Interrelation between extrusion process, formulation and properties of polypropylene/flax fibers composites

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ABSTRACT

This work investigates the effect of extrusion parameters and formulation on the properties of polypropylene / short flax fibers composites. The parameters that were varied during the twin-screw extrusion process were screw configuration, screw rate, extrusion temperature and flow rate. The effect of the location of the feeding zone of flax fibers is also considered. Regarding the composite formulation, the effect of flax and hemp content, presence of coupling agent and of a reactive additive on composite properties were analyzed. Composites obtained with hemp fibers in the same extrusion process were also considered for comparison purposes. The materials were characterized in terms of morphological characteristics, fiber length distribution, rheological, thermal and mechanical properties. The variation of extrusion parameters showed that polypropylene / flax fibers composites have a large process window that can be considered as an advantage of PP/flax compounds. Tensile strength evaluated on injection molded parts increased up to 50% with flax fibers content when coupling and reactive agents are used. Due to their high flexibility, flax fibers are less oriented in the flow direction and the mechanical properties will be more isotropic. Furthermore, these composites showed a good recyclability using an injection molding process by keeping the integrity of their mechanical properties after five reprocessing cycles. The processability of these materials using a cast film line and the mechanical properties of obtained sheets are also presented.

INTRODUCTION

Polypropylene/natural fibers composites have attracted the attention of researchers and manufacturers because of the renewable nature of fibers, good mechanical properties, lack of abrasion during the compounding, low environmental impact, low weight and cost savings compared to PP/glass fibers,

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Kevlar or carbon fibers counterparts. On the other hand, natural fibers have as drawbacks low thermal stability, high humidity sensitivity, limited fiber length and, most importantly, the intrinsic variability. This variability consists in harvest conditions, quality of the soil and climate, geographic location, and the preconditioning. This variability can be observed also when considering the same plant because the fibers obtained from the stem have different mechanical and physical properties compared to fibers obtained from leaves. Composites mechanical properties and, therefore, their final applications, depend on fiber source [1-4], fiber treatment [5-9], the presence of coupling agents [1, 10-12], and compounding parameters.

If a lot of work has been done with regards to natural fiber surface treatment and polypropylene/fiber adhesion, the pre-compounding and the melt compounding aspects have less attracted attention. Concerning the compounding of polypropylene/short cellulosic fibers composites, usually a co-rotating twin-screw extruder is used before injection molding of different object shapes. On the other hand, some work exists concerning the effect of natural fibers content on the fiber damage during composites extrusion but without considering a discussion on the effect of extrusion parameters [13, 14].

This paper discusses the effect of extrusion parameters, i.e. screw configuration, screw rate, fiber feeding zone, temperature, and flow rate on the properties of polypropylene/flax fiber composites for a given composite formulation. Feeding the natural fibers in a continuous extrusion process is often problematic due to its low bulk density, which will be reflected into an inconsistent feeding rate [15]. The increase of the bulk density of natural short fibers is thus necessary to make the composite manufacture possible at small and medium scale. Hence, the flax fibers were fed in a pelletized shape obtained by passing the fibers trough a pellets mill. After setting the optimal extrusion parameters, the effect of fiber concentrations and additives on composite properties was examined. The composites re-processability using an injection molding step and the processability using a cast film line are also discussed.

EXPERIMENTAL PART

Materials

Isotactic polypropylene (iPP) Pro-fax 1274 from Basell BV (Hoofddorp, Netherlands) with weight average molecular weight of 300,000 was used as the composite matrix. Flax and hemp fibers with a content of 10 wt.% impurities, mainly shives, were provided by Schweitzer Mauduit, Canada. The concentration of fibers in polypropylene was varied between 10 and 40 vol.%. The maleic anhydride-grafted polypropylene used as coupling agent was Eastman Epolene-43 (E-43) (AN = 45, M_w = 9,100, with around 4.81 wt% of MA). Its concentration was varied between 2.5 and 5 vol.%. The calcium oxide (CaO) 98 wt.% purity from Laboratoire MAT Inc. was used as reactive filler. It is a basic reactive filler that has the role to absorb moisture in fibers, neutralize acidity of fiber impurities and therefore minimize the oxidation and degradation of fibers during melt processing.

Consequently, CaO helps also to increase strength and modulus of the final composites. The recommended concentration of CaO in PP composites with natural fibers is 3.5 % vol. (10 wt.%) [16]. Glass fibers, 6.5 mm in length, are also used to process equivalent composites with the purpose of comparison.

Extrusion Process

Regarding the appropriate way to feed natural fibers into the extrusion process, a pre-treatment step is necessary because natural fibers are fluffy and hard to feed in this shape without compromising the consistency of fiber flow rate. A proprietary technology was used to produce natural fiber pellets with minimum fiber length degradation. The extruder used to process the composites was a Leistritz 34 mm co-rotating twin-screw having 12 mixing zones and a L/D ratio of 40. Two feeding location are available in this extruder and the capillary die at the exit of the extruder had a diameter of 2 mm.

For the first part of this study, the composite formulation was kept constant, (i.e. 20 vol.% of flax and 2.5 vol.% of E43), while the extrusion parameters were varied. Three screw configurations, (a severe, a medium and a soft one) were tested. The degree of screw severity was differentiated by the ratio of shear disks situated in the 4^{th} and 8^{th} zones, i.e. 10/10 (Figure 1a), 5/5 (Figure 1b) and 5/0 (Figure 1c). For each configuration, the flax pellets were fed in two manners: at the same time as PP into zone 0, and secondly into the 5^{th} zone. For the extrusion temperature, 3 flat profiles (200, 185 and 170°C) were selected and one in which the temperature was decreased constantly from 200°C down to 170°C. Three total flow rates were used: 5, 7.5 and 10 kg/h and the screw rates were 100, 150 and 200 rpm.

In the second part of this study, the optimal extrusion parameters resulting from the first step were kept constant while the composite formulation was varied, and PP composites with flax, hemp and glass fiber contents from 10 to 40 vol.% were produced.

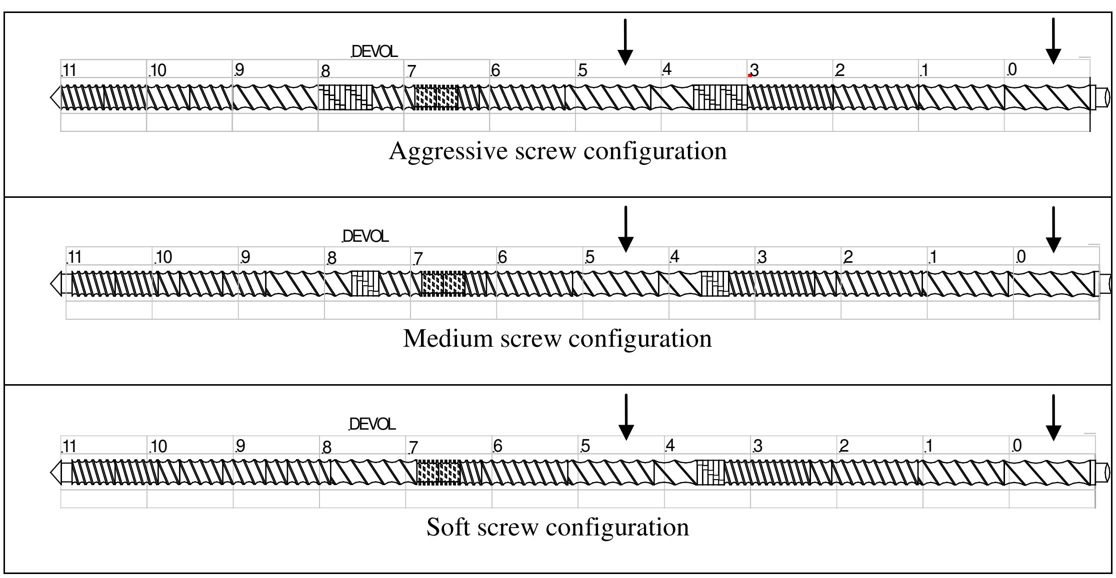

Figure 1. Screw configurations used in extrusion process

Sheet extrusion

The pellets prepared as described above were extruded using a cast film line i.e. a Davis-Standard single-screw extruder equipped with a 20 cm wide flat die. The melt temperature was 180°C and 1 mm thick sheets were calendered between water-cooled rolls maintained at 40°C. Only selected compounds were extruded into sheets, i.e. the ones containing 10 and 30 vol.% flax. Blends containing 10 and 30 vol.% glass fibers were also extruded into sheets for comparison purposes.

Morphology

Scanning electron microscopy (SEM) was carried out on polished composites surfaces, coated or fractured. A coating of gold/palladium alloy was applied on the specimens prior to the observation. A JEOL JSM-6100 SEM at a voltage of 10 kV was used to analyze the dispersion of the fibers into the matrix and the interface between fibers and PP matrix.

Fiber Qualitative Analysis

Fiber length and fiber length distribution of cellulosic fibers obtained from composites after PP dissolution in xylene were measured using a Hi-Res FQA analyzer from OpTest Equipment Inc., Ontario, Canada. The length range was set between 0.04 mm and 10 mm which was the maximum length that the FQA can detect. At least 5000 fibers were measured for each sample and at least 5 measurements were done for each formulation.

Rheology

The rheological properties of the composites were evaluated at 185°C using a rotational rheometer, the Advanced Rheometric Expansion System ARES, with a plate-plate geometry in a dynamic mode. The plate diameter was 25 mm and the gap was around 1.7 mm. Frequency sweeps were carried out to determine complex viscosity over a frequency ranging from 0.1 to 100 rad/s. The tests were conducted for a deformation of 15%. Care was taken to dry the materials at 80°C for 48 hours before testing. The samples were kept under a nitrogen blanket to minimize oxidation and to maintain a dried environment.

Differential Scanning Calorimetry

The DSC method was used to determine the melting and the crystallization temperature of the PP matrix. The tests were done using a TA Instrument's Q2000 calibrated using an indium standard. The samples were heated from 30 to 200°C at 20°C/min, kept at 200°C for 5 minutes to erase the thermal history and cooled again down to 30°C at 20°C/min. The PP heat of fusion was considered 207.1 J/g [17].

Mechanical Properties

The samples designated for mechanical testing were first dried and then injection molded using a Boy injection molding press with a screw temperature of 190°C and a mold temperature of 30°C. The tensile testing was carried out according to ASTM D638 on standard type I dogbone shaped samples with a thickness of 3.2 mm. The impact testing was carried out according to ASTM D259.

The tensile mechanical properties of cast sheets were also measured according to ASTM D638, in both machine and transverse directions. The tests were performed on rectangular 19 mm wide x 15 cm long strips cut out from the sheets. The elastic modulus, the tensile strength and the elongation at break were evaluated. A video extensometer was used to determine the elastic modulus. All reported values are the average of five tests.

Recyclability

The recyclability of flax composites was evaluated using tensile mechanical testing for formulations with 10 and 30 vol.% flax. The composite pellets were reprocessed by injection molding 5 times.

RESULTS AND DISCUSSIONS

Trials to feed the flax fibers into the extruder in their natural shape failed since their low bulk density gave an important variation of fiber flow rate or produced bridges into the feeder. An increase in the bulk density was obtained using a proprietary pellet technology. To facilitate the pelletizing, the bulks of flax fibers were first cut at 6.5 mm and then uniformly wetted using equivalent water content. In the pelletizing process, the wetted flax fibers were pressed through a die plate using two rotating roll mills.

The first row in Figure 2 presents the physical aspect of flax fibers in their original/as received state and after the pelletizing step. There was no thermal degradation or important fiber shortening observed after the pelletizing step. The temperature during the process was kept constant at around 70°C. As measured using a HiRes FQA device, the length average fiber length was around 3.2 mm before and after pelletizing as could be seen in the second row. The third row shows optical micrographs of initial fibers and the ones extracted from the flax pellets. Another significant advantage of this pelletizing step is the separation of the technical fibers (diameter around 100 µm) into elementary fibers (diameter around 20 µm). Prior to extrusion, the flax fiber pellets were dried at 80°C for a minimum 48 hours.

First compounding campaign of composites was dedicated to the variation of extrusion parameters as described previously in Experimental Part. The composite formulation was maintained constant, (77.5 vol.% PP, 20 vol.% flax and 2.5 vol.% E43), while screw configuration, feeding zone of fiber pellets and extrusion parameters were varied. The compounded composites were injection molded into samples for tensile and impact tests and the results are presented in Table I.

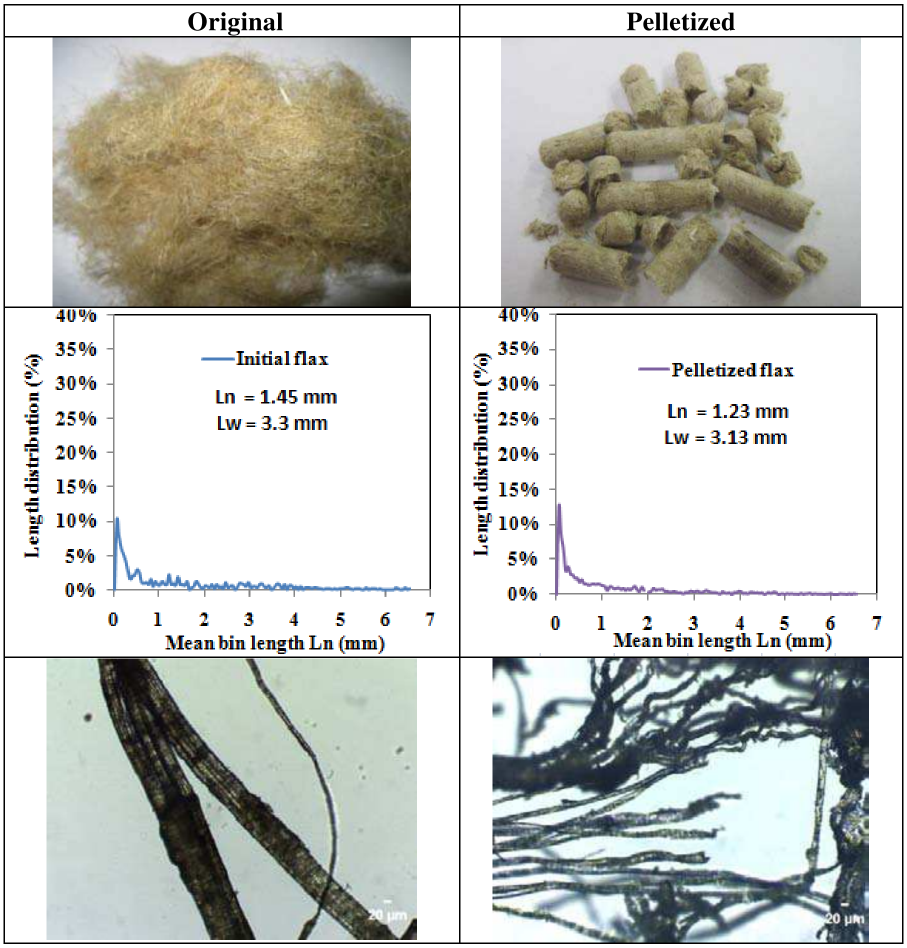

Figure 2. Physical aspect, fiber distribution, and optical micrographs of original and pelletized flax fibers

Injection molding sample of as-received polypropylene pellets has a tensile strength (TS) of 31.9 MPa and a tensile modulus (TM) of 1543 MPa. After extrusion, it was found that PP preserved its mechanical properties and this means that the screw configuration did not damage the matrix integrity. The addition of fibers led to an increase of TS and TM to about 37-39 MPa and 3000-3300 MPa respectively, no matter the extrusion parameters were. Usually, this split out of technical fibers into elementary ones has to take place into the extruder. As explained before, the flax technical fibers were separated in elementary fibers during the fiber pelletizing step that helped further the internal extruder work for fiber dispersion and distribution. The results presented in Table I demonstrate that probably the fiber attrition in the PP matrix was similar no matter the extrusion parameters were. It seems that the extrusion window of PP/flax composites is very large and it can be considered as an advantage of flax composite compounding.

Furthermore, fiber qualitative analysis using Hi-Res FQA was done on fibers extracted by PP dissolution for all composite samples compounded by varying the screw configuration and feeding zone. This study of the fiber attrition as a function of extrusion parameters was done with the purpose to decide on the best screw configuration with a minimum effect on fiber length. The results of FQA analysis are presented in the Table II. The fiber lengths are very similar no matter the extrusion parameters were. They confirm the mechanical properties presented in Table I so further helped us decide on the optimal extrusion operating conditions that were set at 185^{o}C, 100 rpm and 10 kg/h.

TABLE I. MECHANICAL PROPERTIES OF COMPOSITES OBTAINED BY THE VARIATION OF EXTRUSION PARAMETERS

Sample	TS (MPa)	TM (MPa)	IS (MPa)
Reference Polypropylene			
As received	31.9 (0.97)	1543 (64)	1.85 (0.54)
Extruded	31.1 (0.14)	1560 (78)	1.57 (0.37)
Variation of screw configuration and feed zone			
Severe, 0	39.0 (0.29)	3230 (361)	2.5 (0.29)
Severe, 5th	38.5 (0.31)	3232 (208)	3.0 (0.00)
Medium, 0	37.0 (0.28)	2980 (97)	2.7 (0.39)
Medium, 5th	37.7 (0.15)	3050 (107)	2.9 (0.52)
Soft, 0	38.0 (0.45)	3177 (172)	2.0 (0.00)
Soft 5th	38.4 (0.29)	3358 (196)	2.3 (0.37)
Extrusion temperature (^{o}C)			
200	40.2 (0.55)	3465 (137)	2.7 (0.37)
185	37.7 (0.15)	3050 (107)	2.9 (0.52)
170	39.2 (0.32)	3374 (151)	3.0 (0.75)
200 to 170	38.4 (0.22)	3282 (82)	3.0 (0.00)
Total flow rate (kg/h) and rpm			
5, 100	39.0 (0.29)	3356 (177)	2.9 (0.25)
5, 150	37.7 (0.15)	3050 (107)	2.9 (0.52)
5, 200	37.8 (0.19)	3215 (117)	2.4 (0.20)
7.5, 100	38.2 (0.35)	3273 (131)	3.0 (0.00)
7.5, 150	38.3 (0.19)	3379 (133)	2.9 (0.36)
7.5, 200	37.2 (0.76)	3200 (94)	2.5 (0.25)
10, 100	37.0 (0.72)	3182 (153)	2.8 (0.25)
10, 150	37.8 (0.64)	3268 (87)	3.0 (0.00)
10, 200	37.9 (1.08)	3245 (236)	2.8 (0.26)

Table II. Fiber length in composites obtained at the variation of extrusion parameters

Sample	Ln (mm)	Lw (mm)	Lww (mm)
Variation of screw configuration and feed zone			
Severe, 0	0.179	0.471	0.983
Severe, 5th	0.192	0.464	0.978
Medium, 0	0.191	0.443	0.883
Medium, 5th	0.203	0.459	0.894
Soft, 0	0.199	0.419	0.821
Soft 5th	0.197	0.398	0.768

PP/flax and PP/hemp composites were obtained using these optimal extrusion parameters. Figures 3 and 4 present the morphological features of longitudinal polished surfaces of PP/flax, PP/hemp, and PP/glass fibers extruded composites. The selected extrusion parameters gave a high-quality dispersion of fibers, no matter what the fiber type and fiber concentrations were. Comparing the orientation of the natural fibers with the one of glass fibers it is very evident that the glass fibers are more oriented in the flow direction due to their rigidity.

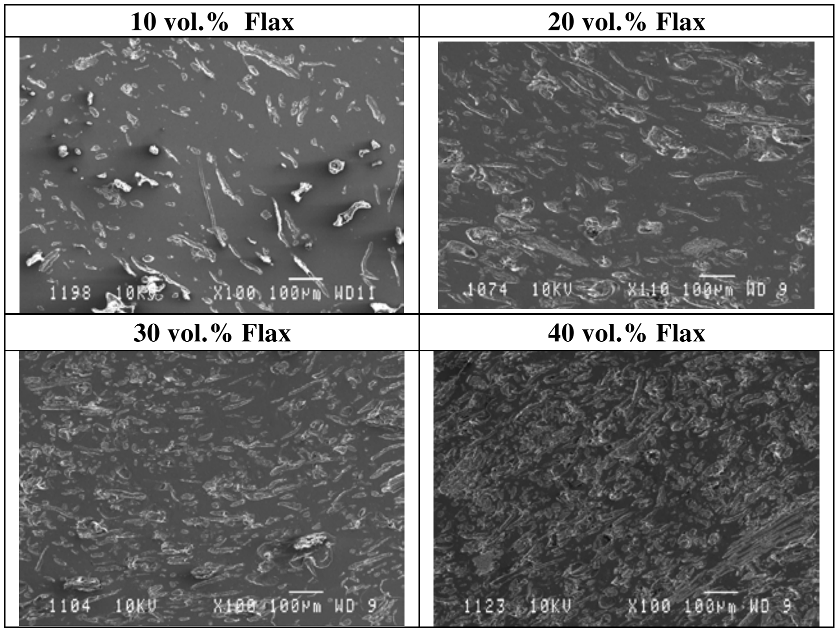

Figure 3. SEM micrographs of longitudinal polished surfaces of flax-composites obtained at 10, 20, 30 and 40 vol.% fibers.

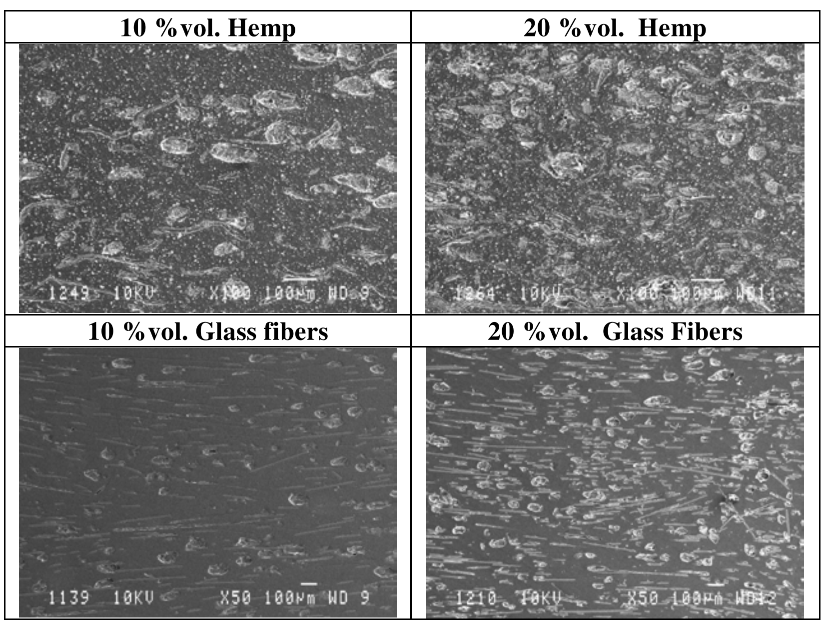

Figure 4. SEM micrographs of longitudinal polished surfaces of hemp-composites obtained at 10 and 20 vol.% fibers (hemp and glass) in the presence of E43 and CaO

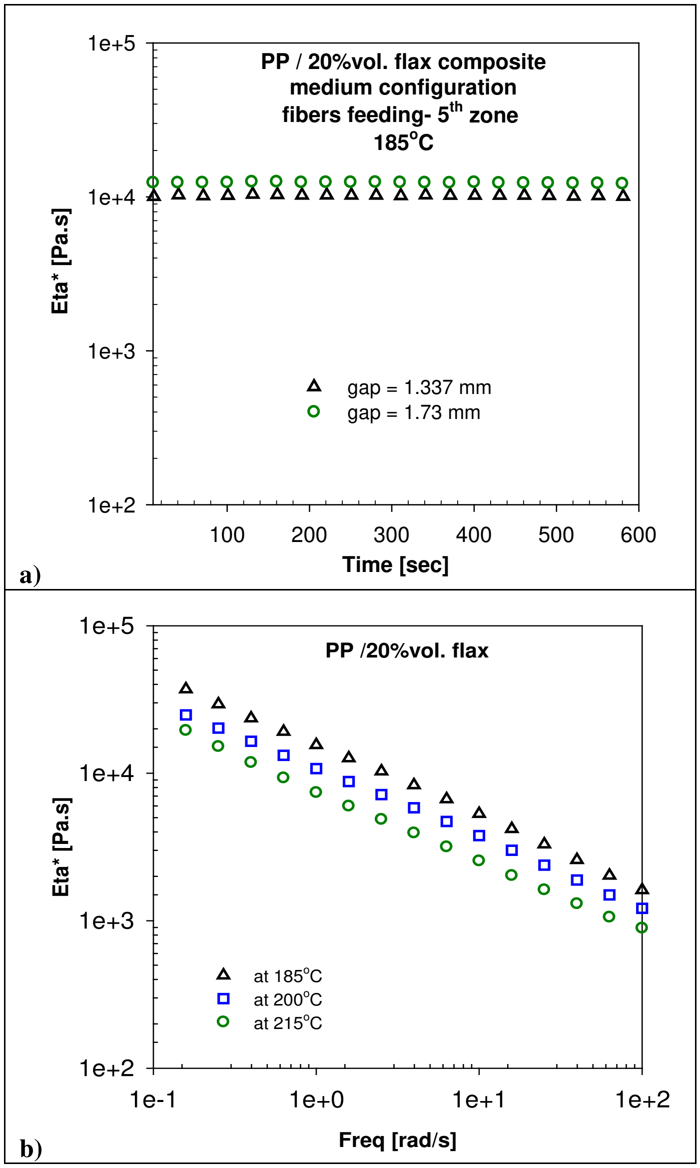

Figure 6. Complex viscosity as a function of testing time and as a function of testing temperature for neat PP/20 vol.% flax fibers

The complex viscosity dependency on test time and test temperature is presented first in Figure 6 for the neat PP/20 % vol. flax fibers. Composites stability in time is important during oscillatory tests to guarantee that the materials do not change their internal composition or structure at the testing temperature. In our case, it seems that the materials are very stable for at least 10 min at 185°C and, therefore, PP and flax fibers did not undergo thermal degradation during testing. Oscillatory tests done at two different gaps proved that composites viscosity depends on this testing parameter. For the rest of the rheological testing, a gap of 1.7 mm was selected. It is well known that the gap has to be three times higher or more than the fiber length to avoid the occurrence of wall-slip phenomena [18]. Figure 6b presents the complex viscosity dependency of PP/20%vol flax on frequency and temperature. The flexible fibers presence in PP matrix fingerprints the composite behavior, i.e. highly shear thinning without any Newtonian plateau. As expected, at increasing the temperature the composite viscosity decreased.

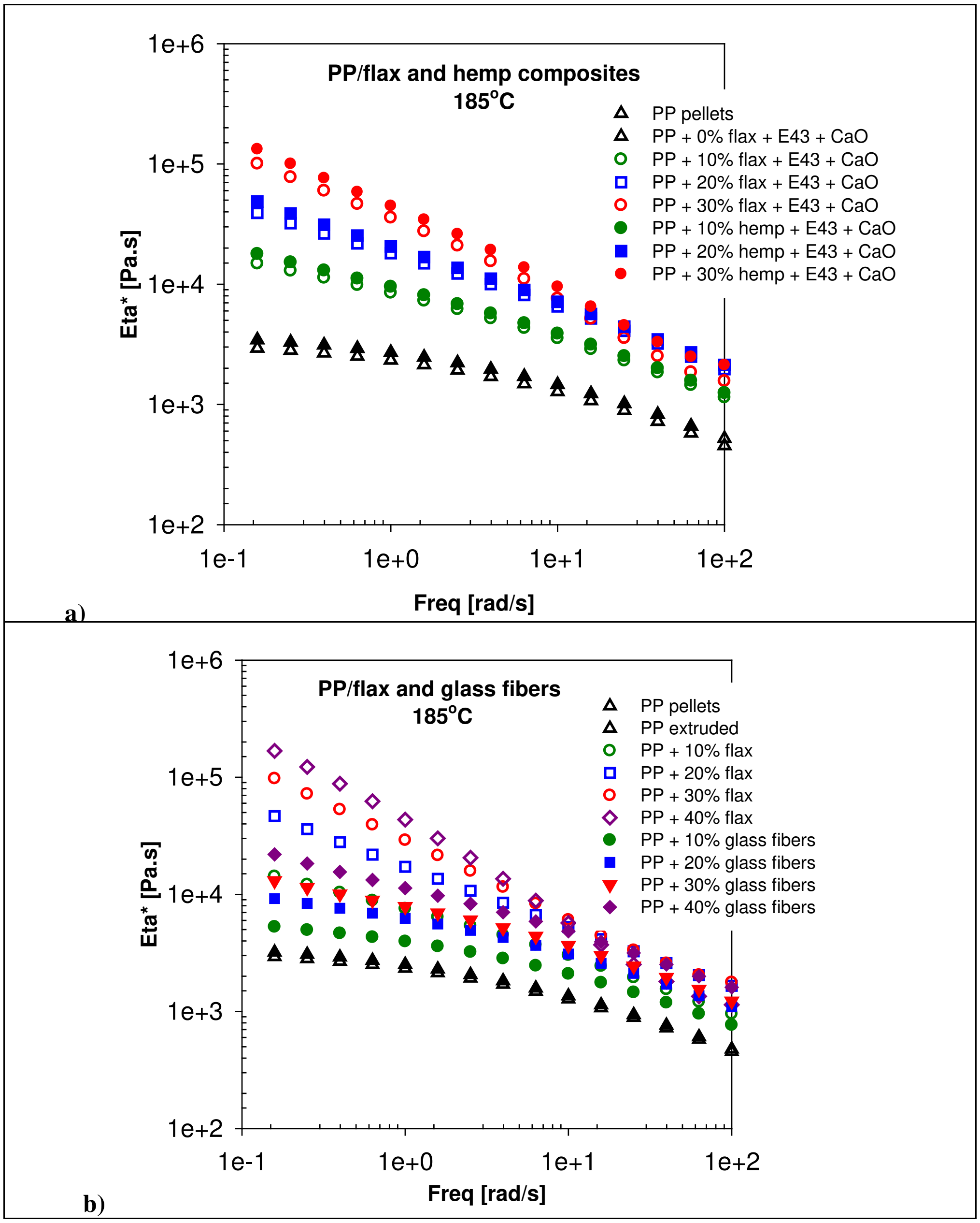

Figure 7. Complex viscosity as a function of frequency: comparison for PP/flax and hemp fibers composites (a) and PP/flax – glass fibers (b) respectively.

Viscosity measurements performed for different PP-flax, PP-hemp and PP-glass fibers composite systems obtained in similar extrusion conditions are presented in Figure 7. The extrusion process did not modify PP viscosity which is another proof that the selected screw configuration and extrusion parameters did not damage the length of PP molecular chains. The viscosity of extruded PP/E43+CaO is slightly higher than for pure PP. Their values are 3030 and 3568 Pa.s for a frequency of

0.1s^{-1}. Increasing the flax fibers content from 10 to 30 vol.% highly increases the composite viscosity from 16962 up to 130617 Pa.s, respectively. The viscosities of hemp fiber composites are slightly higher than for flax fiber composites. This increment in PP/flax or hemp composites viscosity with fiber concentration is due to the fiber flexibility and, therefore, to the increase of fiber-fiber interactions reflected in fiber-fiber entanglements. This can also explain the behaviors observed in Figure 7b. It shows that the complex viscosity of PP/flax composites is always higher that for PP/glass fibres composites at equivalent formulations. Moreover, higher fiber content leads to a more pronounced shear thinning behavior. It should be noted that the increase of the viscosity with fiber content is more noticeable for low frequencies. At high frequencies the effect weakened probably due to the fact that fibers coagulate together and the effect of the matrix becomes dominant.

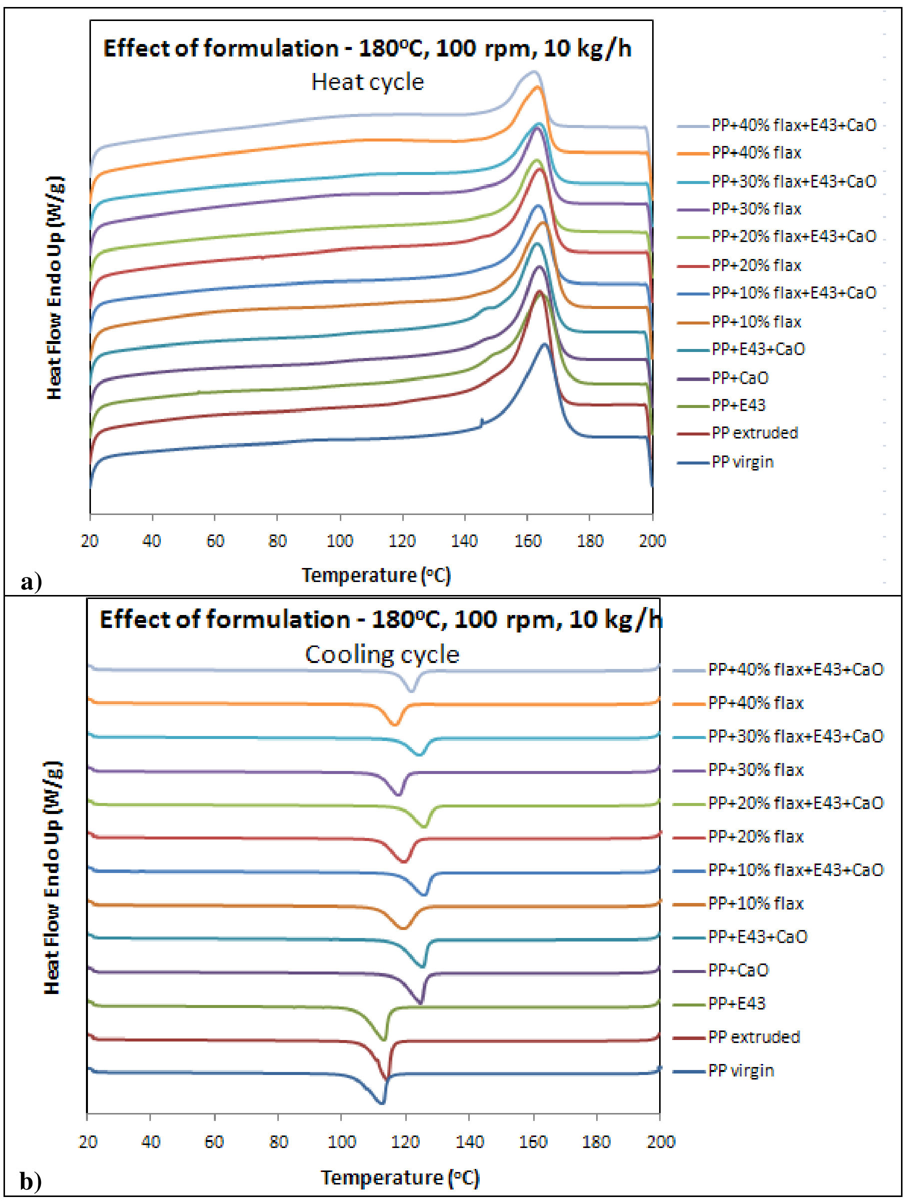

Figure 8. Heating (a) and cooling (b) curves obtained in DSC for PP/flax composites at different formulations

Non-isothermal crystallization behavior of PP/flax composites is presented in Figure 8. The first graphic discloses the heat curves and the second one the cooling curves obtained by DSC. The corresponding melt temperatures (T_m), crystallinity values (X_{ch}, X_{cc}) and crystallization temperatures (T_{cc}) data are additionally presented in Table III only for full formulations.

The heating curves show that the PP and its composites do not crystallize during the heating cycle because they are already fully crystallized. The fiber addition did not change the T_m that is kept at around 163^{o}C. In opposition, the initial crystallinity was highly increased with fiber addition, i.e. from 42% for pure PP up to 70% for the formulation with 40 vol.% flax fibers and CaO. In consequence, the fibers could act as nucleating agents in the crystallization process of composites. The crystallization temperature during the cooling, T_{cc}, was highly affected by the presence of CaO. It accelerates the crystallization that started 5-11^{o}C faster than for composites without CaO. The crystallinity reached during the DSC cooling was kept at about 44%. This crystallinity level of composites was most likely limited by the imposed cooling flow rate of 20^{o}C/min, and therefore by the limited time allowed for matrix crystallization.

TABLE III. NON-ISOTHERMAL DSC DATA FOR PP REFERENCES AND COMPOSITES WITH E43 AND CaO

	EXTRUSION					
	Heating			**Cooling**		
	Tm (°C)	**ΔHm (J/g)**	**Xch (%)**	**Tcc (°C)**	**ΔHcc (J/g)**	**Xcc (%)**
PP	165	85.97	42	112	104.30	50
PP extruded	164	100.00	48	114	109.90	53
PP+E43+CaO	163	87.26	42	125	94.43	47
PP+10%flax+E43+CaO	163	72.25	40	126	75.63	42
PP+20%flax+E43+CaO	163	75.04	50	126	67.71	45
PP+30%flax+E43+CaO	164	74.33	56	124	57.68	44
PP+40%flax+E43+CaO	162	77.77	70	122	50.14	45

Tensile properties of PP/flax composites obtained with the variation of flax concentration are presented in Figure 9. As expected, the tensile strength of composites obtained without compatibilizing additives decreased to some extent with the increase of fiber content. However, the addition of E43 coupling agent and CaO reactive agent promoting fiber/matrix adhesion and has an important effect on tensile strength of the composites. The tensile strength increased from 31 for pure PP up to 43 MPa for composites containing 30 vol.% of flax fibers, and a slight decrease to 40 MPa is observed for the composites consisting of 40 vol.% of flax fibers. Speaking in terms of tensile modulus, a regular increase is observed as a function of fiber loading. The tensile modulus of PP, around 1500 MPa, was increased more than three times when the flax fiber content was 40%vol. The tensile properties of PP/hemp fiber composites was not represented in Figure 9 because these composites shown very similar behavior as PP/flax composites.

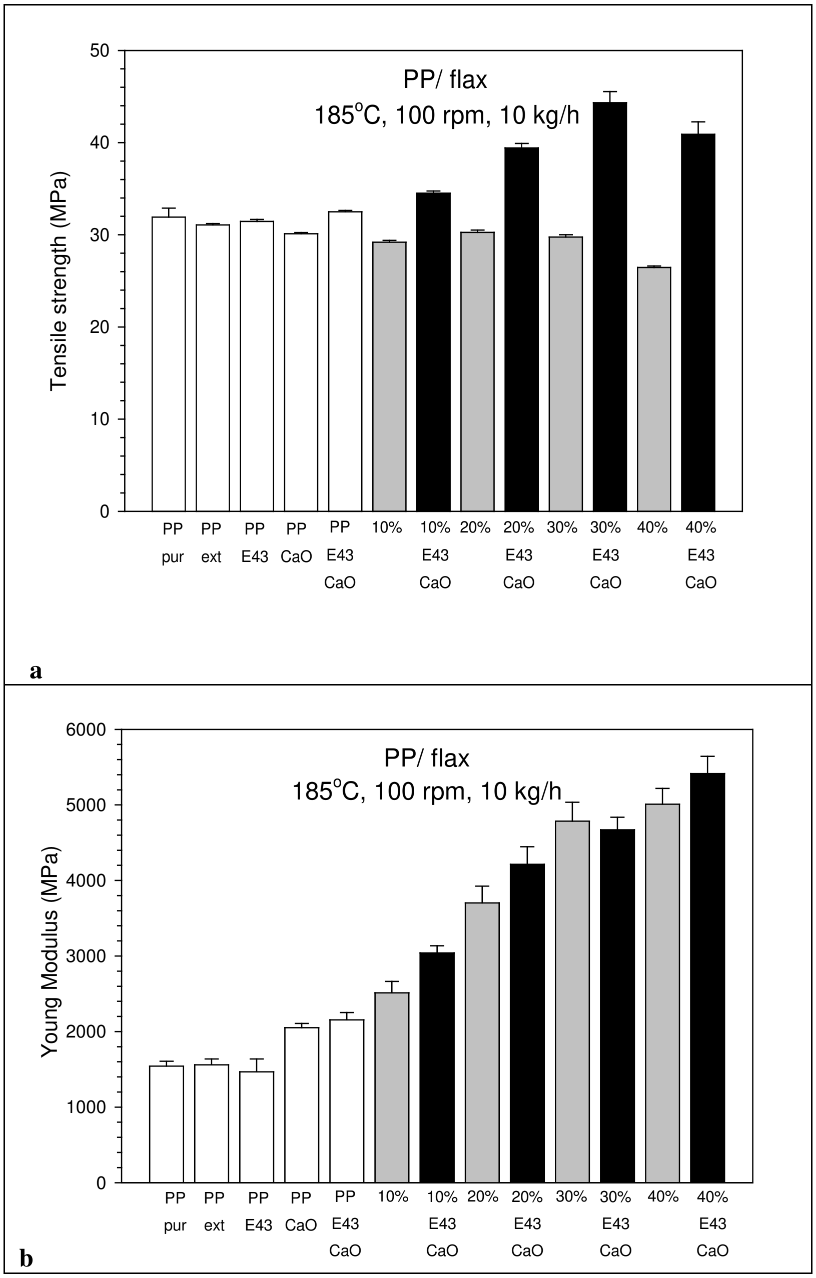

Figure 9. Tensile properties of PP/flax composites:
a) tensile strength and b) tensile modulus.

Micrographs corresponding to fractured surface of 10% flax composites (first column) and 30 vol.% flax composites (second column) without (first row) and with compatibilization (second row) are presented in Figure 10 and unveil the effect of coupling agent addition in PP/fiber adhesion. For composite that contains no additive, there is a smooth rupture surface and also more holes in matrix left by the pull-out flax. This proved a bad fiber-matrix interface. For composite that contained CaO and E43, fibrilar-like rupture and short pull-out flax could be observed. It proved that, at some level, a recovering of their good interface took place. When the matrix formulation contains both E43 and CaO, there is matrix attached or even covered on surface of the fractured triticale, which presented the best interface. This observation corroborates with tensile and flexural results explaining the roll of additives in the adhesion between the matrix and the flax. The excellent lessening of fiber pull out is an important sign of the increment of polymer/fiber adhesion in the compatibilized system, and it confirms the tensile strength higher values with the addition of E43.

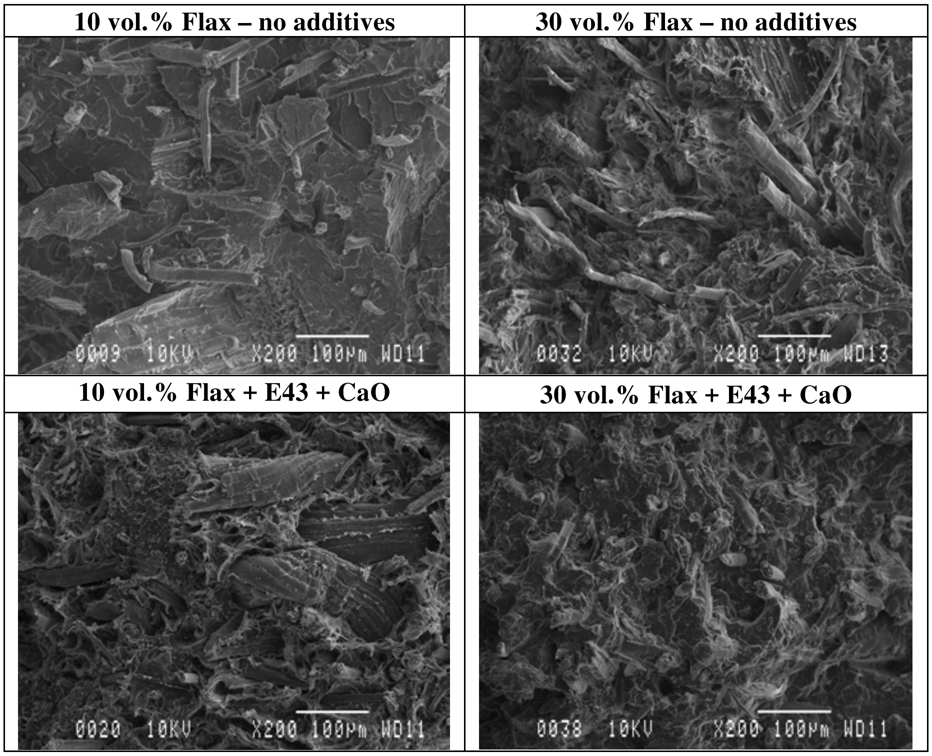

Figure 10. SEM microstructural details of fracture surface of injected samples PP/30 vol.% flax.

The mechanical performance of the cast sheets was also evaluated. The tensile modulus, tensile strength and elongation at break were evaluated in both machine (MD) and transverse (TD) directions. Figure 11 reports the data for the cast sheets of blends containing 10 vol.% flax and glass fibers. The values of the elastic modulus for neat polypropylene are similar in both MD and TD. The blends are less isotropic as the values in TD are slightly lower. This is even more pronounced for the blends containing glass fibers, which can, because of their higher rigidity, be more easily oriented in the machine direction during melt extrusion.

The modulus increases with the addition of flax fibers, with or without additives. The best results were obtained for the compatibilized blends (containing both CaO and E-43). These trends were also observed for the tensile strength. The elongation at break of neat polypropylene was very high (900%) in the machine direction but quite low in the transverse direction (20%). All blends, including the PP/glass fibers, exhibited very low elongation at break (below 10%), in both MD and TD (data not shown).

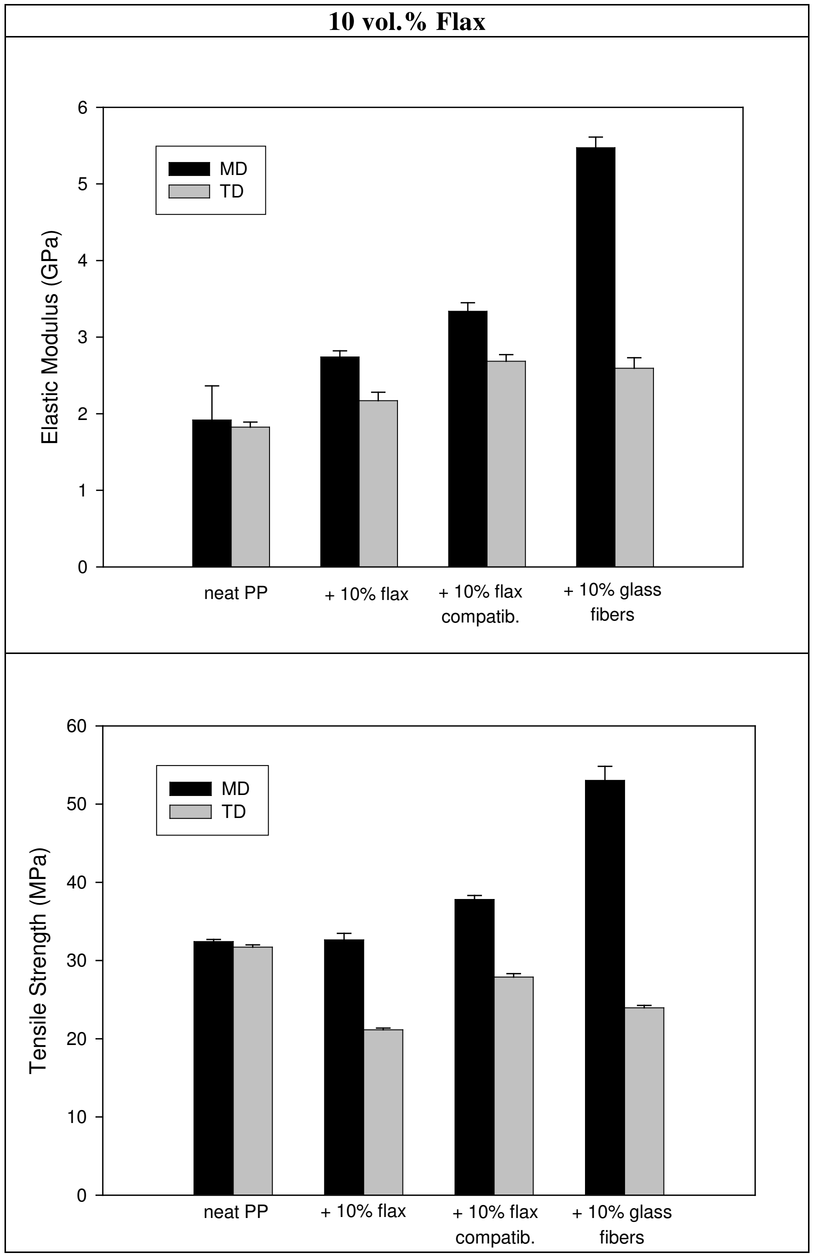

Figure 11. Tensile properties of cast sheets containing 10 vol.% flax and glass fibers. MD: machine direction. TD: transverse direction. a) elastic modulus. b) tensile strength .

Figure 12 shows the results for sheets containing 30 vol.% flax fibers. Again, an increase of the modulus and strength is observed with the addition of fibers. The sheets with flax are also more isotropic than in the case of the glass fibers.

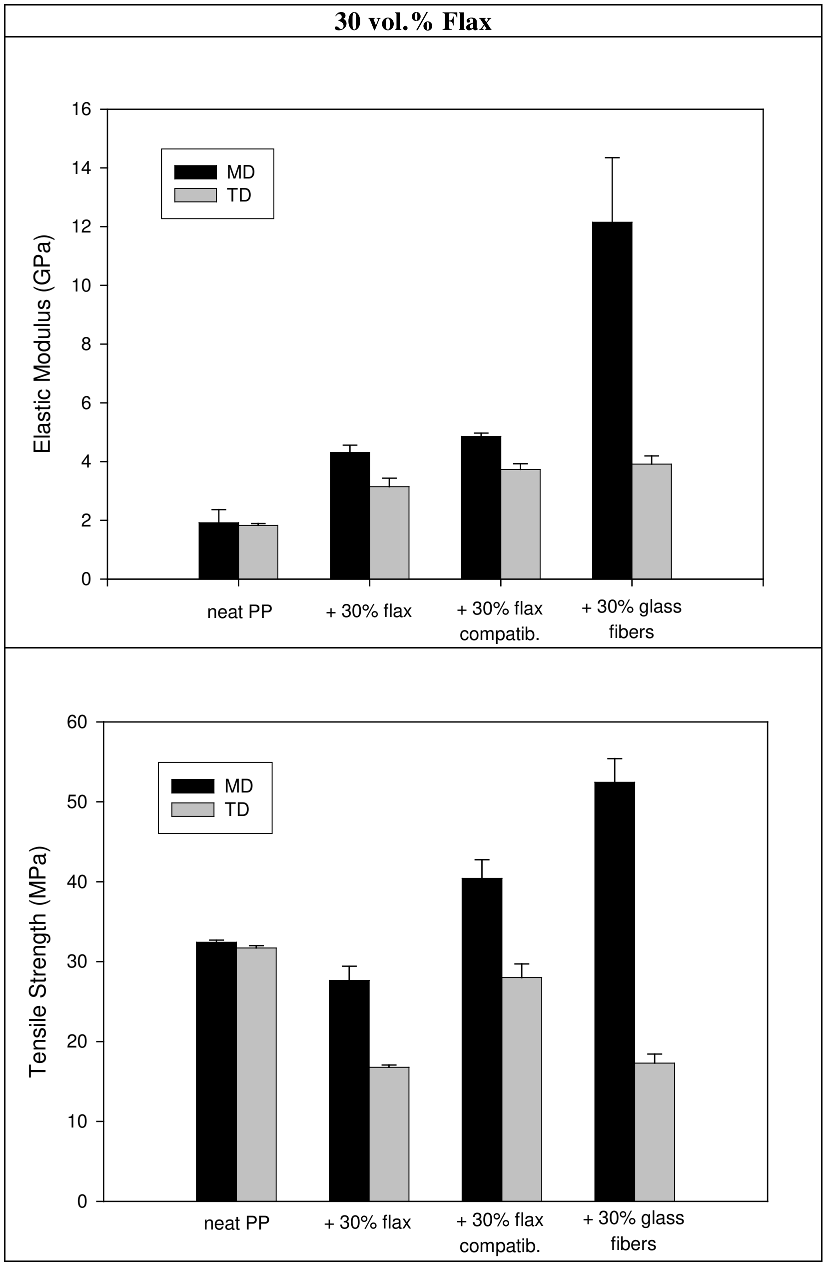

Figure 12. Tensile properties of cast sheets containing 30 vol.% flax and glass fibers. MD: machine direction. TD: transverse direction. a) elastic modulus b) tensile strength.

Results on recycling experiments done on composites made of 30 vol.% flax fibers formulated with E43 and CaO are presented in Figure 13. It can be easily observed that after five injection cycles, the composites did not exhibit any significant change in tensile strength and tensile modulus.

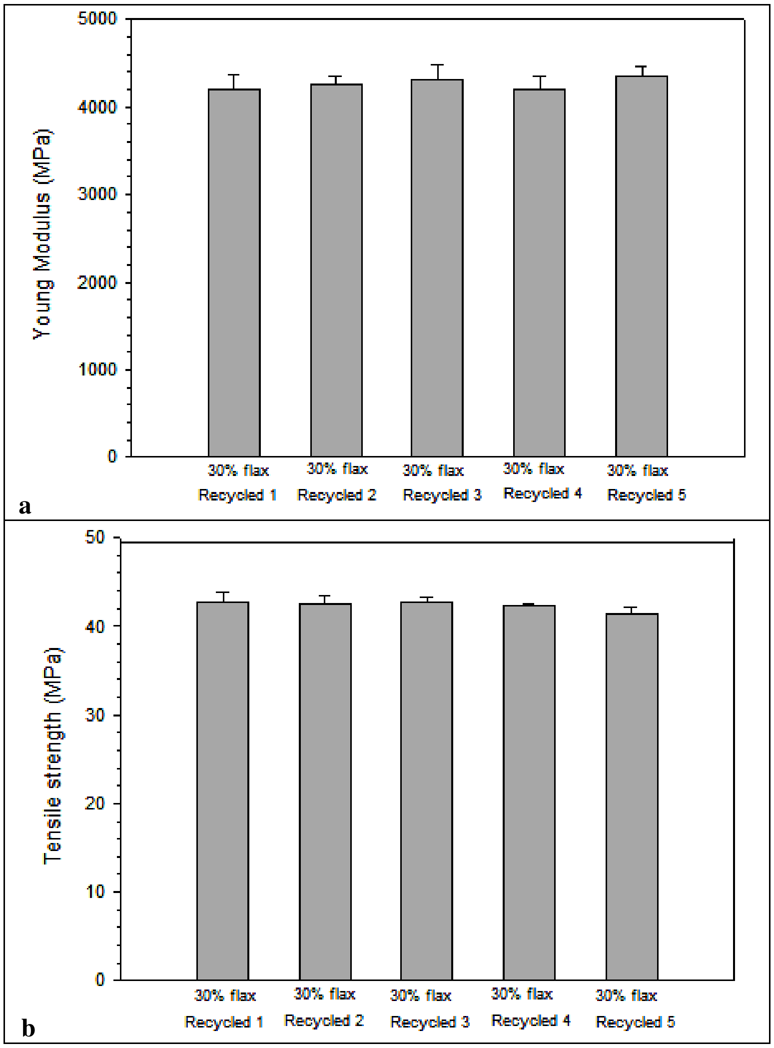

Figure 13. Tensile properties of recycled PP composites with 30 vol.% flax fiber content: a) tensile modulus and b) tensile strength

CONCLUSIONS

Polypropylene/flax and hemp fiber composites were compounded using a twin-screw extruder and specimens were obtained by injection molding. A pre-treatment step on the fibers was found to be necessary to correctly feed them into the extruder, in order to preserve consistent flow rate and, therefore, a consistent fiber concentration in the composite. The pelletizing step of flax fibers was proven to be very beneficial. In addition, during this pelletizing step, the flax technical fibers were split down into elementary fibers without compromising the fiber length. Rheological, thermal and mechanical behaviour improved significantly with fiber content. Tensile strength measured on standard specimens (dogbones) increased up to 40% with the addition of 30 %vol. of flax fibers content when coupling and reactive agents are used. Extruded cast sheets were also manufactured and their mechanical performance evaluated. The values of the elastic modulus for neat polypropylene were similar in both MD and TD. The composites were less isotropic as the values in TD were slightly lower. However, this was even more pronounced for the blends containing glass fibers. The best results (highest tensile modulus and strength) were obtained for the compatibilized blends.

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