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Research Article

Tensile Properties of Fibres spun from Compounded Nanocomposite of Aluminium Silicate Hydroxide/Polyolefin

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Abstract

Nanocomposites fibers of different polypropylene (PP), a polyolefin type - Aluminum Silicate Hydroxide (Kaolinite) were prepared by melt compounding using a two-step process: melt-spinning and hot drawing at various draw ratios. Draw ratio up to 23 with elastic modulus of 19 GPa was observed for K20-HP and draw ratio of 9.5 with 7.6 GPa for K10-ATO were observed. TEM and FESEM revealed good dispersion of the kaolinite nanoparticles in polypropylene matrix, although at higher concentrations and lower draw ratios the nanoparticles showed increasing tendency to form small agglomerates. Thus drawing of K30-HP-30B and K20-ATO nanocomposites is difficult due to large agglomerates formations. Uniform distribution of kaolinite improved mechanical properties of the composite fibers.

Keywords: Polypropylene; Nanocomposites; Kaolinte; Melt spinning; Drawing; Tensile Properties.

Introduction

Significant growth of polypropylene (PP) usage can be attributed to a combination of many factors, e.g. a good balance of physical and chemical properties. Moreover, low density, excellent thermal stability, chemical inertness along with wide design flexibility and simplicity of recycling makes PP an attractive construction material. Many studies have been performed to improve mechanical properties of PP fibers and films since 1964. PP can easily be drawn and crystallized so that high degrees of orientation and crystallinity can be achieved. The maximum values of Young's modulus and tensile strength of PP fibers reported in the literature are 36–40 and 1.5 GPa, respectively [1]. Fibers of PP are employed in many end-use products thanks to their properties such as low density, resistance to moisture and chemicals, sufficient strength and easy processing [2].

PP fibers properties can be enhanced by melt mixing with nanosized particles like carbon nanotubes [3,4] and montmorillonite [5,6]. Nowadays, many reports have been focused on the addition of silica and/or fumed nanosilica (FS) to enhance mechanical properties of polyolefins [7,8] and PP fibers [9,10]. Rottstegge and coworkers [9] tested PP (MFI = 1.5 g/min) fibers reinforced by fumed silica. The obtained materials were of good quality, however, modulus, strain and stress at break were only slightly higher than those of neat PP. Srisawat and coworkers [10,11] studied the influence of FS addition on the properties of PP fibers using PP with high melt flow rate (MFI = 2.5 g/min). They observed not only a higher thermal stability and nucleation effect of the nanofiller, but also a remarkable increase in the tensile strength, as consequence of the incorporation of elongated and flattened silica particles during the spinning process. Dabrowska et al. [7] prepared nanocomposites fibers of isotactic polypropylene – fumed silica by melt compounding using a two-step process: melt-spinning and hot drawing at various draw ratios. At low concentrations the uniform distribution of fumed silica improved mechanical properties of the composite fibers, especially at higher draw ratios. Crystallinity and melting temperature of fibers were found to

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significantly increase after drawing. Higher tensile strength and creep resistance were achieved, while strain at break was rather insensitive to the filler fraction.

Recent literature evidences a lot of progress in the nanofilled bulk materials; on the contrary, there are relatively a few publications on the PP-kaolinite fibers nanocomposites. Latest study found on PP-kaolinite fibers is by Dabrowska et al. [12] in which Polypropylene fibers at various kaolinites content in the range of 1 to 30%(w/w) were produced after direct mixing polypropylene and master batch, and compounding of selected formulation. The dispersion of nanoparticles enhanced the elastic modulus of PP, positively affected the stress at break, and decreased the strain at break for compositions at high nanofiller content.

In order to improve mechanical properties, it is necessary to apply very high stretching ratios. The stretching ratio, often referred as DR (draw ratio) is given by the ratio of the initial section of the fibers and the final section of the stretched fibers. If mass flow of material during the stretching process is constant, the stretching ratio is also given by the ratio between two speeds; the released and collecting drums speed.

Assuming a constant volume, draw ratio can be calculated according to Eq. 1.

$$DR = \frac{V_2}{V_1} = \frac{A_i}{A_f} = \frac{D_i^2}{D_f^2} \dots\dots\dots\dots\dots (1)$$

where, the values indicated by "i" represent the initial values of area and diameter respectively, while the values indicated by "f" are the final area and diameter values of fiber respectively.

Materials and methods

Fiber spinning

Monofilament fibers with the kaolinite fraction between 0 and 30%(w/w) were produced by Thermo Haake (Karlsruhe, Germany) PTW16 intermeshing co-rotating twin screw extruder (screw diameter 16 mm, *L*/*D* ratio 25, rod die diameter 1.50 mm). The temperature profile from the hopper to rod die was gradually increased (T_1=150°C, T_2=170°C, T_3=180°C, T_4=190°C, T_5=200°C).

The extrusion process was performed by using different die size for extruding fibers and 3D print filaments. Accordingly the size of die used and extruded sample was given in Table 1. Types of formulations for extrusion of fibers and filaments were as given in compounding sections, (Part 1 of our paper in this same journal).

Table 1. Die size used for extrusion of fibers and filaments for 3D printing

Sample type	Die diameter (mm)	Extrudate diameter (mm)
Fibers	1.65	0.5 ± 0.1
Filament for 3D	3	1.7

The collection of the fibers and filaments was carried out in a series of steps: the extruded sample leaving the extruder is cooled in a bath, successively passes through a pair of cylindrical shapes and guides that directs the movement of material and this material is collected on a bobbin fixed to an electric motor which has an inverter speed variation.

The extruder screws rotation was optimized in the range of 15 – 20 rpm and residence time of about 15 min depending on the material composition in order to produce fibers having a diameter of about 500 ±100 micron and a filament of about 1.7 mm. The spun fibers or filaments were fast cooled in water at room temperature in order to eliminate orientation and drawing of the fibers immediately after the extrusion. Extruded sample of fibers are wrapped around a bobbin of diameter 40 or 50 mm rotating at about 12 m/min, but the collection of filaments was done manually, because it is a difficult to wrap around a bobbin of about 50 mm diameter (fray-out) due to its thickness. Spinning ratio (SR) can be given by the ratio between die diameter and extrudate diameter (eq. 2).

$$SR = \frac{\emptyset_D^2}{\emptyset_E^2} \quad \dots\dots.(2)$$

where, $\emptyset_D$ is die diameter and $\emptyset_E$ is extrudate diameter.

Collection distance from extruder head is approximately one and half meter. Varying the collection speed induces the variation of stretch ratio on the filaments produced and this has the consequence to vary the diameter of the filaments. From here it can be deduced that by increasing or decreasing the speed of collection, which is playing with the electric motor, it is

possible to obtain different diameters. The diameters of filaments obtained will decrease if the collection speed increases and vice versa.

Fiber drawing

The fibers from extrusion process were drawn in a hot-plate drawing apparatus 1.4 m length (SSM-Giudicisrl, Galbiate, LC, Italy) at 145°C, in order to obtain highly extended fibers. As-spun fibers were unreeled and wound by using acrylic bobbins of 40 or 50 mm diameter rotating from 10 up to 150 rpm, corresponding to a constant feeding rate of 1.26 m/min and maximum collecting rate of about 18.84 m/min (nominal draw ratio of 15). The fiber draw ratio (*DR*) is commonly defined as the ratio between the collection (R_c) and the feeding (R_f) rate, and assuming a constant volume, it can be calculated according to eq. 1.

The diameter was measured by using an optical microscope connected to image processing software (ImageJ®) and also micrometer was used. Fibers with various draw ratios from *DR*=3 up to *DR*=19 were produced. Nominal draw ratio of 1 is referred to as-spun fibers, whereas *DR* greater than 1corresponds to the nominal draw ratio of drawn fiber with the diameter reported.

Fibers production

Compositions for fibers production were consider compounded formulations of $F_{1\text{-}13}$ and $F_{16\text{-}19}$ of three different types of PP matrices (HP500, Atofina and HP500-Borealis blend) with various content of kaolinite [13]. These formulations are used for extrusion of fibers after grinding in to small sizes of about 2 mm sizes. Extruded fibers of about 500 micron are drawn at 145°C in order to increase filler orientation in matrix and improve mechanical properties. As-spun fibers are characterized by TGA to analyse the effect of kaolinite on the thermal stability of PP at different content. As-spun and drawn fibers mechanical properties are analysed by Quasi-static tensile tests and DMA, while microstructural analysis was performed by TEM and FESEM, and XRD and DSC were used to monitor kaolinite crystal structure in the PP matrix as well as changes in the crystallinity of PP in the composites. Depending on their spinnability and drawability selected samples from extruded samples was given priority and their results were discussed in detail.

Results and discussion

Spinning and drawing of fibers

Production of fibers starts from spinning of compounded samples of different neat polypropylene and PP-kaolinite filled at different content. Accordingly three different PP are used for spinning of neat and kaolinite filled fibers: HP500 (neat HP, K10-HP and K20-HP), Atofina (neat ATO, K10-ATO and K20-ATO) and blend of HP with Borealis (K20-HP-20B and K30-HP-30B). The processing temperature in all types of composition gradually increased from the hopper to rod die in twin-screw extruders as follows: 150-170-180-190-200°C. Die diameters used in all case was 1.65 mm and output from the extruder die is assumed to be 1.65 mm. By changing the rotation per minute of twin-screw or collection rate different diameters fibers are collected. The variation in those speeds is also depending on the types of PP and its composite flowability.

The extruder screws rotation was optimized in the range of 15-20 rpm and residence time of about 15 min depending on the material composition. Thus varying collection speed induces the variation of stretch ratio on the fibers produced and this has the consequence to vary the diameter of the filaments. Minimum permitted speed by the electric motor of collecting bobbin of 50 mm was 75 rpm and fibers with a diameter from 0.55 to 0.65 mm were obtained. Figure 1 shows motor rpm versus fiber diameter by keeping screw rotation constant.

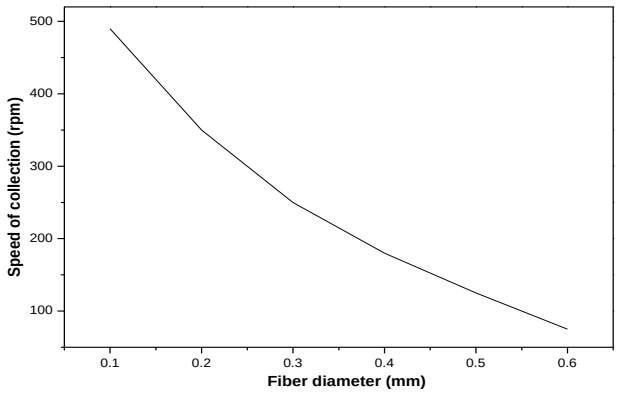

Figure 1. Speed of collection versus fibers diameter variation during spinning process

As-spun fibers were fast cooled in water at room temperature in order to eliminate orientation and drawing of the fibers immediately after the extrusion. The diameters of filaments

obtained will decrease if the collection speed increases and vice versa. Extruded fibers of about 500±100 micron on acrylic bobbin of 50 mm diameters are hot drawn at 145°C in order to increase filler orientation in matrix and improve mechanical properties.

Drawing process is the ratio between the collection (R_c) and the feeding (R_f) rate and fiber draw ratio (DR) can be calculated according to eq. 1 given above by assuming a constant volume. Table 2 describes the initial diameter, maximum attainable draw ratio (DR), speed of feeding and collection using bobbin of 50 mm diameter.

According to the results of drawing in table 2, it was found that a draw ratio up to 19 for neat HP and a draw ratio up to 16 for K10-HP, K20-HP-20Band K30-HP-30B while draw ratio up to 23 was obtained for K20-HP. Maximum draw ratio of 9.5 were found for ATO-kaolinite nanocomposites at 10% of kaolinite content. Drawing of extruded filament of neat Atofina was not performed and the one with 20% of kaolinite is difficult due to formation of larger agglomerates as it were seen under morphological study.

Table 2. Comparison of drawability of neat PP and kaolinite filled nanocomposites at different compositions.

Sample type	Kaolinite content (wt%)	Initial dia. (mm)	Speed (m/min)		Maximum Draw Ratio
			Feed	Collect	
HP	0	0.42	0.78	15.72	19.0
	10	0.48	0.78	13.66	16.0
	20	0.50	0.94	22.30	23.0
	20-20B	0.40	0.94	15.83	16.0
	30-30B	0.56	1.26	20.52	16.0
ATO	0	0.54	-	-	-
	10	0.42	1.57	15.81	9.5
	20	0.50	-	-	-

Quasi-Static tensile tests

Mechanical properties of as-spun and drawn fibers using different neat polypropylene as a matrix (HP type, ATO type and HP-B mixture) with kaolinite from 10 to 30% of kaolinite are analysed. Effect of kaolinite content and drawing ratio on elastic modulus, strength at yield, strength at break, strain at yield and strain at break will be discussed in this section.

A first series of fibers was produced with Atofina PP MFI of 1.2 g/min (ATO). Representative stress – strain curves of the quasi-static tensile tests for as-spun (DR = 1) fibers of neat ATO and ATO with 10% of nanofiller content are reported in figure 2. As observed from stress versus strain curves addition of 10% kaolinite enhances the mechanical properties other than strain at break compared to neat Atofina PP. But at a higher composition of 20% of kaolinite, it gives much less mechanical properties other than elastic modulus compared to neat ATO and ATO-K10 nanocomposites due to formation of larger agglomerates as it was seen in microstructural study.

Another series of fibers was produced with HP500 MFI 0.18 g/min (HP) and mixing it with Borealis MFI of 2.6 g/min (B) at higher filler content. Representative tensile stress-strain curves of as-spun sample of neat HP and HP-kaolinite nanocomposites at 10% and 20% kaolinite content was shown in Figure 3.

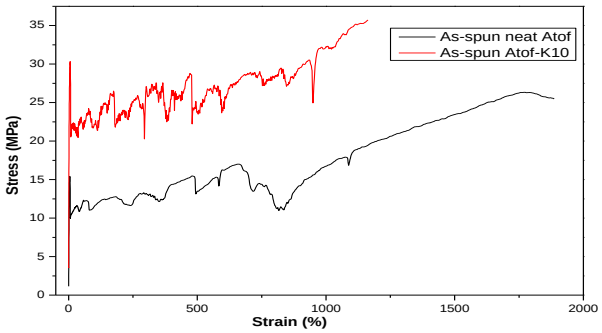

Figure 2. Representative tensile stress-strain curves of as-spun fibers of neat Atofina and Atofina nanocomposites filled at 10% of kaolinite

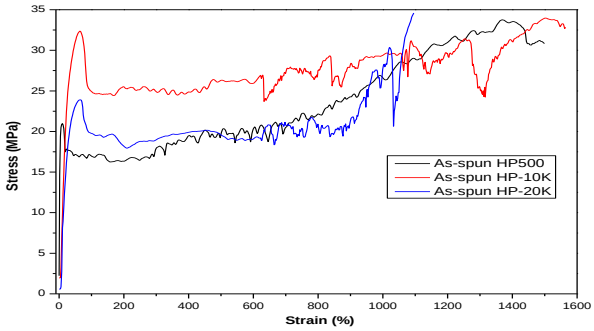

Figure 3. Representative tensile stress-strain curves of as-spun fibers of neat HP and HP nanocomposites filled at 10% and 20% kaolinite

The most relevant mechanical properties of as-spun fibers of neat and kaolinite filled HP, Atofina and HP-Borealis blend polypropylene are summarized in Table 3. From table 3, it was found that the modulus of as-spun fibers (DR = 1) shows the tendency of increasing with the kaolinite content, which is 0.6 GPa for neat ATO to about 1.25 GPa for ATO-kaolinite filled nanocomposites. Considering properties at yield, strength highly increased in Atofina with 10% of kaolinite content and decreased with 20% of kaolinite content while strain at yield highly decreased with the increase of kaolinite content compared to neat Atofina. From mechanical properties at break it is possible to observe strength at break increased for K10-ATO and decreased for K20-ATO while the strain at break highly decreased with kaolinite content relative to neat Atofina polypropylene.

Similarly for HP500 PP matrix it is important to understand that for all the compositions with high nanofiller content of as-spun fibers shows significantly higher elastic modulus as observed. In particular improvement from 0.75 GPa for neat HP500 up to 1.48 GPa for K20-HP-20B. Tensile stress at yield (σ_y) slightly increases for the compositions with nanofillers content of K10-HPand K20-HP-20B while for highest nanofiller content i.e. K30-HP-30B slightly lower values were observed. Improvement in yield strength can be a signal of good distribution of filler in a matrix.

Table 3. Quasi-static tensile properties of as-spun HP, HP-B and ATO polypropylene and its nanocomposites fibers at different kaolinite content

PP type	Kaolinite content (wt%)	Elastic modulus [GPa]	Stress at yield, $\boldsymbol{\sigma}_y$ [MPa]	Strain at yield, $\boldsymbol{\varepsilon}_y$ [%]	Stress at break, $\boldsymbol{\sigma}_{b,}$ [MPa]	Strain at break, $\boldsymbol{\varepsilon}_b$ [%]
ATO	0	0.6 ± 0.0	16.8 ± 2	5.8 ± 1.6	27.0 ± 2.0	1890.1 ± 19.1
	10	1.2 ± 0.0	24.8±7.8	4.5 ± 1.6	31.0 ± 7.0	1091.1 ± 101.0
	20	1.3 ± 0.0	13.1 ± 7.6	1.8 ± 0.6	17.0 ± 4.1	151.0 ± 22.0
HP	0	0.8 ± 0.0	24.8± 3.9	7.6± 1.7	29.3± 2.0	1221.6± 30.4
	10	1.2 ± 0.2	32.4± 0.2	5.6± 0.8	38.8± 7.4	1730.0± 206.0
	20	1.0 ± 0.2	20.9 ± 4.1	4.3 ± 0.9	32.9 ± 1.8	1176.0 ± 9.9
	20-20B	1.5 ± 0.3	27.7 ± 3.8	3.3 ± 1.0	27.8 ± 3.9	119.5 ± 9.2
	30-30B	1.3 ± 0.2	19.2 ± 1.6	2.3 ± 0.7	7.8 ± 3.5	6.3 ± 0.5

Enhancement of the elastic modulus and strength at yield was observed together with lower strain at yield, especially very strong reduction in strain at yield ($\boldsymbol{\varepsilon}_y$) was observed for the compositions with highest nanofiller content (K30-HP-30B). If the properties at break will be compared it can be noted that slight increase in stress at (K10-HP and K20-HP) relative to neat HP and reduction in other cases is observed. Strain at break shows a significant decrease as the content of fillers increases. It can be conclude that an introduction of a high melt flow index polymer (Borealis, MFI 26 g/10min) facilitates the uniform distribution of kaolinite in the K20-HP-20B matrix as the elastic modulus and yield strength is higher than that of K20-HP.

But as the content of filler increases it results in formation of agglomerates and decrease of mechanical properties is observed in K30-HP-30B (Table 3). Also extrusion of K20-ATO and K30-HP-30B filaments shows non-uniform distribution of fillers i.e. formation of agglomerates in longitudinal direction which acts as stress concentration centers, and results in decrease of mechanical properties and difficulty in drawing process. This experiment was performed in order to evaluate the effect of kaolinite content on mechanical properties of PP nanocomposites and also effect of drawing by considering nanocomposites containing equal amount of kaolinite. Representative stress – strain curves of the quasi-static tensile tests for as-spun (DR=1) and drawn fibers (DR=7) of Atofina and HP500 polypropylene matrix at 10% of kaolinite content are reported in figure 4.

It is important to note that undrawn fibers manifest a clear yield point at a low strain followed by a wide plateau (of cold drawing) and a strain hardening region until the break point. During this phenomenon tensile strength further increases and the stress whitening, due to the crystallization of aligned macromolecules takes place [14]. Drawing process produces a strong orientation of the macromolecules along the draw

direction and the strain-induced crystallization in the amorphous regions, which accounts for the increase in the fibers stiffness and the disappearance of yielding phenomena.

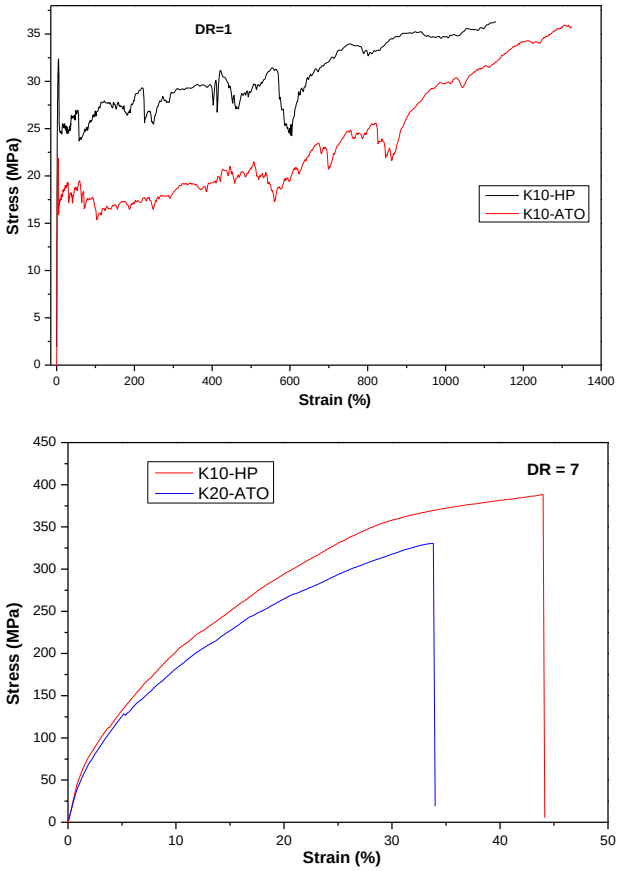

Figure 4. Representative tensile stress-strain curves of (a) as-spun (b) drawn sample of neat PP and kaolinite filled nanocomposites

Tensile modulus (E) of the PP-kaolinite nanocomposites fibers as a function of the draw ratio is represented in tigure 5. In this figure the results for the compositions with nanofiller content from 10 up to 30% were depicted in HP, ATO and HP-B mixture PP matrix. In case of the neat PP and nanocomposites fibers enhancement in elastic modulus in all draw ratio range was obtained. It is evident that kaolinite particles raise the fibers stiffness, especially at higher draw ratios (DR>10), modulus of PP-Kaolinite nanocomposites of K10-HP, K20-HP and K20-HP-20B fibers are significantly increasing over neat HP polypropylenes. It is also important to see that for compositions with HP polypropylene at 20wt% of kaolinite, better drawability with maximum modulus equal to 19 GPa was obtained (DR=23) in comparison to 11.5 GPa of neat PP (DR=19). A negative effect of high concentrations of kaolinite on modulus is observed for composition with 30% of filler (K30-HP-30B) that results in decrease of modulus and drawability, which can be explained in terms of the aggregate formation and incomplete filler dispersion.

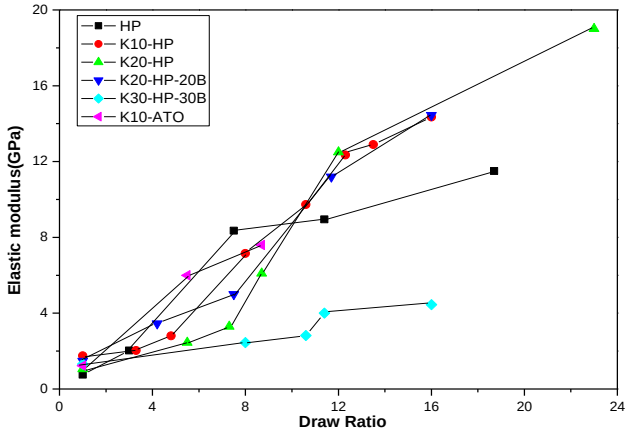

Figure 5. Elastic modulus of neat and nanocomposites PP fibers with different fractions of kaolinite as a function of draw ratio

High melt flow PP, K10-ATO shows less drawability but has improved elastic modulus at lower draw ratio. Production of K20-ATO shows formation of high agglomerates which is difficult for drawability and analyzing mechanical properties. In case of 10 and 20% HP-kaolinite nanocomposites good dispersion of kaolinite aggregates within the matrix may lead to relatively lower stress concentration and cracking nucleation phenomena and consequently it is responsible for an improvement of strength properties.

It is well known that the stress at break of compositions usually decreases with the addition of the nanofiller. Figure 6 shows stress at break of different neat PP and PP-kaolinite nanocomposites at fractions of 10, 20 and 30% of kaolinite filled fibers as a function of draw ratio.

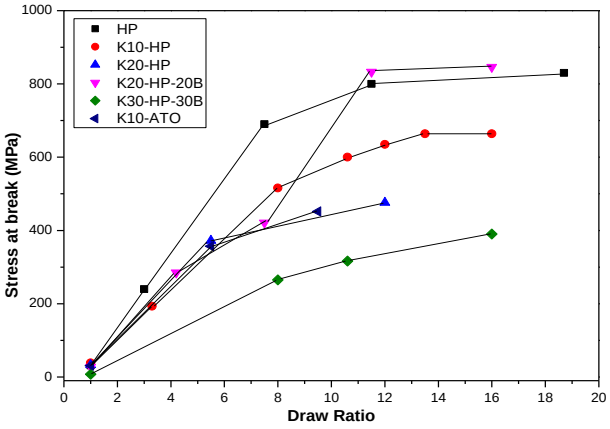

Figure 6. Stress at break of different neat PP and PP-kaolinite nanocomposites at fractions of 10, 20 and 30% of kaolinite filled fibers as a function of draw ratio

Considering the results for fibers with 10, 20 and 30 wt% kaolinite content, it can be seen that higher stress at break was observed only at higher draw ratio. In particular for DR=12, stess for neat PP (800 MPa) was lower than reported for K20-P-20B (833 MPa). In this specific case it is important to understand differently from common cases where the stiffening effect is accompanied by reduction of the tensile strength, in this case of K20-HP-20B at high draw ratio of fibers a parallel enhancement of both modulus and stress at break was observed (Figure 7). In all other cases the stress at break of kaolinite filled PP is lower than that of neat HP polypropylene.

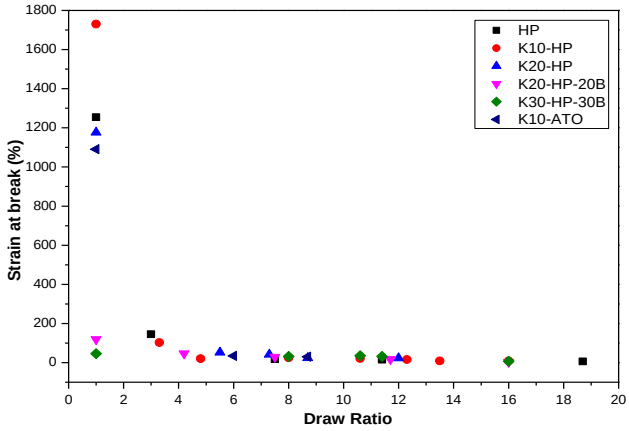

Figure 7. Strain at break of different neat PP and PP-kaolinite nanocomposites at fractions of 10, 20 and 30% of kaolinite filled fibers as a function of draw ratio

Strain at break versus draw ratio for different neat and PP-K nanocomposites fibers shown in figure 7 illustrate a significant reduction of strain at break for a high draw ratio. In both neat and nanofilled fibers the behaviour was very similar in drawing process i.e. as the drawing ratio increases the strain at break decreases in both cases. The decrease of the strain at break for higher filler loading might be due to the decreased deformability at the interface between the filler and matrix. From these results it is possible to conclude that for fibers drawn with higher draw ratio (DR>10) significant improvement in modulus is observed for 10 and 20% kaolinite and decrease in modulus for 30% filler content. Also at higher draw ratio stress at break of compositions decreases with the addition of the nanofiller, but it is improved at K20-HP-20B, and significant reduction of strain at break was observed. This behaviour can be related to the homogeneous kaolinite dispersion inside the polymer matrix, which enhanced the filler-polymer interfacial adhesion and is responsible for the improvement of mechanical properties.

Efficiency of filler

The efficiency of filler (EF) for each composition is evaluated from the difference of nanocomposites modulus (E_{NC}) and modulus of neat PP normalized to the volume fraction of the filler (f) and modulus of PP (E_{PP}).This approach in the case of aligned fillers is described by eq. 3.

$$EF = \frac{E_{NC} - E_{PP}}{f\,E_{PP}} \quad \ldots\ldots\ldots\ldots (3)$$

The filler efficiency in table 4 was calculated by taking modulus of 0.59 GPa for neat Atofina and 0.75 GPa for neat HP500 PP matrix at DR=1. These results implies that the effective crystallinity of 20 wt% kaolinite content is very poor compared to 10wt% kaolinite in Atofina polypropylene matrix. Depending on the tensile modulus effectiveness and other mechanical properties results observed for as-spun fibers it is easy to conclude that fibers of K20-ATO is difficult to be drawn. Therefore, the drawing process was selected preliminary for thefibers of K10-ATO nanocomposites and a maximum result ofdraw ratio of about 10 was found with 7.6 GPa of elastic modulus and strength of about 450 MPa.

Table 4. Values of filler efficiency for as-spun fibers

Draw ratio	Fiber	Elastic modulus (E_{NC}) (GPa)	Filler fraction from TGA	Filler efficiency
1	K10-ATO	1.23	0.064	17
1	K20-ATO	1.27	0.156	7.4
1	K10-HP	1.2	0.074	8.1
1	K20-HP	1.07	0.164	2.4
1	K20-HP-20B	1.48	0.153	6.4
1	K30-HP-30B	1.31	0.242	3.1

In a similar manner the effective tensile modulus calculated for K10-HP and K20-HP with the results of 8.1 and 2.4, respectively shows that the efficiency of filler with 20% kaolinite content is very poor compared to 10% kaolinite in HP polypropylene matrix.

Conclusion

According to the above results of spinning and drawing of K-HP and K-ATO fibers, it is possible to conclude that K-HP fibers are performing well in: thermal stability, drawability, mechanical properties, hosting high content of fillers and facilitating filler dispersion. Nanofiller presence affects the spinnability and drawability especially in high MFI PP and at higher content of kaolinite in the polymer matrix. Hot drawing in fibers induces the rupture of kaolinite aggregates and controls the intercalation or partial exfoliation of kaolinite in the PP matrix. Formation of larger agglomerates acts as stress concentration centers and results in difficulty of drawability and reduction in mechanical properties of fibers.

Conflicts of interest

Authors declare no conflict of interest.

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