

**Research** Article

# Structural analysis 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. The drawn fibres were characterized for their microstructure using X-ray diffraction (XRD), Transmission Electron Microscopy (TEM), Field Emission Scanning Electron Microscopy (FESEM), and Thermogravimetric Analysis and the resuls are presented in this paper. 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. Drawing of K30-HP-30B and K20-ATO nanocomposite is difficult due to large agglomerates formations. In as-spun fibers only one  $\beta$ -relaxation peak can be observed while two peaks of  $\beta$  and  $\alpha$  occurs for drawn fibers in DMA analysis. XRD also reveals a strong orientation of kaolinite along the chain axis after drawing of nanocomposite. There is a significant change in crystallinity transform of the  $\alpha$ -phase structure into a  $\gamma$ -phase. Even though TEM micrographs of K20-HP shows overcrowded kaolinite particles it is possible to observe improvement of kaolinite dispersion with drawing process.

**Keywords:** Polypropylene; Nanocomposites; Kaolinte; Transmission Electron Microscopy; Field Emission Scanning Electron Microscopy.

## 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 = 15 g/10 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 = 25 g/10 min). They observed not only a higher thermal stability and nucleation effect of the

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nanofiller, but also a remarkable increase in the strength, as consequence of tensile 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: meltspinning 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 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 wt% 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. The drawing was explained in detail in our earlier paper [13].

## Materials and methods

## Fibers

Fibre production, drawing, and the tensile properties of the fibres produced were presented already [13]. Compositions for fibers production were consider compounded formulations of F1 -13 and F16 - 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 2mm sizes. Extruded fibers of about 500 micron are drawn at 145 0C 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. Asspun 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.

### Dynamic Mechanical analysis

Dynamic mechanical thermal analyses were performed in order to evaluate the tensile behavior of the prepared nanocomposites at different temperatures. The tests were performed with a DMA Q800 testing machine (TA Instruments). The experiments were carried out in tensile mode by using single cantilever clamp on samples by applying a sinusoidal strain with a frequency of 1 Hz and amplitude of 64 microns. In order to prevent buckling of the fibers a prestress of 0.01 N was applied. A heating rate of 3°C/min, from -100 to 150 °C, was selected for all tests.

## X-ray Diffraction analysis

The X-ray diffraction (XRD) studies on as-spun fiber, filaments for 3D printing and drawn samples of PP-kaolinite was carried out. When a focused X-ray beam interacts with repeating planes of atoms that form a crystal lattice, part of the beam is transmitted, part is absorbed by the sample, part is refracted and scattered, and part is diffracted

## Transmission Electron Microscope analysis

TEM is an imaging technique where a beam of electrons is focused onto a specimen and passed through it causing an enlarged version to appear on a fluorescent screen or layer of photographic film. It gives two-dimensional pictures which might require a little bit of interpretation. TEM analysis has been performed on the ultramicrotomed cross section of as-spun, filaments and drawn nanocomposites fibers in order to get information about nanofiller dispersion inside the polymeric materials, their orientation after drawing and 3D printing, and also interfacial adhesion of the components.

## Scanning electron microscopy analysis

The fractured surfaces were observed through a Carl Zeiss AG Supra 40 field emission scanning electron microscope (FESEM), operating at an acceleration voltage of 3 kV. SEM is used to assess the surface morphology of neat polypropylene and PP-kaolinite nanocomposites. Specimens were immersed in liquid nitrogen for about 60 min and broken in a brittle manner.

#### Thermal Analysis

Thermal Analysis was performed by a TGA Q5000-IR from TA Instruments, in a temperature range of 30 to 700°C with heating rate of 10°C/min. Specimens of about 14 mg were tested in both air and nitrogen atmosphere at a flow rate of 100 ml/min.

#### **Results and discussion**

#### Dynamic mechanical analysis

DMTA analysis was performed on the as-spun and drawn fibers at selected draw ratioson neat HP and K10-HP to obtain deeper information on mechanical properties and molecular mobility. Due to a very high surface area of the nanoparticles in the PP-kaolinite nanocomposites, the applied sinusoidal stress is expected to be easily transferred from the matrix onto the kaolinite particles. Storage modulus (E') and loss modulus (E") of the neat HP and HPkaolinite fibers in range -100 to 150°C are reported in Fig. 12 (a) and (b) for as-spun and drawn fibers. For DR = 1 there is an increase in the storage moduli with the nanofiller content, K10-HP, which is observed over all temperature range used (Figure 1). With the drawing process, the storage modulus of neat PP and K-PP fibers increases over the whole temperature range used, which can be attributed due to the chain orientation induced bv drawing. Storage modulus of the composites with higher nanofiller fractions could be decreasing due to the formation of nanofillers aggregates, which reduces the filler effectively. Similar to the storage modulus (E'), loss modulus (E") of PPkaolinite composite fibers rises with the draw ratio and kaolinite content.

PP displays three relaxations (transitions, loss peaks) localized in the range of about -80°C  $(\gamma)$ , 10°C ( $\beta$ ) and 100°C ( $\alpha$ ), where  $\beta$  is the dominant relaxation. In the crystalline polymers the  $\alpha$  transition is commonly considered to be associated with the molecular motion within the crystalline regions, the  $\beta$  transition corresponds to the glass transition of the amorphous regions, and the  $\gamma$  transition is believed to be related to limited molecular motions of short chain sections in both amorphous regions and crystalline domains [14-16].

The intensity of the  $\beta$  relaxation depends on various factors, such as crystalline fraction, orientation of the amorphous phase, and nanofiller fraction. For all as-spun fibers only one  $\beta$  relaxation peak at around 13°C can be observed (Figure 2), whose position does not significantly change with varying nanoparticle content. But for drawn fibers double peak was observed. Neat HP fibers at draw ratio of 10 shows double peaks at 23 and 76°C while K10-HP at a draw ratio of 10 shows two peaks at (15 and 0.9°C) may be due to defects and 85°C and at draw ratio of 15 it shows double peaks at -24 and 76°C.



Figure 1. Mechanical properties: (a) Tensile modulus,(b) stress at break and (c) strain at break as a function of inverse of draw ratio for neat PP and nanocomposites with different amount of kaolinite

This implies that after drawing process,  $T_{\beta}$  shifts to higher temperatures for neat HP from 13 to 23°C at a DR = 10. However, it is quite

unusual the  $T_{\beta}$  values for K10-HP shifts to lower temperatures due to the drawing process. The effect was observed for drawn fibers (DR  $\geq$  10) and it can be attributed to the longitudinal defects developed during drawing that have been associated to a reduced density (and hence higher molecular mobility and lower  $T_{\beta}$ ), in accordance to literature results. In fact, the higher the drawing, the longer the extension of the band defects, almost along all the fibers length [17,18] at very high draw ratio.



Figure 2. DMA results of storage and loss modulus for neat HP and K10-HP nanocomposites (a) as-span and (b) drawn samples

As far as the  $\alpha$  loss modulus peak is concerned, it can be seen that this relaxation is not observed for the as-spun fibers, while for drawn materials a peak occurs at 76 °C for neat HP at DR=10 and K10-HP at DR=15 and 85°C for K10-HP at DR=10. The magnitude of the  $\alpha$ loss modulus peak increases proportionally to the draw ratio and/or filler fraction. This enhancement observed after drawing is probably related to the increase in the fraction of crystalline phase induced by the drawing process. This  $\alpha$  relaxation shifts to higher temperatures with increasing nanoparticle content and/or draw ratio. Owing to the kaolinite

content at DR = 10, the peak shifted from 76°C for neat HP to 85°C for K10-HP. It is believed that higher crystalline continuity and/or dimensions achieved at high draw ratios hinder the underlying molecular motion so that the onset of the transition is shifted towards higher temperatures [1,19].

#### **XRD** analysis

WAXS experiments were conducted to monitor kaolinite crystal structure in the PP matrix as well as changes in the crystallinity of PP in the composites. XRD Rietveld fitting of PP-Kaolinite powder using a modulated turbostratic model for the stacking disorder along the 001 direction was given in figure 3. The crystal information of PP is reflected predominantly on the higher angle  $2\theta$  peaks. The peak at  $18.58^{\circ}$  in figure 3 is characteristic of the ∝-phase which suggests that the PP matrix is predominantly in its &-form both in the neat polymer and in the PP-kaolinite composites. Drawn fibers show the relative intensity of the peaks formation at  $21.18^{\circ}$  and  $21.78^{\circ}$ , and the peak at 16.88<sup>0</sup> exhibits a significant decrease.

An additional peak formed for a drawn K10-HP fiber at 21.18 and 21.78° is attributed to a  $\gamma$ -form PP crystal lattice. These results suggest that the crystal lattice structure of the PP matrix is altered, and also there is a characteristic peak of the  $\gamma$ -phase formation at 20.278°. This implies that there is a significant change in crystallinity transform of the  $\propto$ -phase structure into a  $\gamma$ phase. This is also corroborated by the crystallization and melting temperatures obtained in DSC.

The degree of crystallinity (the intensity and height of peaks) is decreasing with respect to nanoparticle addition which probably results from the presence of large agglomerates. This can be evidenced by looking the peak at 16.88<sup>°</sup> which exhibits a significant decrease.X-ray transmission images of as-spun and drawn K10-HP fiberscollected in trasmission on a modified MAUD software development with an Image Plate detector was shown in figure 3.

By Rietveld fitting [20,21] of the X-ray fiber diffraction images, collected in transmission, polypropylene fibers dispersion could be obtained quantitatively along with the texture and deformation of the kaolinite basal planes. For instance, Figure 4 reports the raw diffraction image in transmission for as-spun K10-HP fibers of 500 micron and drawn fibers of 160 micron. Thus after drawing the polypropylene-kaolinite shows a strong orientation along the chains axis, the kaolinite texture is characterized by a (001) fiber like orientation with the basal/faulted planes perpendicular to it.



Figure 3. XRD Rietveld fitting of PP-Kaolinite powder using a modulated turbostratic model for the stacking disorder along the 001 direction

### Microstructure characterization

Microstructural studies of K10-HP fibers of as-spun and drawn at different draw ratio was performed by TEM on ultramicrotomed cross sections and it was compared with filaments for 3D printing and 3D printed filament. Also the dispersion of kaolinite in K20-HP-20B, K30-HP-30B and K20-ATO fibers are evaluated by SEM on the fractured surfaces of the composites.

### Transmission Electron Microscopy

TEM images of ultramicrotomed cross sections of K10-HP and K20-HP as-spun fibers and with selected draw ratios are presented in Figure 4. А set of low-magnification micrographs of all studied composites shows that the kaolinite particles are dispersed quite uniformly in the polymer matrix, but frequently they tend to form small agglomerates. In general, the amount and size of the agglomerates decreases for drawn samples, that is TEM micrographs reveals that the kaolinite nanoparticle agglomerates are partially destroyed at higher draw ratios (Figure 5).

Even though TEM micrographs of K20-HP shows overcrowded kaolinite particles it is possible to observe improvement of kaolinite dispersion with drawing process. By comparing micrograph of K20-HP drawn at DR = 9 and DR = 20 it was clearly seen that dispersion and disaggregation of kaolinite particles increases with draw (Figure 6). The nanofiller dimensions are found between 1  $\mu$ m and 100 nm. But it is very difficult to estimate exactly the size of the aggregates because they are non-isometric and randomly dispersed in matrix.



Figure 4. X-ray transmission image of HP fibers with 10% kaolinite (a) as-spun 500  $\mu$ m (b) after drawing 160  $\mu$ m (DR=10)



Figure 5. TEM micrographs of K10-HP at different diameters of as-spun and draw ratios (a) as-spun filaments of 670  $\mu$ m (b) drawn fibers of 220  $\mu$ m, DR = 9 (c) as-spun filaments of 400  $\mu$ m (d) drawn fibers of 120  $\mu$ m, DR = 11



Figure 6. TEM micrographs of K20-HP of; (a) as-spun filaments of 680  $\mu$ m (b) drawn fibers of 230  $\mu$ m, DR = 9 (c) drawn fibers of 150  $\mu$ m, DR = 20

The size of the aggregates is, therefore, strongly dependent on orientation of particles. It can be concluded that the drawing process induces the rupture of kaolinite aggregates and controls the intercalation and/ or partial exfoliation of kaolinite in the PP matrix, which accounts for improvement of the mechanical properties of nanofilled fibers.

### Field Emission Scanning Electron Microscopy

dispersion of The kaolinite in polypropylene was also evaluated by FESEM on the fractured surfaces of the composites. Figure 7(a) and (b) shows dispersion of fillers in the matrix of HP-20B with 20% (w/w) kaolinite fibers. Addition of 20% Borealis (MFI of 2.6 g/min) in HP500 (MFI of 0.18 g/min) facilitates dispersion of kaolinite as shown in image of K20-HP-20B, figure 7(a) and (b) even though there is tendency of formation of agglomerate due to hydrogen bonds formed between kaolinite particles. But at higher content of kaolinite i.e. for K30-HP-30B addition of Borealis does not facilitate the dispersion of kaolinite. As it is shown in figure 7(c) and (d) there is a lot of agglomeration which affect the properties of nanocomposites detrimentally. Figure 7(e) and (f) shows FESEM micrograph of Atofina (MFI of 1.2 g/min) with kaolinite of 20% and it reveals that there is a lot of kaolinite agglomerate formation.

Figure 7(a), (c) and e reveals formation of holes due to weak interfacial adhesion between PP and kaolinite and there is subsequent plastic deformation in the morphology of kaolinite filled nanocomposites. This is due to the wide difference in character between kaolinite and the PP matrix. The amount of holes also implies the content of kaolinite, i.e. the numbers of holes formed in K30-HP-30B is much higher than that of K20-HP-20B. The most common failure mechanism during the deformation process of particulate-filled polymer composite is debonding at the filler-matrix interface. From the micrograph Figure 7(c) at a magnification of 120 debonding process of kaolinite aggregates created large holes, which means that kaolinite filler can be pulled out completely from the PP matrix through the interfacial failure as the filler-matrix adhesion is relatively weak. The reason for poor adhesion between kaolinite and PP was probably because of the difference in surface free energy (or polarity). Thus it is possible to conclude that the drawability reduction in and mechanical properties of fibers of K30-HP-30B and K20-ATO observed in drawing and mechanical testing is due to lack of kaolinite dispersion in polymer matrix and large agglomerate formation. These results show clearly the **PP-kaolinite** kaolinite content used in nanocomposites should be at a limited content. In this case it is also better to understand that the formation of agglomerates is not only depending kaolinite content but also on the on polypropylene type.



Figure 7(a)-(b). FESEM micrograph of the fractured surface of K20-HP-20Bfibers (a) X450 (b) X15,000



Figure 7(c)-(d). FESEM micrograph of the fractured surface of K30-HP-30B fibers (a) X400 (b) X100,000



Figure 7(e)-(f). FESEM micrographs of the fractured surface of K20-ATO fibers (a) X120 (b) X50,000

## Thermogravimetric analysis

In order to analyze the influence of nanofiller at different content on the thermal stability of HP, ATO and HP-B mixture polypropylene, TGA analysis was performed in air atmospheres and representative TGA curves of fibers are reported in figure 8. In these curves, it is possible to observe three different zones. The first zone (30 to 250°C) shows no significant weight loss, second zone (250 up to 422°C) shows high mass loss due to thermal decomposition of polymer matrix and the last zone which is between temperature zones of (422°C up to 700°C) in air atmosphere, shows almost no significant mass loss since the residuals are not decomposed.



Figure 8. TGA curves and its derivatives of neat and nanofiller PP-K fibers air atmosphere

The main important results in the analysis of TGA for characterization ofthermal stability, which are % weight at 420-430°C and the residual yield at temperature of 700°C were shown Table 1. Based on the results, it is possible to conclude that the percentage weight at 420-430°C and residual yield of each PP-K increases nanocomposites with increasing kaolinite content. The comparison of thermal stability was carried out in terms of selected decomposition temperatures. in particular degradation at 420-430°C. As shown in table 1 percentage remains in these temperature range was varying based on the polymer matrix. Low melt flow index polymer (HP500, 0.18 g/min) nanocomposites with kaolinite has relatively low loss compared to higher melt flow index polymer (Atofina, 1.2 g/min). For example the percentage remains at temperature range of 420-430°C is 10.1, 20.6 and 30.3% for K10-HP, K20-HP and K30-HP-30B respectively while it was 9.1 and 20.4% for K10-ATO and K20-ATO respectively, which is almost expected percentage content of filler added compared to 0.85% for neat HP polypropylene. Following Gilman suggestion [22], this behaviour is due to the hindered thermal motion of the polymer molecular chain. Residual mass at 700°C is directly dependent on the nanofiller content and type of PP matrix, ranging between 7.6, 16.4 and 24.2% for composition of K10-HP, K20-HP and K30-HP-

30B respectively while it was 6.4 and 15.6% for K10-ATO and K20-ATO, respectively. The incorporation of clay into a polyolefin matrix enhances its thermal stability by acting as a superior insulator and mass transport barrier to the volatile products generated during decomposition, making the diffusion path of the oxygen more complicated, and thus retarding the thermo-oxidative process [23].

Table 1. TGA result of different PP/kaolinite nanocomposites fibers at different compositions

Composition	Theoretical Kaolinite (%)	% of Ashes at 700°C	Difference	% at 420-430°C
Neat HP	0	0	0	0.9
K10-HP	10.0	7.6	-2.4	10.1
K20-HP	20.0	16.4	-3.6	20.6
K20-HP-20B	20.0	15.3	-4.7	19.3
K30-HP-30B	30.0	24.2	-5.8	30.3
K10-ATO	10.0	6.4	-3.6	9.1
K20-ATO	20.0	15.6	-4.4	20.4

### Conclusion

XRD reveals strong orientation of kaolinite along the chains axis after drawing nanocomposites fibers. Morphology analysis by SEM and TEM confirmed good dispersion of the filler even though they tend to form small agglomerates frequently due to hydrogen bonds formed between kaolinite particles. But at higher kaolinite content there is a formation of larger agglomerates which also depends on PP type; HP forms larger agglomerates at 30% while ATO forms at 20% of kaolinite content.

### **Conflicts of interest**

Authors declare no conflict of interest.

## References

- Toshio K. High-modulus and highstrength polypropylene fibers and films. In Polypropylene an a-z reference (ed.: Karger-Kocsis J.) Kluwer Academic Publishers, Dordrecht; 1999.
- [2] Wishman M, Hagler GE. Polypropylene fibers. In 'Handbook of fiber science and technology fiber chemistry' (eds.: Lewin P, Pearce EM) Marcel Dekker, New York; 1985.
- [3] Kearns JC, Shambaugh RL. Polypropylene fibers reinforced with

carbon nanotubes. Journal of Applied Polymer Science. 2002;86(11):2079– 2084.

- [4] LópezManchado MA, Valentini L, Biagiotti J, Kenny JM. Thermal and mechanical properties of single-walled carbon nanotubes–PP composites prepared by melt processing. Carbon. 2005;43(3):1499–1505.
- [5] Rangasamy L, Shim E, Pourdeyhimi B., Structure and tensile properties of nanoclay–PP fibers produced by melt spinning. J Appl Polym Sci. 2011;121(6):410–419.
- [6] Joshi M, Shaw A, Butola BS. Studies on composite filaments from nanoclay reinforced PP. Fibers and Polymers. 2004;5(3):59–67.
- [7] Dabrowska I, Fambri L, Pegoretti A, Slouf M, Vackova T, Kolarik J. Spinning, drawing and physical properties of PP nanocomposite fibers with fumed nanosilica. Express Polymer Letters. 2015;9(3): 77–290.
- [8] Zhang Y, Yu J, Zhou C, Chen L, Hu Z. Preparation, morphology, and adhesive and mechanical properties of ultrahighmolecular-weight poly ethylene/SiO2 nanocomposite fibers. Polymer Composites. 2010;31(8):684–690.
- [9] Rottstegge J, Qiao YK, Zhang X, Zhou Y, Xu D, Han CC, Wang D. Polymer nanocomposite powders and melt spun fibers filled with silica nanoparticles. J Appl Polym Sci. 2007;103(4):218–227.
- [10] Srisawat N, Nithitanakul M, Srikulkit K.
  Spinning of fibers from polypropylene/silica composite resins.
   Journal of Composite Materials.
   2011;46(2):99–110.
- [11] Srisawat N, Nithitanakul M, Srikulkit K. Characterizations of fibers produced from polypropylene/silica composite. J Mater Miner. 2009;19(20:53–58.
- [12] Dabrowska I, Fambri L, Batistella M, Lopez-Cuesta JM. Compounding and spinning of polypropylene nanocomposites with kaolinite. 16th European conference on composite materials, Seville, Spain; 2014.
- [13] Kilole T, Luca F, Nalankilli G. Compounding of nanocomposite made of

aluminium silicate Hydroxide/Polyolefin. International Journal of Industrial Engineering. 2018;2(1):21–30

- [14] Dorigato A, D'Amato M, Pegoretti A. Thermo-mechanical properties of high density polyethylene -fumed silica nanocomposites: effect of filler surface area and treatment. Journal of Polymer Research. 2012;19(5):9889–9900.
- [15] Matsuoka S., Relaxation phenomena in polymers. Hanser, Munich; 1992.
- [16] Boyd RH. Relaxation processes in crystalline polymers: Molecular interpretation-A review. Polymer. 1985;26(7):1123-1133.
- [17] Abo El, Maaty MI, Bassett DC, Olley RH, Dobb MG, Tomka JG, Wang IC. The formation of defects in drawn PP fibres. Polymer. 1996;37(3):213–218.
- [18] Amornsakchai T, Olley RH, Bassett DC, Al-Hussein MOM, Unwin AP, Ward IM. The influence of initial morphology on the internal structure of highly drawn polyethylene. Polymer. 2000;41(2):8291– 8298.
- [19] Alcock B, Cabrera NO, Barkoula NM, Reynolds CT, Govaert LE, Peijs T. The effect of temperature and strain rate on the mechanical properties of highly oriented polypropylene tapes and all-PP composites. Composites Science and Technology. 2007;67(5):2061–2070.
- [20] Lutterotti L, Bortolotti M, Ischia G, Lonardelli I, Wenk HR. Rietveld texture analysis from diffraction images. Zeitschriftfür Kristallographie Supplements, 2007;26(1):125–130.
- [21] Lutterotti L. Total pattern fitting for the combined size-strain-stress-texture determination in thin film diffraction. Nuclear Instruments and Methods in Physics Research, 2010; 26(8): 334-340.
- [22] Gilman, JW. Flammability and thermal stability studies of polymer layeredsilicate clay/ nanocomposites. Applied Clay Science. 1999;15(6):31–49.
- [23] Costantino U, Gallipoli A, Nocchetti M, Camino G, Bellucci F, Frache A. New nanocomposites constituted of PE and organically modified ZnAl-hydrotalcites. Polymer Degradation and Stability. 2005;90(12):586–590.

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