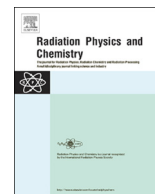Development of self-reinforced low-density polyethylene using γ-irradiation cross-linked polyethylene fibres
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Development of self-reinforced low-density polyethylene using γ-irradiation cross-linked polyethylene fibres

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ABSTRACT

In this study, we investigated low-density polyethylene (LDPE) self-reinforced composites in which the fibres were irradiated with gamma rays prior to compression moulding. The tensile tests of the single fibres revealed that the hot air-assisted melt-spun fibres had better mechanical performance than the same LDPE matrix material. The irradiation cross-linked the fibres, which made it possible to widen the temperature window of the film-stacking method we applied for composite production. The irradiated fibres did not fuse at 120 °C, which was above their crystal melting temperature (T_m = 106 °C). The effect of irradiation on the mechanical properties of fibres themselves and the composites reinforced with these fibres were investigated. Based on the mechanical and crystalline properties we obtained, the fibres irradiated with an absorbed dose of 200 kGy were chosen as reinforcement for the composites. We performed dynamic mechanical analysis and creep tests on the composites and found that irradiation had a minor effect (10% increase) on storage modulus, while creep strain decreased greatly. The composites with non-irradiated fibres had a creep strain 60% higher at a stress of 2 MPa at the end of the test. LDPE composites with irradiated fibres did not only have improved mechanical performance but they retain the main advantage of self-reinforced composites, which is simple recycling.

1. Introduction

There is a great demand for the production of environmentally friendly green composites for engineering applications. Nowadays one of the main goals is to create alternatives for glass or carbon fibre reinforced composites with a thermoset polymer matrix, which are widely used due to their high strength and modulus. Because of their relatively low density, these conventional composites are preferred in the automotive industry, for sporting goods and for numerous other applications, where light weight is essential (Ahmad Rasyid et al., 2019; Bakis et al., 2002; Buketov et al., 2019; Jalalvand et al., 2015; Juhasz et al., 2018; La Mantia and Morreale, 2011; Mochane et al., 2019).

One of the major drawbacks of the widely used cross-linked matrix composites is that their recycling is often complicated and energy-consuming. This problem can be partially solved with the use of thermoplastic matrices, which can be recycled by simple melt processes. During these processes, the fibres are fragmented and after a few re-processing steps, they act as stress concentration locations initiating

fractures, leading to a rigid and brittle material. Overall, the recycling of conventional composites leads to the deterioration of mechanical properties, and even the performance of the neat matrix is often not achieved. (La Mantia and Morreale, 2011; Pickering, 2006).

Perhaps the best solution to this problem is the use of self-reinforced polymer composites consisting of thermoplastic materials. As they can be re-processed by melting, which includes the complete melting of the fibres, fibre fragmentation is not an issue. According to one approach, self-reinforced polymer composites are materials in which both the matrix and the reinforcing material are from the same polymer (Gao et al., 2012). Another view is that self-reinforced polymer composites consist of matrix and reinforcing materials from the same polymer family (Kmetty et al., 2010). Whichever approach is taken, it is a basic requirement that during manufacturing, the matrix melts but the fibres do not. In the first approach, this is ensured by the higher melting temperature of the fibres caused by their high intrinsic order (e.g. orientation and crystalline ratio) achieved during the fibre forming process. In the second case, the slight differences in the chemical structure

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also play a role in the different behaviour, which results in a wider processing temperature window, thus ensuring more reliable production. In the first case, the material obtained after recycling (the temperature raised above the melting temperature of the fibres) has properties close to that of the original matrix material. In the latter case, the properties of the product are different from the original matrix to an extent but still suitable for the manufacture of plastic products (Gao et al., 2012; Kmetty et al., 2010; Rojanapitayakorn et al., 2005).

In composite science, irradiation treatment (like electron beam or γ-ray) is mainly utilized as an alternative to thermal curing and for surface modification to enhance the interaction between the reinforcing material and the matrix, for example by the pre-treatment of the fibres. Irradiation is also used for the sterilization of materials in the case of medical applications. The free radicals formed during irradiation can initiate both chain scission and cross-linking, which also affect the processability of the polymer. For example, cross-linking has a negative impact on melt processing and may even render the polymer unmeltable. However, this can be advantageous as it extends the processing temperature window for the production of self-reinforced composites, by rendering the fibres unmeltable (Zhang et al., 2005; Zhou et al., 2016).

There are several papers published on the development of self-reinforced polyethylene (PE) composites. In general, ultra-high-molecular-weight polyethylene (UHMWPE) is used as reinforcing material, and high-density or low-density polyethylene (HDPE; LDPE) as matrix material. Since the crystalline melting temperature of UHMWPE is significantly higher than that of the HDPE and LDPE, the processing of self-reinforced PE is relatively easy. The main problem of these composites is the poor interfacial bonding between the phases, despite the chemical similarity of the constituents. Another issue is that the presence of only a few wt% UHMWPE results in a massive increase of melt viscosity, which hampers processing and leads to moderate changes in mechanical properties. Up to now, several methods have been successfully used to overcome these problems, such as shear-induced crystallization or the application of HDPE wax (Cohen et al., 1997; Hofmann et al., 2017; Pegoretti et al., 2000; Zhuang and Yan, 2006).

There are some papers in which radiation cross-linking was applied with self-reinforced polyethylene composites, but in all cases, the main purpose was to modify some functional properties (e.g. oxidation resistance, wear properties) of the composites, other than mechanical properties. In these cases, irradiation was not a technological step in the production of the composite (Huang et al., 2016; Xu et al., 2019).

Our aim was to produce a self-reinforced polyethylene composite where both the reinforcing and the matrix phases are from the same polymer. First, we determined the optimal absorbed dose for the radiation cross-linking of the fibres we obtained. Then, we optimized the processing parameters for film-stacking to achieve the best mechanical performance.

2. Experimental

2.1. Fibre and film generation

We prepared self-reinforced composites by film-stacking method. DOW LDPE PG 7008 type low-density polyethylene (LDPE, σ_M = 8 MPa; E = 160 MPa; ε_B = 450%; T_m = 106 °C; MFI = 7.7 g/10 min (190 °C/2.16 kg)) was used for the preparation of both the reinforcing fibres and the matrix. The LDPE fibres were produced by hot air-assisted melt spinning. A dual slot melt blowing die was attached to a LE8-24C type single-screw laboratory extruder (LabTech, Thailand). The die contained 40 fibre-forming capillaries, with a diameter of 330 μm each, arranged in a single row. The single zone of the extruder and the die was set to 250 °C and a hot air stream at a pressure of 1 bar helped to avoid the sticking of fibres. The temperature of pressurized air was set to 150 °C with two inline heaters (AHP-7562, Omega, UK) positioned parallel to the melt blowing die.
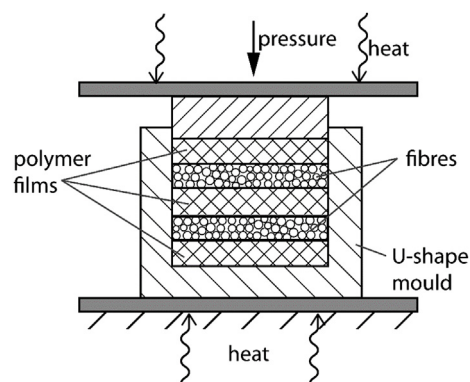

Fig. 1. Schematic drawing of the applied film-stacking method.

A drum with a diameter of 160 mm was used as the fibre collector. The rotation speed was 60 rpm and the distance between the die and the collector was 150 mm.

LDPE thin films were prepared with a LF-400 type laboratory extruder followed by a small film blowing line (LabTech, Thailand). The diameter of the blowing die was 50 mm and the balloon produced had a diameter of 200 mm. The temperature of the extruder and the die was 190 °C and the thickness of the film was 100 μm.

2.2. Irradiation and composite processing

The fibres were irradiated with an SSL-01 panoramic type ^{60}Co-γ source. The samples were irradiated with 50, 100, 150 and 200 kGy in an atmosphere of air at ambient temperature, at a dose rate of 1 kGy/h. These absorbed doses were chosen based on our previous experiments with LDPE irradiation (Mészáros et al., 2012a, 2012b).

We produced self-reinforced composites by film-stacking (Halasz et al., 2015; Lucchetta et al., 2016) with a Polystat 300S type hydraulic hot press (Maschinenfabrik Fr. Schwabenthan & Gomann, Germany). First, a 5-layer (film-fibre-film-fibre-film) structure was laid in a U-shape metal mould (Fig. 1), in which the fibres were placed uniaxially. The length of the mould was 300 mm (fibre direction), and its width was 15 mm. The fibre content of all composite samples was 25 wt%. The samples were compression moulded for 2 min with a compression pressure of 15 MPa after reaching the chosen set temperatures of 110 and 120 °C. The thickness of the samples was around 1.5 mm.

2.3. Morphology and thermal analysis

Scanning electron microscopy (SEM) was performed on the fibres with a JEOL 6380 LA type scanning electron microscope (Jeol Ltd., Japan), after they were sputtered with gold. The average fibre diameter was calculated from the measurement of 100 individual fibres. Measurements were made manually with the UTHSCSA Image Tool software.

We determined the thermal properties of the fibres by differential scanning calorimetry (DSC), with a Q2000 DSC (TA Instruments, USA) device. The tests were performed on samples with a mass of ~5 mg in an inert atmosphere (N_2; 50 ml/min flow rate) with a heating and cooling rate of 5 °C/min. The degree of crystallinity of the fibres was determined and 293.6 J/g (Lv et al., 2018) was used for the heat of fusion of the 100% crystalline LDPE.

To prove that the irradiated fibres became unmeltable, we carried out a heat-shock test with a Q500 type thermogravimetric analyser (TGA, TA Instruments, USA). The samples with a mass of ~5 mg were heated from room temperature to 120 °C with a heating rate of 50 °C/min, then kept there isothermally for 5 min and then were cooled back with a cooling rate of 50 °C/min to room temperature. These heat-treated samples were photographed with an Olympus BX51 type optical

microscope (Olympus, Germany) equipped with a digital camera.

2.4. Mechanical and thermomechanical characterization

The tensile properties of the fibres were measured with a Zwick Z005 (Zwick, Germany) type universal tensile tester. The load cell we used had a measurement range of 20 N with a 1 mN resolution. We chose the frame method described for single carbon fibres in the ISO 11566 standard for single fibre tests (Qian et al., 2010; Sui et al., 2019). The fibres were glued to paper frames with a frame window of 25 mm, corresponding to the gripping distance. The fibre together with the paper frame was gripped into the rubber-coated jaws of the fibre-gripping unit. The sides of the window frames were then cut, thus only the fibre remained amongst the grips. The tensile speed was set to 10 mm/min. The modulus of 10 individual LDPE fibres was evaluated as the slope of the tensile curves between 0.05% and 0.25% strains.

Dynamic mechanical thermal analysis (DMA) of the self-reinforced composites was carried out with a DMA Q800 type device (TA Instruments, USA) in tensile mode. The frequency, the testing temperature range and the heating rate was 1 Hz, 25–150 °C and 2 °C/min, respectively. This DMA device in tensile mode was also used for the creep tests of the composites. 30 mm × 8 mm (length × width) test specimens were used for both experiments. In the creep test, a constant 2 MPa stress was applied at 30 °C for 15 min for a single sample of each type. The gauge length was set to 10 mm for both experiments.

3. Results and discussion

3.1. Analysis of the fibres

A photograph of the fibres produced is shown in Fig. 2a. The fibres seem homogenous and no defects occurred (they neither stuck together nor broke) during melt spinning. This is also proven by the SEM images (Fig. 2b). The surface of the fibres was smooth and the diameter did not change significantly along the fibres. The average diameter was 67.4 μm with a 7.6 μm standard deviation.

3.2. The effect of irradiation on the fibres

Fig. 3 shows the second heating curves of the DSC test of the irradiated fibres. Irradiation had a minor impact on the melting of the crystalline phase. The vertical dashed line at 110 °C indicates where crystal melting is complete. It is also clear that at 120 °C, the crystallites completely melt in all the cases. We supposed that radiation-induced cross-linking makes the fibres unmeltable: only the chains of the crystalline phase unfold, but the cross-linked amorphous parts retain the fibre shape due to the limited mobility of the macromolecules. As non-irradiated polyethylene films completely melt above 110 °C, this makes it possible to produce self-reinforced composites at elevated temperatures. Based on the DSC results, we chose processing temperatures of 110 °C and 120 °C for film stacking to produce self-reinforced composites.

Table 1 shows the results of the evaluation of the DSC curves. The crystalline melting peak slightly decreases together with the degree of
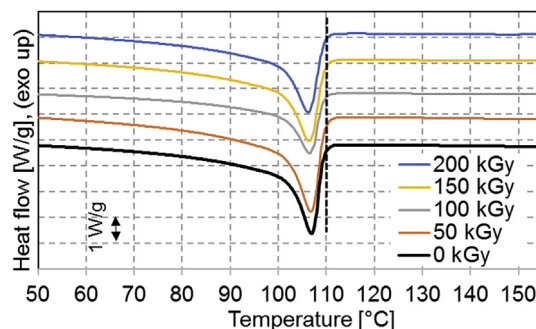
Fig. 3. DSC curves of the second heating of the irradiated fibres.

Table 1
The melting peak temperature (T_m) and the degree of crystallinity (X) of the materials tested.

Absorbed dose [kGy]	T_m [°C]	X [%]
0	106.9	40.3
50	106.9	40.0
100	106.6	37.4
150	106.4	38.1
200	106.2	38.7

crystallinity as a function of the absorbed dose. This can be attributed to the presence of so-called frozen free radicals: while radicals are also formed in the crystalline regions of the polyethylene during irradiation, their reaction is hindered by chain mobility. However, during the first heating step, these chains also become mobile due to the melting of these regions, thus they can participate in the cross-linking reactions, as well. These newly formed cross-links disrupt recrystallization during cooling, thus leading to a decrease in the crystalline fraction (Khonakdar et al., 2006).

To prove that the fibres become unmeltable above the crystalline melting temperature, we performed a heat-shock test. The irradiated sample retained its shape even if the treatment temperature (120 °C) was far above the crystalline melting temperature, while the reference sample (without irradiation) melted completely (Fig. 4).

Fig. 5 shows the tensile properties of the fibres. As a result of fibre formation, the tensile strength of the material increased 4-fold and the tensile modulus increased by 25%, while the strain at break decreased by 85% compared to those of the bulk material (σ_M = 8 MPa, E = 160 MPa and ε_B = 450%, respectively), which indicates some molecular orientation along the fibre. This also means that the fibres seemed suitable for reinforcement in a self-reinforced composite. As the absorbed dose was increased, the tensile strength gradually decreased (Fig. 5a) from 32 MPa to 22 MPa, while the tensile modulus did not change significantly (Fig. 5b). Strain at break somewhat increased at higher absorbed doses. The changes in the mechanical properties are the result of multiple phenomena: the increase of cross-linking density, the decrease of the crystalline fraction and the oxidative degradation of the polymer chains during irradiation due to the presence of air.

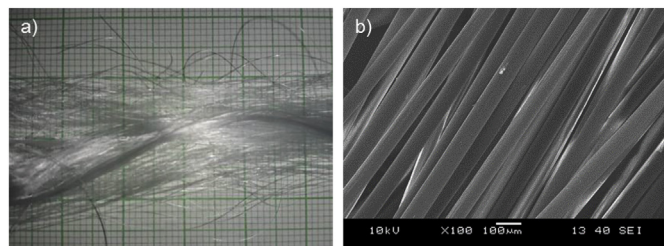The DSC results and the changes in mechanical properties are in line

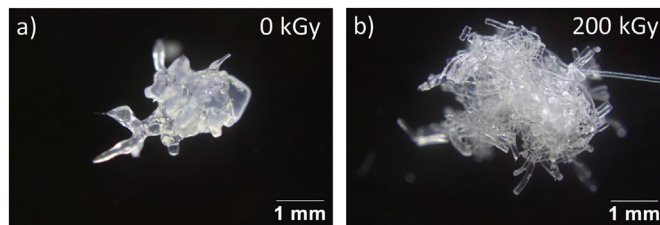**Fig. 2.** A photograph (a) and a SEM micrograph (b) of the fibres produced.

Fig. 4. Optical microscopic images of the non-irradiated (a) and the irradiated (b) fibres after heated at 120 °C for 5 min.
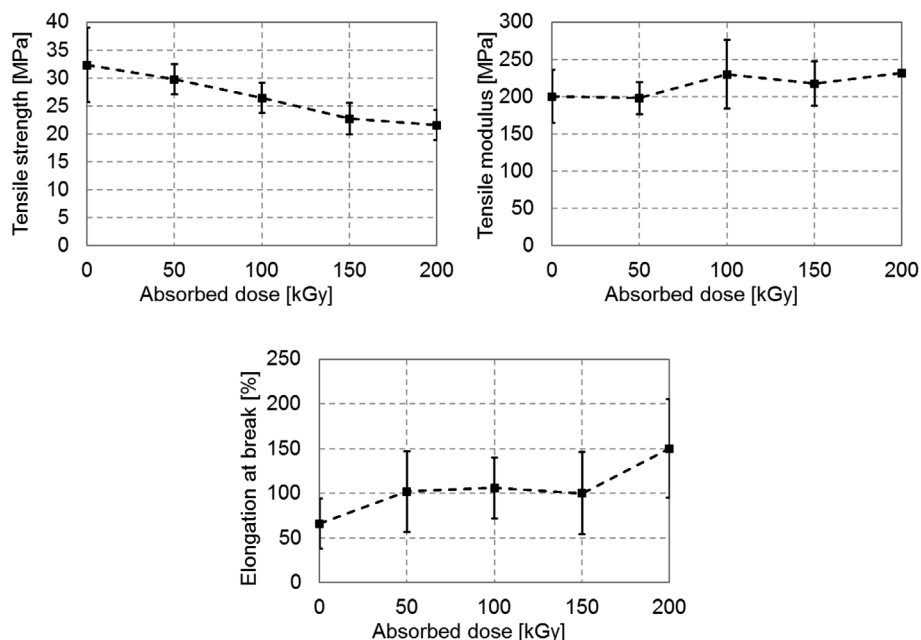
Fig. 5. Tensile properties of the irradiated fibres (a) tensile strength (b) tensile modulus (c) elongation at break.
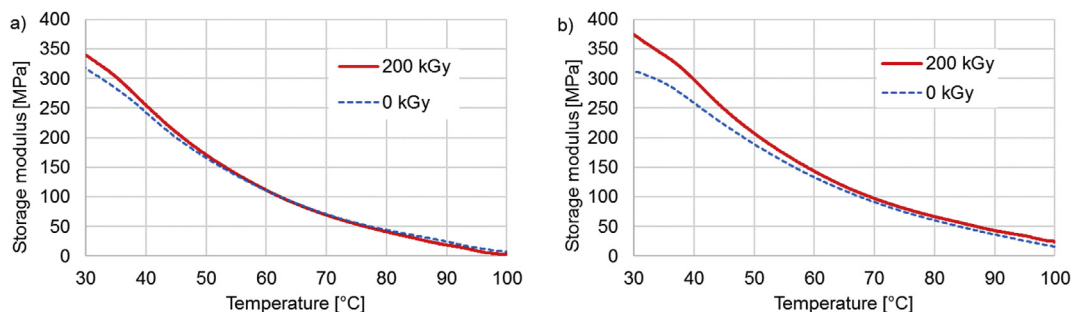

Fig. 6. Storage modulus of the self-reinforced composites produced (a) at 110 °C and (b) at 120 °C.
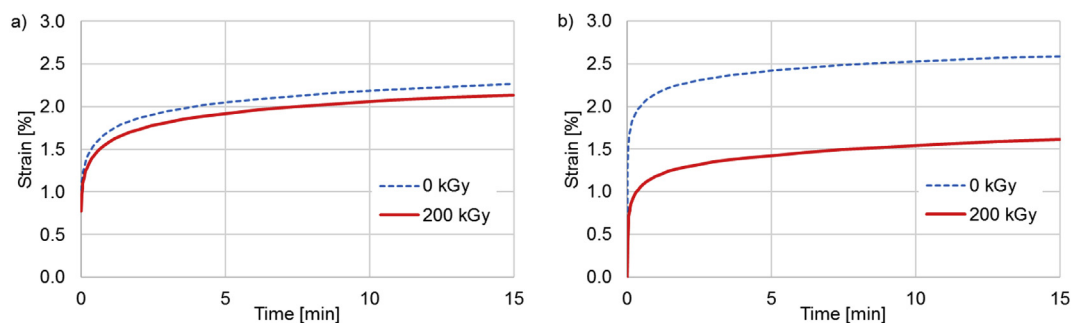

Fig. 7. Creep behaviour of the self-reinforced composites processed (a) at 110 °C and (b) at 120 °C.

with the results obtained by other authors for the irradiation of ultra-high molecular weight polyethylene (de Boer and Pennings, 1981; Lewis, 2001; Zhao et al., 2011). The slight decrement in case of the crystalline melting temperature can be explained with the defects in the crystallites formed due to the irradiation (Zhao et al., 2011). It is known that the tensile strength of the various polyethylenes decreases with increasing absorbed doses as it was experienced in our case, too. This phenomenon can be explained by chain scission occurring due to the irradiation (de Boer and Pennings, 1981). Typically, elongation at break decreases with the increasing radiation dose for polyethylene fibres, while in our case, it increased. It can be explained with the fact that in our case, the orientation of the molecules is on a lower level compared

to an industrially produced ultrahigh molecular weight polyethylene fibre, and moreover, we used LDPE, which is a highly branched polymer. This means that the molecules can easily move relative to one another compared to a highly oriented polymer with few branches. In the latter case, the presence of the crosslinks decreases the orientation of the molecules, which causes a decrement in elongation at break. In our case, crosslinking occurs mainly between the branches, therefore the result is a rubber-like structure, which results in higher elongation at break.

Based on the results, we chose fibres with a 200 kGy absorbed dose for the production of self-reinforced composites. In this case, crosslinking density is expected to be the highest, as our findings are in good

agreement with the literature (Ziaie et al., 2005). This ensures that the fibres remain in fibre form at high temperatures, while their tensile strength is still three times greater than that of the bulk material.

3.3. Investigation of the self-reinforced composites

At first, DMA tests were carried out. Fig. 6 shows the storage moduli of the composites we produced. The moduli of the irradiated fibre-re-inforced composites are higher compared to those of the non-irradiated composites, regardless of the processing temperature. Moreover, increasing the processing temperature produced better results in this case, while it did not affect the storage modulus of the reference material (*e.g.* the value of the storage modulus at 30 °C increased from 330 to 367 MPa). The explanation of the results can be that within the material containing non-irradiated fibres, the fibres completely melted during production above T_m, as expected. On the other hand, the irradiated fibres did not melt and at higher temperatures, the melted matrix material may have penetrated between the fibres and interacted with them more efficiently.

The creep properties of the materials clearly show the effectiveness of fibre irradiation and the benefit of applying higher processing temperatures (Fig. 7). Ultimately, the results are very similar to those of the storage moduli. For the composites containing non-irradiated fibres, higher strains can be observed compared to composites containing cross-linked fibres. In the case of composites containing non-irradiated fibres, the strain is increased with increasing processing temperature, due to the melting of the fibres at higher temperatures. The composite processed at 120 °C had significantly lower strain as a function of time compared to any other material. The strain of this material was only 1.62% at the end of the test, while for the rest it was above 2%. The results can be explained again with the better consolidation and the presence of the more thermally stable fibres, which again is an advantage of irradiation.

4. Conclusions

In this study, we presented a new way of processing self-reinforced composites in which the constituents were the same LDPE material. To make the composites, we made films by extrusion film blowing and generated reinforcing fibres by melt spinning. The single-fibre tensile tests revealed that the fibres had a 4-fold higher tensile strength and a 25% higher modulus than those of the bulk material. The fibres were then irradiated at different doses (50–200 kGy) and based on that, we found that irradiation significantly changed their mechanical performance and also the melting characteristics. Irradiation had no significant effect on the melting of the crystalline phase (T_m = 106 °C), but the amorphous phase did cross-link, which was proved with a heat shock test carried out in a TGA, in which the irradiated fibres did not melt at 120 °C.

The DMA and the creep tests of the self-reinforced LDPE fibre composites revealed that irradiation had a positive effect on both the storage modulus and the creep behaviour of the materials tested. The non-irradiated LDPE fibres had a reinforcing effect, but the composites with irradiated fibres performed even better. That can be attributed to the fact that at higher compression moulding temperatures, the matrix might have been able to penetrate better in between the fibres without melting those. The difference was especially visible in the creep test results, where the cross-linked fibres led to significantly lower creep deformations.

CRediT authorship contribution statement

László Mészáros: Conceptualization, Supervision, Writing - review & editing. **Yahya Kara:** Data curation, Investigation. **Tamás Fekete:** Writing - original draft. **Kolos Molnár:** Validation, Writing - original draft, Writing - review & editing.

Declaration of competing interest

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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