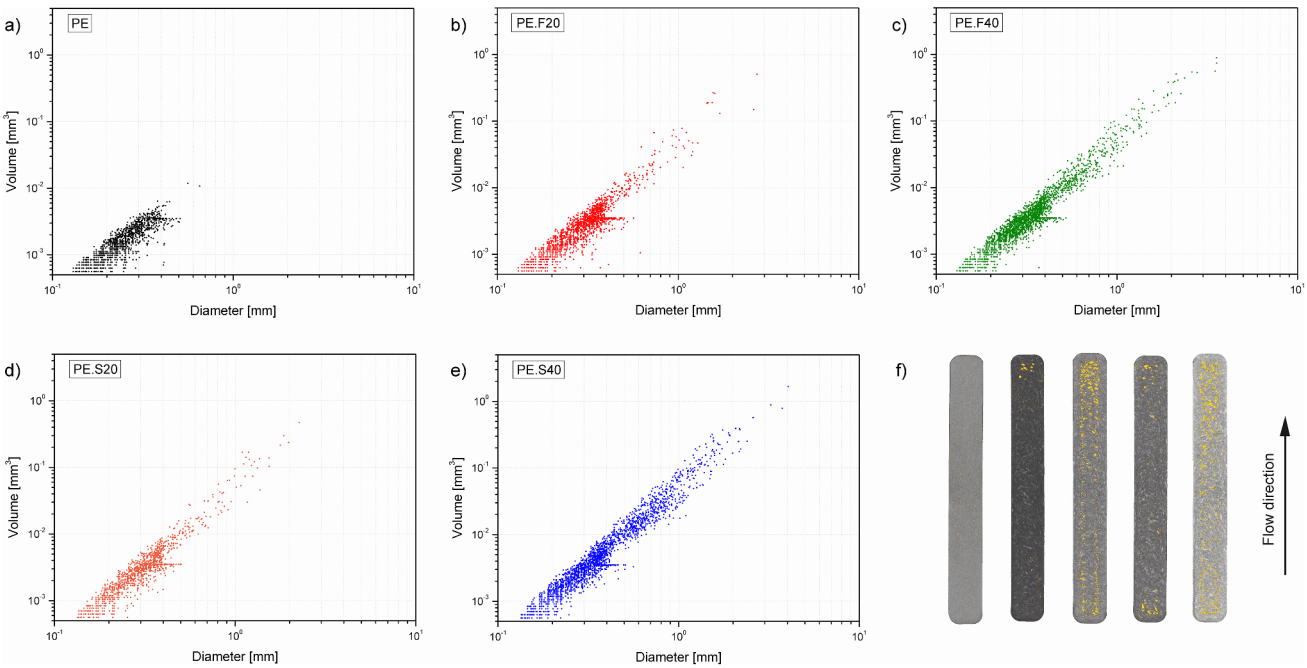


Fig. 3 Characterization of injection molded samples porosity distribution

shredded irregular particles are more porous than fiber-filled composites because of the fillers' larger specific surface area [38].

The results of TMA measurements in the form of changes in length (dL/L_0) and average values of the coefficient of thermal expansion determined in three temperature ranges (-100-25; 25–100; -100–100 °C) are shown in Fig. 4. The courses of changes in the elongation of the samples unambiguously indicate a much more favorable effect of fibrous fillers on the dimensional stability of the specimens subjected to temperature changes. For 20 wt% R-filled composites, the shape of the $dL/L_0(T)$ curve is similar to that of unmodified PE, and the average values for these materials series in all three ranges are comparable. In the case of composites filled with fibers, a change in the course of the curve and a significant limitation of α can be noted, which is probably related to the formation of a network of physical interactions between the fibers dispersed in the matrix, which significantly restricts their mobility caused by changes of polymer thermal expansion. The

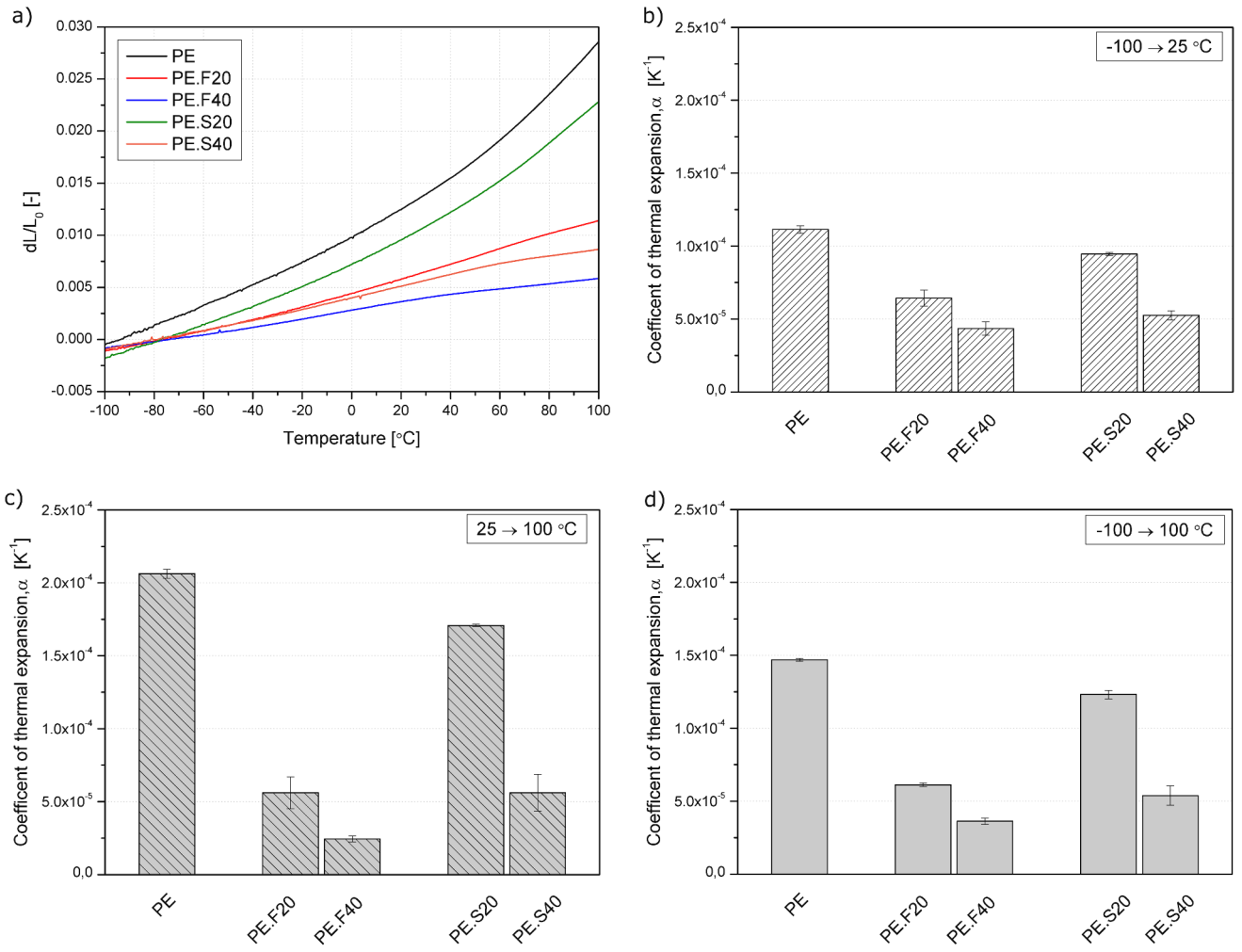


Fig. 4 Course of samples dimension change (**a**), and mean values of coefficient of thermal expansion in the temperature ranges of -100–25 °C (**b**), 25–100 °C (**c**), -100–100 °C (**d**)

sample containing 40 wt% shredded reed shows comparable average α values to PE.F20 in the 25 to 100 ºC range. In the range most important from the perspective of using PE composites (-100 to 25 °C), the most beneficial results were obtained for the sample containing 40 wt% *Arundo* fibers. The obtained research results are related to the partial orientation of the fillers with an increased shape coefficient and greater consistency for creating 3D steric hindrances in the sample volume, as found in previous work of authors [32], where the materials containing fibers showed higher values of storage modulus than those made with shredded material in the whole range of temperature studied (-100 to 100 ºC). This increased stiffness is related to the hindering of polymer chain movements due to the introduction of fibers, as also shown here for CTE. Besides, the orientation of fibers was demonstrated in capillary rheometry testing.

In addition to the arrangement of the fillers and their mutual interactions, the interphase volume surrounding the filler dispersed in the polymeric matrix plays a vital role, as demonstrated in the work of Tripathi and Dey [39]. A reduction of CTE is expected with increased interfacial adhesion between filler and matrix. A lower porosity in the injection molded samples was obtained for fibrous composites, and higher interfacial interactions also occur in such composites, demonstrated by higher mechanical properties [32]. Yang et al. [12] confirmed an additional reduction of thermal expansion of PP-wood flour composite by increasing interfacial interactions with the introduction of maleic anhydride grafted polypropylene (MAPP). Increased adhesion at the interface of PE-fiber composites, resulting from an increase in the shape factor of fillers, causes a more prominent limitation of the mobility of macromolecules and, consequently, a decrease in the coefficient of thermal expansion. These results are in agreement with our earlier work [32], in which the calculated values of adhesion factor based on DMA indicated a reduction in interactions in the temperature range of HDPE β-relaxation [40] above 0 °C for composites containing shredded part plants and better interfacial adhesion in

composites with fibrous fillers. For the automotive industry, the coefficient of thermal expansion should be lower than $5 \cdot 10^{-5}$ K^{-1}; composites at 40% loading accomplish this limit, and it is almost reached for 20% fibrous composites, meaning these composites might find an application in the manufacturing of internal door panels [10], at least for internal covering.

Measuring the thermal diffusivity of the material by the Ångström method allows for obtaining information about the sample's ability to conduct heat relative to heat storage. Figure 5 presents the mean values of thermal diffusivity and specific heat capacity of injection molded polyethylene and composite samples. Thermal diffusivity gradually rises for composite samples with increasing filler content, with a more distinct effect observed for the PE.F40 series. Increasing the share of lignocellulosic filler translated into increased pore content, resulting in reduced thermal conductivity, as also found by Prisco [41]. Thermal diffusivity was found to decrease with increased content of banana fiber in a PP matrix, while no specific trend is seen for Cp. In this work by Paul et al. [42], the density increases with the fiber loading, although porosity values were not measured. This might explain the opposite behavior found; a higher porosity of composites would result in reduced thermal conductivity and diffusivity. Considering Eq. (1), based on the results D, it can be concluded that the probably dominant effect was replacing a significant part of the polymer by volume with the natural filler, which decreased specific heat. The introduction of the oriented filler with an increased aspect ratio (fibers) probably increased the contact surface of the oriented fiber fillers in the flow direction, which resulted in a change in thermophysical parameters and a more pronounced impact of this filler on D. The results are contrary to those presented by Kalaprasad et al., [43] who found that sisal fibers had a marginal effect on the change in thermal resistance, while in the same temperature range test used in the Ångström method, LDPE-sisal composites were characterized by reduced thermal diffusivity. The difference in the conducted tests resulted mainly from the method of sample preparation, forced significant orientation in the case of work results, and the lack of porosity resulting from process features. Changes in thermal diffusivity are related to the free path in the material; the presence of porosity and other structural defects will change this parameter. The presence of pores in the considered case probably caused a decrease in the thermal conductivity of composites, while the orientation of natural fibers significantly influences changes in the thermal conductivity of composites with matrixes of thermoplastic polymers. [43] In the case of monodirectional aligned fibers, the thermal conductivity in the fiber direction is independent of the fiber content. Randomly oriented fibers, as observed in injection-molded specimens, will rather reveal a behavior close to the case of discussed materials evaluated in a perpendicular direction to natural fiber alignment. According to results presented by Tazi et al., [44] the addition of lignocellulosic fillers leads to a decrease in the specific heat capacity of the polymer composites, that is, these fillers provide thermal insulating behavior, as this property is directly related to the possibility of changing the temperature of the heated material by the same amount of heat. Similarly, the specific heat capacity values measured by differential scanning calorimetry at 25 °C, i.e., the initial temperature of the measurement D by the Ångström method, for composite materials, are slightly lower than those of unmodified PE. However, a different tendency can be noted when analyzing the values at 100 °C,

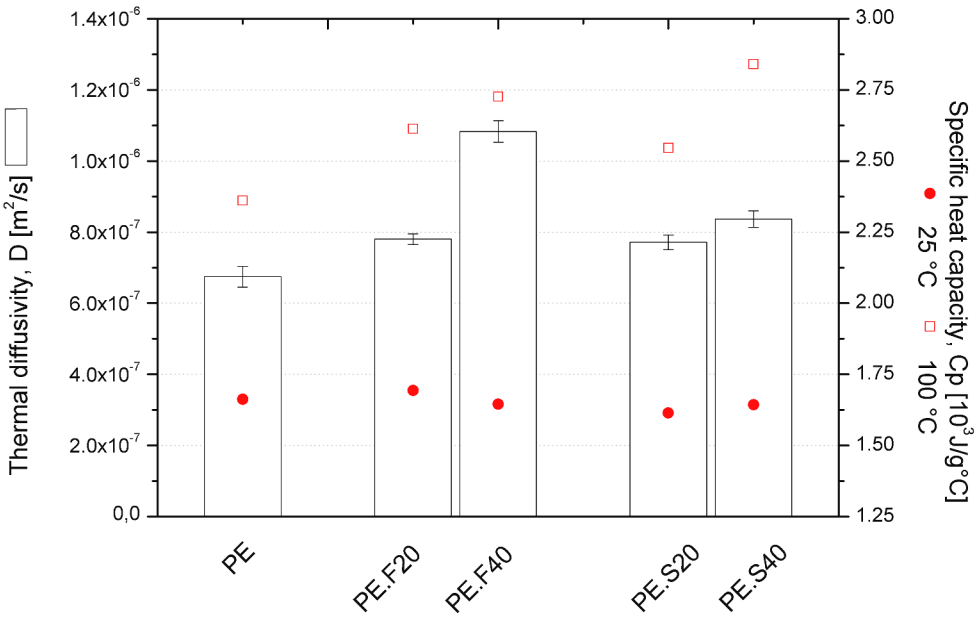


Fig. 5 Thermal diffusivity by Ångström method and specific heat capacity of PE and its composites

the maximum temperature of the range used in dilatometric measurements. ADR- and R-filled composites are characterized by an increase in Cp compared to the PE series. However, increasing the filler share resulted in an additional increase in this value.

Conclusions

This research work has shown the effect of lignocellulose fillers on the thermal properties of polyethylene-based composites, finding that the aspect ratio of the filler plays a significant role in such properties. The orientation of the fibers during the processing by injection molding allows for a reduced porosity of the final samples despite their higher aspect ratio compared to the shredded material. This phenomenon of preferential orientation of the fibers would also imply a higher surface contact between the polymer and the filler, resulting in a greater extent of modification of the thermal properties of the composite. Therefore, the dimensional stability of the composites with fiber is improved in all the studied ranges, with this improvement being even higher for the range from 25 to 100 ºC. The higher thermal diffusivity found for composites with fibers compared to shredded particles, particularly for high loading of the lignocellulose material, is also related to the orientation of the fibers during the processing.

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Author Contribution M.B, Z.O. the study conception and design; L.S. material preparation; M.B., P.M., A.K., Z.O., L.S. data collection, and analysis; M.B., Z.O. the first draft of the manuscript writing and edition; M.B, Z.O. review process. All authors read and approved the final manuscript.

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Data Availability The data that support the findings of this study are available upon reasonable request.

Declarations

Generative AI in Scientific Writing The authors declare that they did not use generative AI systems while preparing and writing the manuscript.

Competing Interests The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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