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Starch-grafted-polypropylene/kenaf fibres composites. Part 1: Mechanical performances and viscoelastic behaviour

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ABSTRACT

The mechanical performances and viscoelastic behaviour of starch-grafted-polypropylene/kenaf fibres composites were investigated on composites prepared by melt compounding and compression molding. In particular, the effects of various amounts (10, 20 and 30 wt.%) of kenaf fibres having two different initial aspect ratios (L/D = 30 and 160) were analyzed.

The processing-induced variation of fibre length was quantified by optical microscopy observations. Young modulus, stress at break, impact resistance and hardness values of starch-grafted-polypropylenes were remarkably improved by kenaf fibres. Moreover, creep tests have shown a noticeable enhancement of the creep stability of composites with fibres loading.

1. Introduction

In recent years, great attention was focused on the development of environmental friendly materials which combine synthetic polymers with plant based fibres [1,2]. In particular, sisal, flax, hemp and kenaf fibres have been proven to have noteworthy reinforcing effect and good potentiality for replacing glass fibres in certain applications [3–6]. Moreover, natural fibres are biodegradable, environmentally safe, coming from renewable resources and non-abrasive during processing [1,3]. Kenaf fibres have received much attention in polymeric matrix composites fabrication for their economical and ecological advantages. In fact, they are able to grow easily and quickly under very different climates [7]. Several recent reports focused on the investigation of the properties of various types of kenaf-based plastic composites [7]. In most cases fully synthetic and non-biodegradable matrices have been considered, such as polypropylene [1,3,8–13], high density polyethylene [14], polyurethane [15], unsaturated polyester resin [16,17], and epoxy resin [18]. A great attention has been also devoted to kenaf-reinforced biodegradable matrices, such as poly (lactic-acid) [19–21], thermoplastic starch copolymers [22,23], poly(butylene succinate) [24] and soy based bioplastic [2]. Unfortunately, the most widely used bioplastics, are often either too expensive or too sensitive to ageing and humidity, and are not rigid and resistant enough to be used to manufacture durable engineering parts [25]. Therefore, material producers have developed hybrid (biphasic) environmental-friendly polymers where a bio-based matrix is associated with a more conventional polyolefin (generally polyethylene or polypropylene). In particular starch-grafted-polypropylene may provide a suitable alternative solution [26].

In the present work a biphasic thermoplastic matrix such as Gaialene® (Roquette, France [26]) made of polypropylene grafted with starch at a 48/52 weight ratio has been reinforced with various amounts of kenaf fibres having two different aspect ratios. The thermo-mechanical properties of the resulting composites have been deeply investigated.

2. Materials and methods

2.1. Materials

Starch-grafted-polypropylenes G906PF and G906PJ used as matrices in this work, were kindly supplied by Roquette S.A (Lestrem, France), in the form of pellets. According to the technical data sheets, they have a density of 1.1 g/cm^3 (ISO 1183), a melt flow index MFI of 1.1 g/10 min (ISO 1133, 190 °C, 2.16 kg) for G906PF and 40 g/10 min (ISO 1133, 190 °C, 10 kg) for G906PJ, and a melting temperature of 160 °C.

Kenaf fibres were provided by Kenaf Natural Fibre Industries Sdn. Bhd. (KFI), Malaysia. In this work kenaf fibres with two different length over diameter (L/D) ratios were used: short kenaf fibres (SKF, L/D ≈ 30) and long kenaf fibres (LKF L/D ≈ 160). According to previous analyses, they are composed by 45–57 wt.% of cellulose, 21.5 wt.% of hemicelluloses, 8–13 wt.% of lignin and 3–5 wt.% of

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pectin [7] and the average density is 1.4 g/cm^3 [7,12]. The kenaf fibres used in this investigation have not been subjected to surface treatment.

2.2. Composites preparation

Prior to use, kenaf fibres were overnight dried at 80 °C under vacuum, in order to reduce the humidity content to less than 3 wt.%. Starch-grafted-polypropylenes G906PF and G906PJ were also dried in a "Bryair MORETTO" circulating air drier for 12 h at 50 °C. Various composite compositions (see Table 1) were prepared by melt blending in an internal mixer Rheomix 600 by Thermo Haake® (Karlsruhe, Germany). The following processing parameters have been used: temperature 175 °C, rotor speed 60 rpm, residence time 7 min. Finally, square sheets about 1 mm thick were obtained by compression moulding in a Carver® hydraulic laboratory press at 170 °C followed by fast cooling. ISO 527 1BA tensile specimens were punch-cut from the obtained sheets.

2.3. Electron and optical microscopy

The morphology of polymers and composites were observed by means of a Zeiss Supra 40 field emission scanning electron microscope (FESEM) at different magnifications. Samples were cryo-fractured in liquid nitrogen and metalized before observations.

The average dimensions of both SKF and LKF, were determined before and after processing. In particular, fibres length and diameter were determined through an image analysis software (Image J) on digital picture taken by an optical microscope. In order to evaluate fibres length after processing, fibres were extracted from composites by matrix dissolution in xylene at 150 °C in a Soxhlet device. Image analysis has been performed on a significative number of fibres in the range between 100 and 200 before processing and between 300 and 400 after processing for both SKF and LKF.

2.4. Mechanical testing

All tensile tests were performed on ISO 527-2 type 1BA specimens. In particular, quasi-static uniaxial tensile tests were performed by an Instron® (Norwood, MA, USA) model 4502 tensile testing machine equipped with a 1 kN load cell. Elastic modulus was evaluated deforming the specimens up to a strain of 1% at a cross-head speed of 0.25 mm/min. The strain was measured by a resistance extensometer Instron® model 2620–601 with a gage length of 12.5 mm. According to ISO 527 standard, the elastic modulus was evaluated as a secant value between deformation levels of 0.05% and 0.25%. Tensile tests up to fracture were performed at a higher cross-head speed (10 mm/min) without the extensometer. At least 10 specimens were tested for each sample.

Tensile impact tests were performed by a CEAST® (Turin, Italy) impact pendulum under tensile configuration. The hammer was released from a height selected to reach an impact velocity of 2 m/s with an impact energy of 4.38 J (hammer mass of 2.191 kg). Tests were carried out at room temperature (23 °C) and a minimum of 5 specimens were tested for each sample.

Shore D hardness of composites was measured using a commercial durometer by AtsFaar (Milano, Italy).

Creep tests under tensile configuration were performed by a TA Instruments DMA Q800 apparatus (New Castle, DE, USA) on rectangular strips with dimensions $15 \times 5 \times 1$ mm^3. Creep experiments at 30 °C were carried out under an applied stress of 1.3 MPa.

Table 1
Burger's model fitting parameters for the creep response of G906PF- and G906PJ –based composites.

Composition	E_M (GPa)	E_K (GPa)	η_M (GPa s)	η_K (GPa s)	R^2
G906PF	0.2931	0.1313	416.67	26.27	0.9782
G906PFSKF10	0.6688	0.4432	1250	94.29	0.9686
G906PFSKF20	0.8750	0.7143	2000	99.21	0.9497
G906PFSKF30	1.9051	2.0653	10000	389.67	0.9055
G906PFLKF10	0.6186	0.4068	1250	71.37	0.9652
G906PFLKF20	0.8061	0.5864	1666.67	104.72	0.9618
G906PFLKF30	1.3824	1.5263	5000	224.45	0.9089
G906PJ	0.2876	0.1530	500	27.83	0.9724
G906PJSKF10	0.7754	0.5759	1666.67	117.54	0.9615
G906PJSKF20	1.1830	0.8615	2500	191.46	0.9594
G906PJSKF30	2.0982	2.2381	10000	334.05	0.9047
G906PJLKF10	0.6439	0.4302	1428.57	82.72	0.9634
G906PJLKF20	0.9134	0.7001	2000	118.67	0.9534
G906PJLKF30	1.9650	2.0734	10000	199.37	0.9113

3. Results and discussion

3.1. Morphology

Cryo-fractured cross-sections of neat matrices G906PF and G906PJ are shown in Fig. 1a and b, respectively. In both cases, two phases can be clearly distinguished. The dispersed phase is the thermoplastic starch, with particles having diameters approximately varying from 0.5 to 3 μm and from 0.2 to 1.5 μm for G906PF and G906PJ, respectively. The continuous phase is the polypropylene.

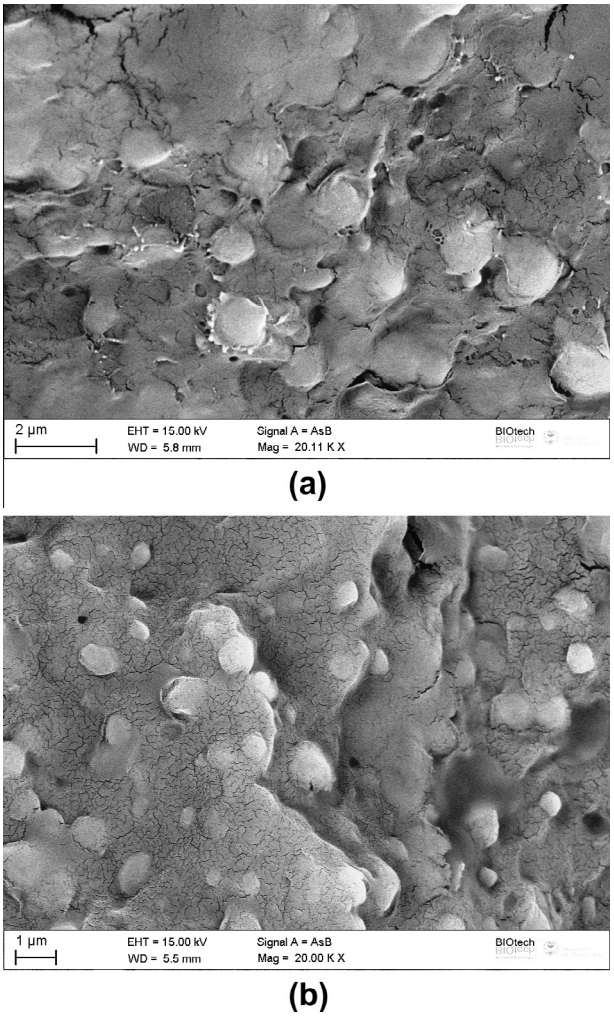

Fig. 1. FESEM micrographs of (a) G906PF and (b) G906PJ neat matrices.

It is clear that the particles of thermoplastic starch are homogenously dispersed and well bonded to the polypropylene matrix. Due to the good compatibility between the two phases, no debonding phenomena were observed. This morphological feature is also confirmed by the observation reported by Tessier et al. [25] on a similar matrix.

In Fig. 2 images of both G906PF- and G906PJ-based composites reinforced with 20 wt.% of SKF or LKF are reported. Neither interstices at the interface between the matrix and fibres, nor debonding or pull out of fibres from the matrix can be observed. All these morphological features are clear indications of good wettability. In fact, a certain chemical affinity between the matrix starch component and the kenaf fibres could be expected by considering that both constituents contain hydroxyl groups and ether links in the glucose units and polysaccharides.

The fibre length distribution of both SKF and LKF before and after processing are presented in Fig. 3 for both SKF and LKF fibres. As expected, a sharp decrease of fibres length is observed after processing. In fact, length values decrease from an initial average value of 1.5 mm for SKF and 11 mm for LKF to respectively, 0.59 mm and 0.41 mm. Concurrently, also the average aspect ratio (reported in Fig. 4) of SKF and LKF fibres decreases after processing from the initial values of 30 and 160 to 18 and 17, respectively. The reduction in length and aspect ratio is certainly due to fibres breaking during the compounding stage in the high-shear mixer. It is worthwhile to note that at the end of the processing stages the investigated composites contain fibres of similar aspect ratio, independently from the initial length of the kenaf fibres.

3.2. Tensile behaviour under quasi-static conditions

Tensile moduli of G906PF and G906PJ at various contents of both SKF and LKF fibres are compared in Fig. 5. As expected, the rigidity of composite materials increases with kenaf fibres content. Young's modulus increases by a factor of 2 with the addition of 10 wt.% of kenaf fibres. With 20 wt.% and 30 wt.% weight of kenaf fibres, moduli values increase by a factor of 3 and almost 5, respectively if compared to neat matrices. The trend is practically the same for both G906PF and G906PJ matrices. It is also quite evident that, for any given kenaf content, the initial length of the fibres does not play a role on the tensile modulus of the composites. This evidence could be explained by considering that the fibres are shortened under the selected processing conditions, especially during the melt compounding stage in the internal mixer as already evidenced in the previous paragraph. The final length of SKF and LKF is similar in the tested specimens. Moreover, Young's modulus values obtained in the present study are much higher than those reported by Law and Ishak [9] for a PP/MAPP/short kenaf fibres system, while comparable values were obtained by Liu et al. [2] on kenaf fibre reinforced soy based biocomposites/kenaf. According to Ochi et al. [19], the section of kenaf plant from where fibres are taken from also plays a major role in determining the mechanical properties of composites. He suggests that fibres should be taken from the section closest to the ground to optimise mechanical properties of single kenaf fibres. Moreover, John et al. [1] showed that the highest properties can be reached when woody fibres are separated from the bast core of kenaf fibres.

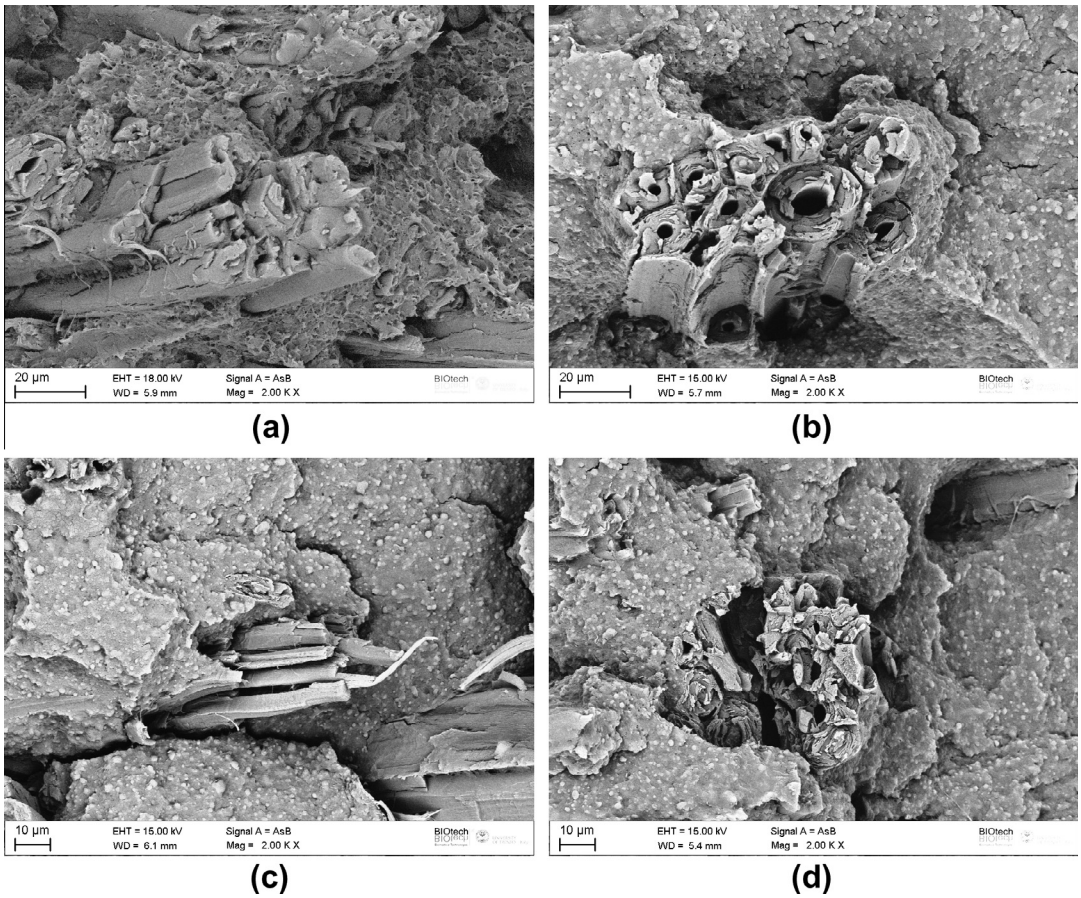

Fig. 2. FESEM micrographs of (a) G906PF/SKF, (b) G906PF/LKF, (c) G906PJ/SKF, and (d) G906PJ/LKF based composites at a fibre content of 20 wt.%.

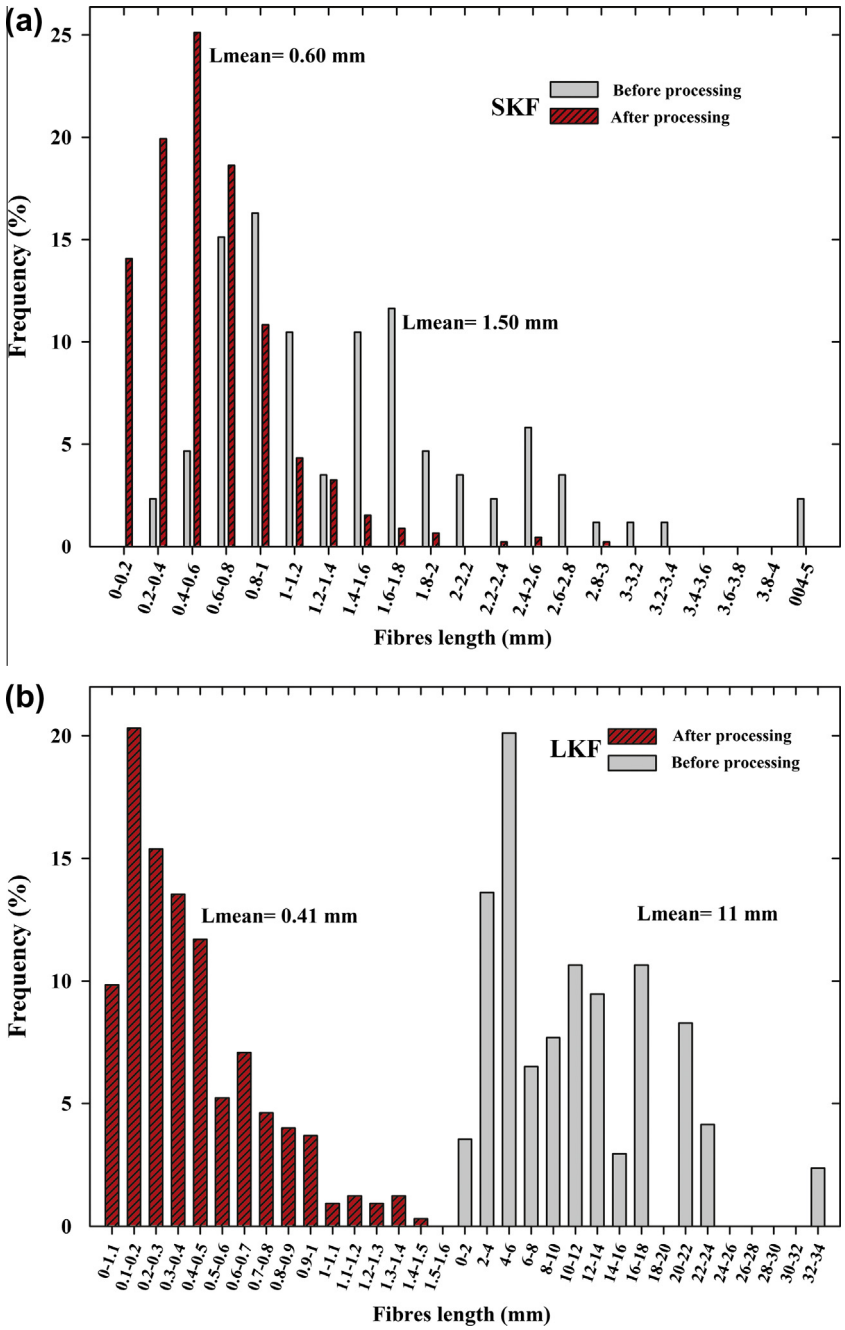

Fig. 3. Histograms of length distribution for (a) SKF and (b) LKF, before and after processing. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The following empirical equation is often used to predict the elastic modulus (E_R) of composites containing fibres that are randomly oriented in a plane:

$$E_R = \frac{3}{8}E_L + \frac{5}{8}E_T \tag{1}$$

where E_L and E_T are respectively the longitudinal and transverse moduli for a unidirectionally aligned short fibre composite having the same aspect ratio and fibres volume fraction. Moduli E_L and E_T can be calculated on the basis of the Halpin–Tsai semi-empirical model [27], and specifically through the following equations:

$$E_L = E_m \frac{1 + \xi_L \eta_L V_f}{1 - \eta_L V_f} \tag{2}$$

$$E_T = E_m \frac{1 + \xi_T \eta_T V_f}{1 - \eta_T V_f} \tag{3}$$

where E_m is the matrix modulus, V_f the volume fraction, while ξ and η are parameters defined as:

$$\xi_L = 2\frac{L}{d}; \quad \xi_T = 2; \quad \eta_L = \frac{\frac{E_f}{E_m} - 1}{\frac{E_f}{E_m} + \xi_L}; \qquad \eta_T = \frac{\frac{E_f}{E_m} - 1}{\frac{E_f}{E_m} + \xi_L}$$

where L/d is the fibre aspect ratio and E_f is the fibre modulus. Subscripts L and T refer to the longitudinal and transverse directions, respectively.

Fig. 6 illustrates the evolution of elastic moduli values predicted by the Halpin–Tsai equations for G906PJ composites reinforced with both SKF (Fig. 6a) and LKF (Fig. 6b) fibres. The same plots also

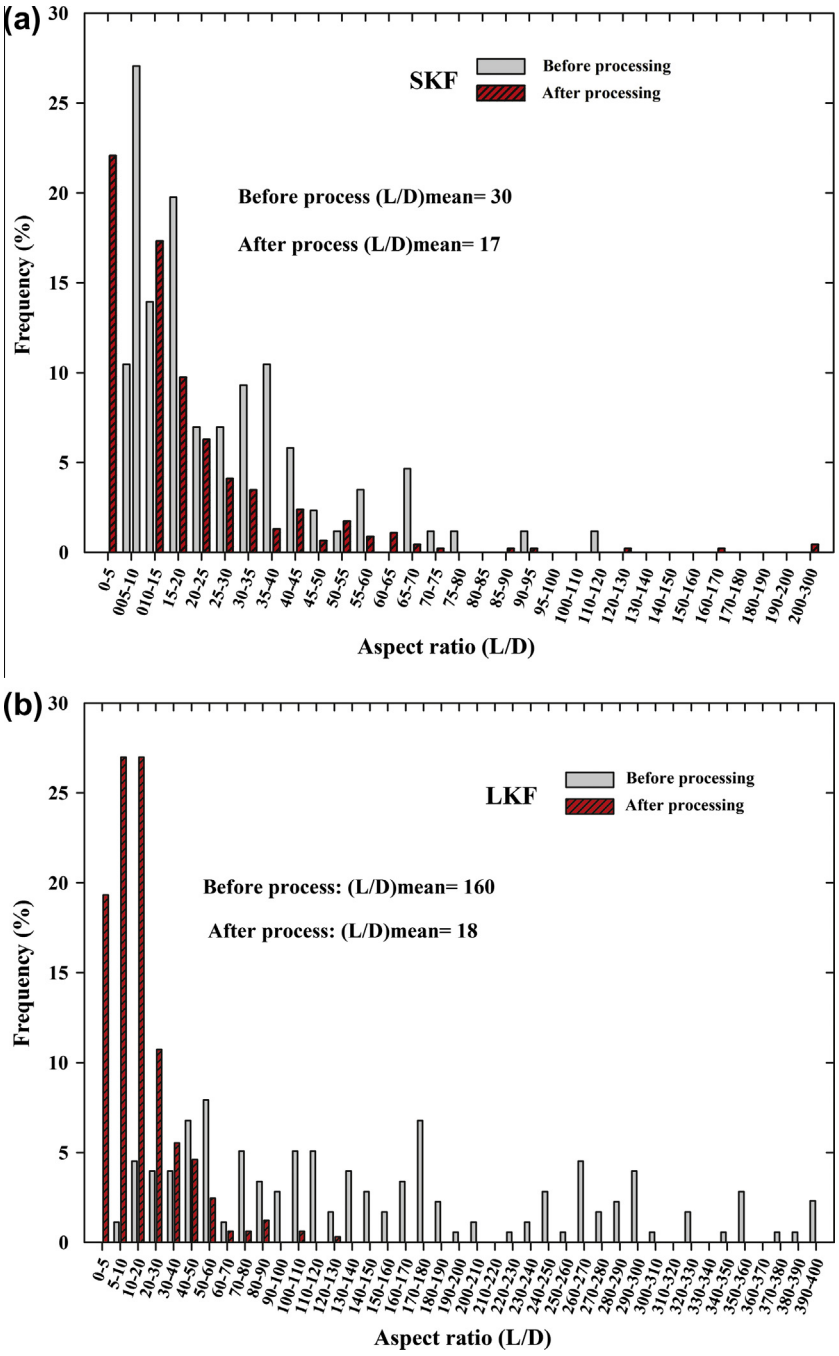

Fig. 4. Histograms of aspect ratio distribution for (a) SKF and (b) LKF, before and after processing. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

report the experimental data for a comparison. From the comparison between moduli predicted at various aspect ratio with the experimental data, it is possible to estimate the theoretical aspect ratio after processing. It is found to be approximately situated between (L/D) = 15–20 for both SKF and LKF kenaf fibres. The experimental determination of fibre aspect ratio indicated values of about 18 for LKF and 17 for SKF, in good agreement with the model's indication.

The effect of fibre weight fraction on the tensile stress at break of the composites is reported in Fig. 7. Both matrices are positively affected by the addition of kenaf fibres. The remarkable increase of tensile strength is a clear indication of a good fibre/matrix stress transfer. Thermoplastic starch is a complex homopolymer of α-D-glucose units containing hydrophilic groups [28]. The presence of these hydrophilic groups allows Gaialene matrices to have good adhesion and wettability with kenaf fibres. When reinforced with 30 wt.% of kenaf fibres, the tensile strength of both matrices is improved by a factor of about 2.5. These results agree with those reported by other authors [6,7,19] using various matrices reinforced with kenaf fibres. For example, Ochi [19] reported that the tensile and flexural strength of kenaf reinforced PLA composites, increase linearly with fibre contents up to 50 wt.%. According to results obtained by this researcher, kenaf fibres exhibits higher strength values in terms of tensile and flexural properties, as compared to other natural fibres when reinforcing PLA. However, Zampaloni et al. [12] noticed that the addition of 30 and 40 wt.% of kenaf fibres in a PP matrix provides tensile and flexural strength comparable to 40 wt.% of flax and hemp fibres. In contrast, the tensile

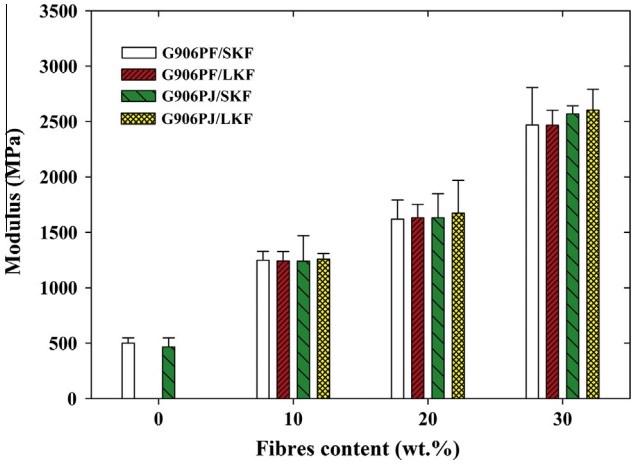

Fig. 5. Elastic modulus of G906PF- and G906PJ-based composites with various amounts of SKF and LKF. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

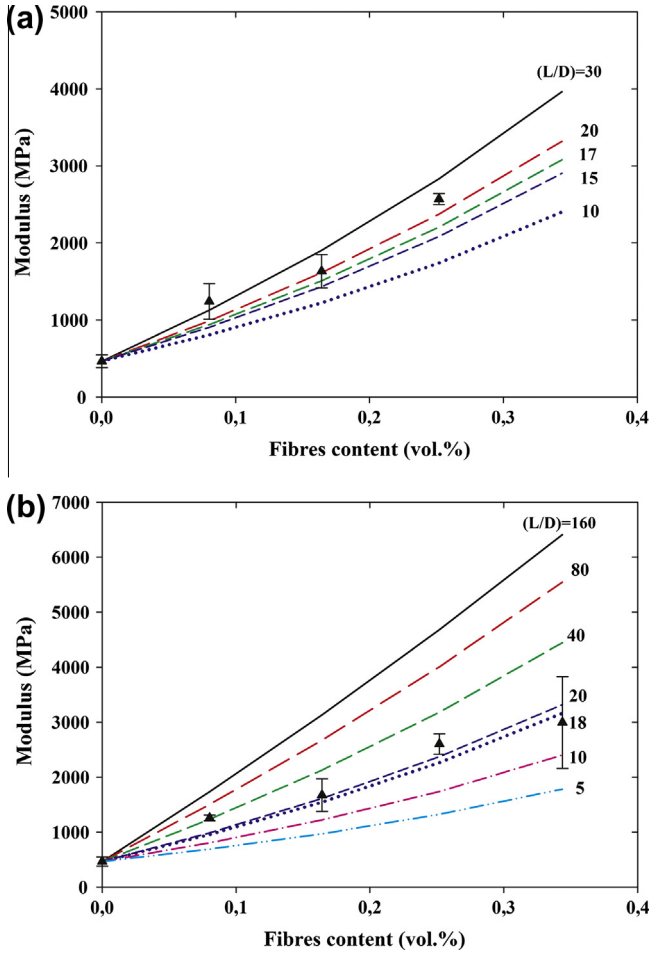

Fig. 6. Experimental (▲) and theoretical (lines) moduli values of G906PJ based composites reinforced by (a) SKF and (b) LKF. Predictions are based on the Halpin–Tsai model. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

strength is higher and almost doubled when compared against coir and sisal systems. From the results of Fig. 7 it also emerges that the initial aspect ratio of the fibres only slightly affects the tensile strength of the composites at the lowest fibre content.

The values of tensile deformation at break are reported in Fig. 8. It is worthwhile to note that the addition of only 10 wt.% of kenaf

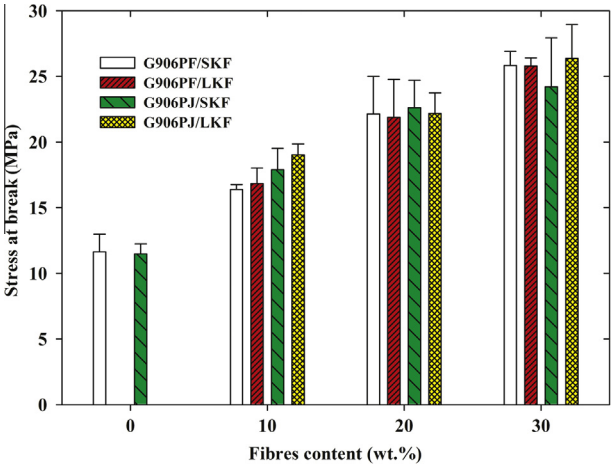

Fig. 7. Stress at break values of G906PF- and G906PJ-based composites with various amounts of SKF and LKF. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

fibres dramatically decreases the ultimate tensile elongation of both G906PF and G906PJ matrices. For further addition of kenaf fibres, the deformation at break decreases, following an exponential decay.

3.3. Impact resistance

The impact energy at break, i.e. the tensile fracture energy normalized over the cross-sectional area of the specimen, of the investigated materials is reported in Fig. 9a. It can be observed that the presence of kenaf fibres leads to a remarkable decrease of the impact energy at break of the composites. In fact, neat Gaialene matrices do not break under the selected impact conditions, while composites break with impact energy values in the range from 50 up to 100 kJ/m^2. This result is surely due to the strong reduction of the molecular mobility provoked by the addition of kenaf fibres with a consequent change of the fracture behaviour of from ductile to brittle. Similar results have been reported by Law and Ishak [9] for PP/kenaf composites with or without maleic anhydride grafted polypropylene (PPgMA) as a coupling agent.

The maximum impact stress experienced during the tensile impact tests is reported in Fig. 9b as a function of the fibre weight

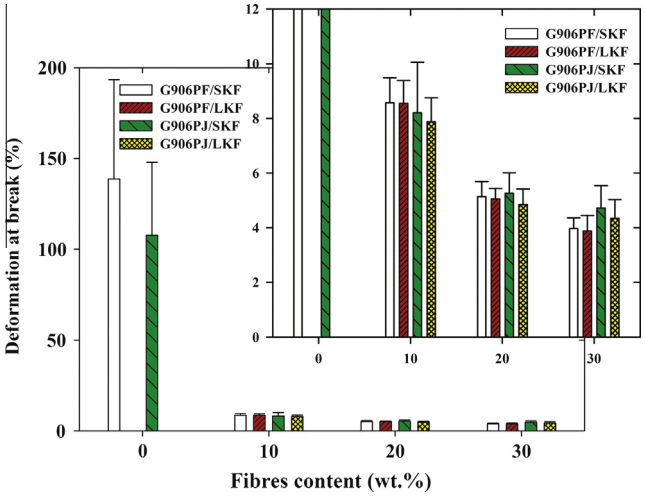

Fig. 8. Deformation at break values of G906PF- and G906PJ-based composites with various amounts of SKF and LKF. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

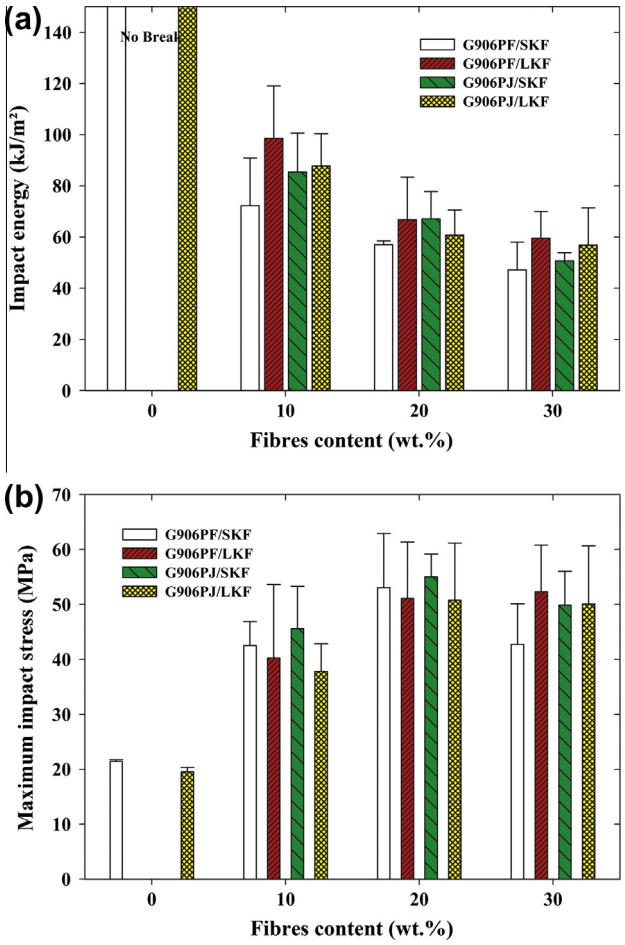

Fig. 9. (a) Impact energy and (b) maximum impact stress of G906PF- and G906PJ-based composites with various amounts of SKF and LKF. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

content. Due to the transition in the failure mode, the maximum impact stress represents the yield point for the neat matrices and the stress at break for the composites. A remarkable increase in the maximum impact stress with the incorporation of kenaf fibres can be observed. For both matrices, impact stress values double after addition of 10 wt.% of kenaf fibres. With the increase in kenaf weight fraction to 20 wt.% and 30 wt.%, the values increase moderately. The observed trend in the maximum stress under impact conditions seems to be related to the increase of quasi-static tensile strength previously described.

3.4. Shore D hardness

Hardness values of G906PF and G906PJ matrices reinforced with various amounts of kenaf fibres are summarized in Fig. 10. Both matrices display a similar initial hardness of about 45 Shore D which markedly increases with a linear trend with fibres loading. This behaviour is related to the decrease of flexibility and increase of stiffness observed on the composites [29,30]. Mishra et al. [31] has shown also an increase in hardness for unmodified unsaturated polyester composites reinforced with up to 45 wt.% of sisal, hemps and banana fibres. After this value of fibre content a decrease of hardness was observed. A similar trend was also reported when fibres were treated by a compatibilizing agent (maleic-anhydride) but a higher critical value of fibre content (50 wt.%) and higher hardness values were reported. Authors related this increase in

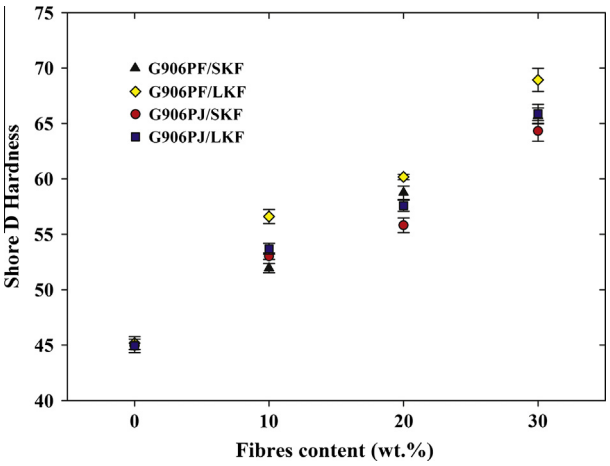

Fig. 10. Shore D hardness of G906PF- and G906PJ-based composites with various amounts of SKF and LKF. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

hardness to good fibres dispersion into the matrix and also to a strong interfacial bonding between fibres and matrix.

3.5. Creep stability

Whenever products made of thermoplastics are exposed to a long-lasting dead load, their propensity to creep, which affects their dimensional stability, becomes a critical characteristic [32].

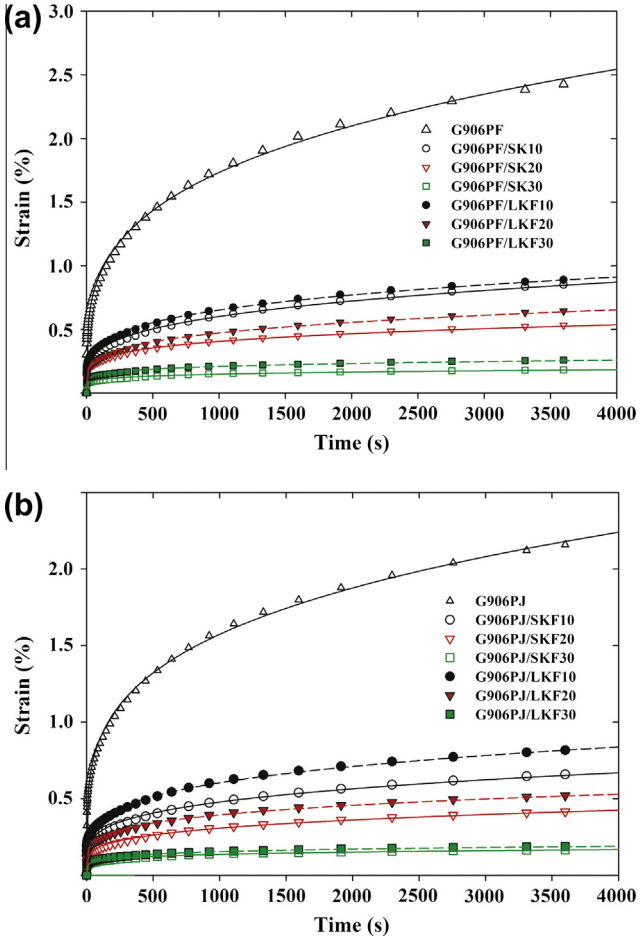

Fig. 11. Creep strain of (a) G906PF-based and (b) G906PJ-based composites with various amounts of SKF and LKF. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The effect of the addition of kenaf fibres on the creep strain of neat Gaialene matrices and Gaialene/kenaf composites is reported in Fig. 11. As the kenaf fibres content increases, the creep strain is strongly reduced. This behaviour represents a noticeably improvement of the creep stability of Gaialene matrix induced by the presence of rigid fibres. Even if the stiffening effect was practically independent from the initial aspect ratio of the kenaf fibres, the creep under constant load seems to be slightly effected by the differences in fibre length.

In order to analyse the creep behaviour of the investigated materials, a four elements Burgers physical model was used. This model divides the creep strain into three components [33], represented by an instantaneous deformation resulting from the Maxwell spring, a viscoelastic deformation resulting from Kelvin units, and a viscous deformation resulting from Maxwell dashpot:

$$\varepsilon(t) = \frac{\sigma}{E_M} + \frac{\sigma}{E_K}\left[1 - \exp\left(\frac{-E_K}{\eta_K}t\right)\right] + \frac{\sigma}{\eta_M}t \quad (4)$$

where $\varepsilon(t)$ is the creep strain, σ is the stress applied, t is the time, E_M and E_K are the elastic constant of the elastic elements (springs) in the Maxwell and Kelvin–Voigt units, respectively, while η_M and η_K are the constants of the viscous elements (dashpots) in the same units. Fitting of experimental data allowed us to determine the parameters of the Burgers model, which are reported in Table 1. It can be clearly noticed that for all composites all four parameters increase as fibres content increased. The increase in E_M and E_K parameters reflects the reduction of the instantaneous and the viscoelastic deformation promoted by kenaf fibres. On the other hand, the increase in viscosity η_M indicates a reduced plastic (irreversible) flow.

4. Conclusions

This present study describes the effect of kenaf fibres with different loading and fibre aspect ratio on the mechanical properties of starch-grafted polypropylene. A good fibre/matrix interaction has been evidenced by SEM observations. Consequently, tensile modulus and ultimate properties, hardness and tensile impact stress, were found to increase considerably with fibre loading. Due to the observed reduction in tensile strain, also the impact energy and elongation at break of the composites were lower than those of neat matrices. Short-term tensile creep tests have proven that kenaf fibres remarkably improve the creep stability. A four parameters Burgers model was successfully used to model the creep behaviour of the investigated materials.

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