

Rheology of Natural Fibers Thermoplastic Compounds: Flow Length and Fiber Distribution

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ABSTRACT: The presented study investigates the flow length and the corresponding fiber content distribution in the injection-moulded natural fiber reinforced thermoplastics and its relation to fiber type and processing parameters such as injection pressure, temperature, injection rate and mould tempering by increasing die temperature. In this research, polypropylene compounds with nominally 30 wt % hemp and sisal fibers are investigated. The influence of the injection pressure (500 and 1000 bar), melt temperature (180°C, 200°C, and 220°C), and die temperature (23°C and 80°C) on the fiber content distribution all over the sample is investigated. An increasing linear trend of fiber content along the spiral length is observed as an evidence of a fiber/polymer multiframe system. A pattern for fiber content distribution with respect to the fiber length along the injected spiral can be distinguished, where the longer fibers are usually found at the end of the injected part and the shorter fibers remain near mould entrance point. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2013**, *000*, 39861.

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INTRODUCTION

In comparison with the synthetic fiber composites; natural fibers are characterized by their attractive price, low density, and lower abrasion. The energy consumption needed for the production of synthetic fibers is much more than that needed for a similar quantity of natural fibers.^{1,2}

Unlike the synthetic fibers, natural fibers have a wide variation in diameter and length which in turn affects the expected mechanical behavior of the composite. The variation in natural fiber dimensions results from a contribution of fiber type, fiber maturity, harvesting time as well as processing methods adopted for the extraction of fibers, which all affect the diameter stability of the fiber. Source, age, separating techniques, moisture content and history of the fiber also play an important role in the filament and individual fiber properties.²⁻⁴

The implementation of natural fibers in thermoplastic composites is attractive for different industrial sectors like automobiles and construction.⁵ However, the incomplete characterization of the rheological behavior of such composites hinders their marketing and further applications. Additionally, the versatility of the fibers' sizes and properties requires a comprehensive study

to investigate the effect of fiber type on the rheological behavior. The most applicable types of natural fibers for industrial purposes in the market are the bast (e.g., flax, hemp, kenaf...) and leaf (e.g., sisal, abaca...) fibers. Therefore, this study focuses on hemp and sisal fibers as case studies.^{6,7}

For the purpose of industrializing the natural fiber composite products; building models, to describe the composite flow pattern during injection, are required. Hence, rheological study of the natural fiber composites is essential to define the viscosity changes because of the variation in the processing parameters. However, if the material does not have a constant fiber content, the viscosity and the flow pattern will consequently, not behave as a single phase flow. In other words, different fiber contents during injection, point to the establishment of a multiframe system that should be considered. This is ensured in natural fiber thermoplastic composites produced by injection moulding. Such a multiframe system can be studied with the help of spiral flow moulds. Spiral injection mould represents a one-dimensional flow for the polymer/fiber composite. By analyzing the fiber content at discrete points along the spiral, the flow behavior of both polymer and fibers can be assessed to determine if they are flowing with the same rate as one unit or following different behaviors.⁸

According to literature, the phase separation occurs between the reinforcing fibers and the polymer melt at the stage of entering the mould.⁶ This is influenced to a great extent by the differences in the densities and the dimensions of the fibers. This phase separation explains the heterogeneity of the fibers distribution along the injected spiral. In their research about 25 wt % glass reinforced polymers, Kubat et al. mentioned that the phase separation which occurs during injection influences the fibers to flow in the middle of the flow channel. This in turn leads to the increase in velocity of the fibers at the middle plane and causes this inhomogeneity phenomenon.^{9–11}

For a better investigation of the fibers, dimensional measurements are essential for determining the factors affecting the flow of the melt inside the mould during the injection moulding process. Reinforcing fibers dimensions can be investigated using several methods.^{12–14} One popular method, because of its high accuracy, is the dynamic image processing. Imaging of fibers is performed using a light source with an exposure time of about 1 ns. An adaptable high-speed camera for optimal illumination and capturing of flowing fibers is integrated. This high-speed camera captures up to 450 images of 1024 x 1024 square pixels in $10 \times 10 \mu\text{m}^2$. Image preprocessor and gigabit digital transmission line is connected to computer. The measuring ranges vary from 1 up to 6,830 μm , which provides high accuracy and reliable results.^{15,16}

It is expected that the change in natural fiber content in injection moulded products will definitely affect the expected mechanical properties.¹⁷ Therefore, this work aims to understand this phenomenon along a one-dimensional injection moulded product (spiral). It is not logical to assume similarity in behavior between the glass fiber composites and natural fiber composites in injection moulding. This is because glass fibers have a consistent geometry and do not suffer thermal degradation or fiber detachment (defibrillation) as natural fibers do.

EXPERIMENTAL

Materials

Used natural fibers in this study are sisal and hemp fibers with densities of 1.50 and 1.48 g/cm^3 , respectively.¹⁸ Two different polypropylene/ natural fiber compounds are investigated. The first compound is PP-S, which is a polypropylene/sisal compound of a nominal fiber content of 30 wt % as given by the producer. The polypropylene is characterized with its high fluidity suitable for injection moulding. Melt flow rate (MFR) is 100 g/10 min at 230°C/2.16 kg conditions. Tensile strength at yield is 26 MPa. The second compound is PP-H with 30 wt % Hemp fibers nominally. Polypropylene is characterized with MFR of 15 g/10 min and yield strength of 20 MPa. The original reinforcing fibers in the compounds are commercially available and geometrically investigated by the dynamic image processing. Fibers are extracted by Decalin and their dimensions found to be 1035 and 778 μm in length and 71 and 55 μm in diameter for sisal and hemp, respectively.

Mould

The mould used for performing the spiral test is an Archimedean Spiral with an outer diameter of 250 mm and step 9.5

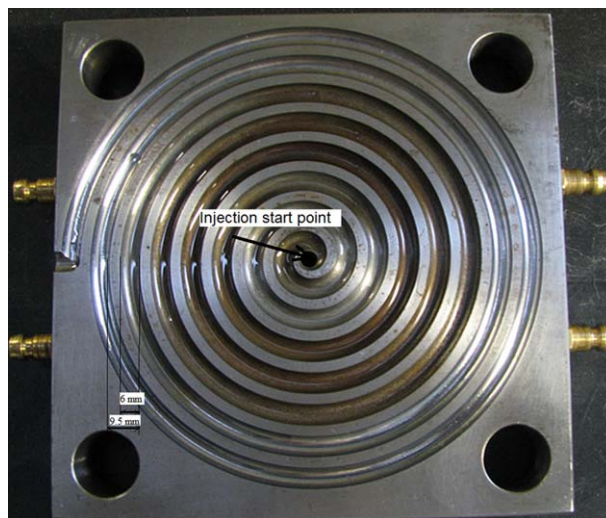


Figure 1. Spiral mould. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

mm as shown in Figure 1. The spiral flow cavity is 6 mm wide and has a curvature of $R = 5$ mm.

Injecting Parameters

The two compounds under investigation are dried overnight at 60°C. Afterward, the compounds are injection moulded using the spiral mould shown in Figure 1 at different melt temperatures, different mould temperatures, and injection pressures indicated in Table I. The injection machine is an Allrounder 320C-600/250, Arburg GmbH, Germany, with a 30 mm diameter screw. Table I shows the injection parameters used in this research.

Ten Samples for each temperature–pressure combination are injected and the corresponding spiral lengths are measured using a special template as shown in Figure 2.

Fiber Extraction

After injection moulding, spirals lengths are first measured and then fibers are extracted from spirals. To be able to investigate the fiber content distribution along the length of the spirals; fibers have to be extracted from the spirals using Decalin (industrial organic solvent of bicyclic structure that dissolves polypropylene). One centimetre long segments are cut from the spiral at a 10 cm step along the spiral length starting with the sprue as the Zero and reference point as shown in Figure 2.

Segments are separately weighed before proceeding with fiber extraction. The extracted fibers are then dried and preserved for further geometrical investigation process. This process is

Table I. Injection Parameters

Parameter	Values
T Melt [°C]	180, 200, 220
Injection Pressure [bar]	500, 1000
T Mould [°C]	23, 80
Injection Rate [$\text{cm}^3 \cdot \text{min}^{-1}$]	20, 50

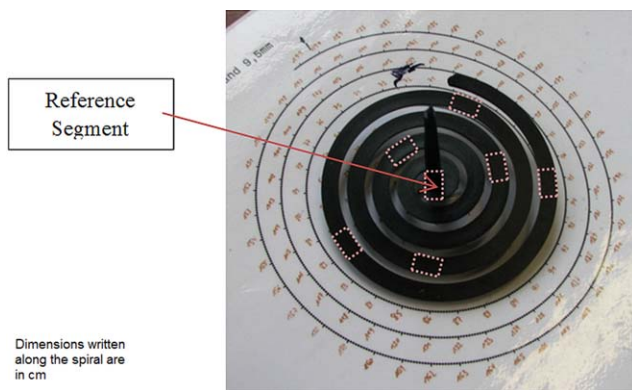


Figure 2. Spiral length measuring template. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

repeated three times for each compound on three different spirals and then the average value of the fiber content with the corresponding standard deviations are calculated.

Fibers Investigation

Fibers are investigated for the length and diameter distribution with an optical imaging method on QICPIC RODOS/L Analyzer- Sympatec GmbH-Germany. In this apparatus, fibers are investigated in the dry state with air pressure dispersion of 4 bar for best results.

Fibers topography is also investigated on a digital microscope Keyence VHX-500F, Japan, with a polarization filter.

RESULTS AND DISCUSSION

Effect of Injection Pressure, Melt Temperature, and Mould Tempering on Flow Length

In this section, the influence of the different injection parameters on the spiral flow length are discussed. Spiral flow length is the length covered by the injected material in the spiral till it stops because of its freezing. For the two compounds PP-S and PP-H, a neat linear trend can be observed by increasing the melt temperatures for 80°C and 23°C room temperature (RT) moulds as shown in Figures 3 and 4.

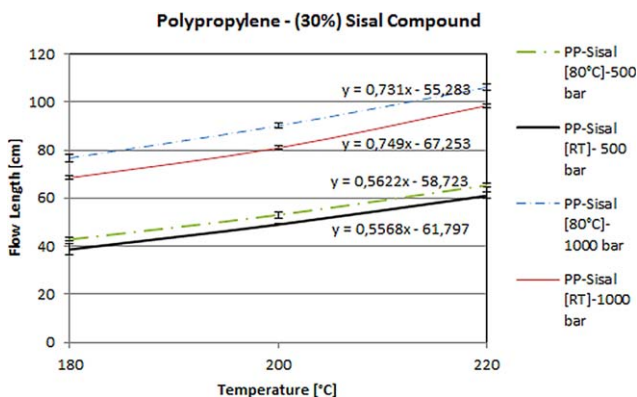


Figure 3. Flow length in relation to temperature, pressure, and mould tempering for PP-S compound. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

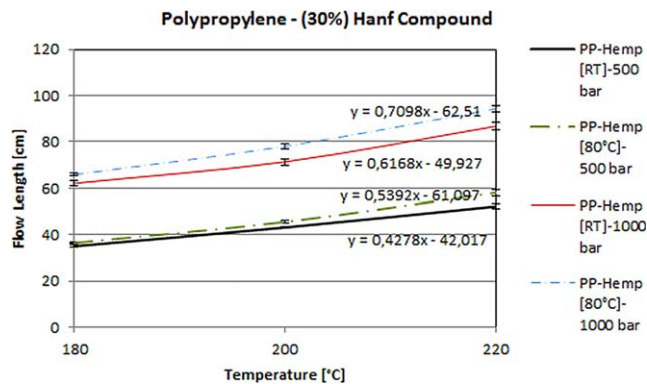


Figure 4. Flow length in relation to temperature, pressure, and mould tempering for PP-H compound. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

In the graph, a steadily increasing trend of the flow length with the increase of the melt temperature can be easily distinguished. Starting with melt temperature of 180°C and 500 bar injection pressure and nontempered mould (mould temperature = RT), the average flow lengths of the PP-Sisal and PP-Hemp compounds are 38.6 and 35.1 cm, respectively, with standard deviations of 2.3 for PP-S and 0.6 for PP-H. Tempering the mould at 80°C influenced the flow of the two compounds inside the mould positively. The average PP-S flow length increased by 4.1 cm and the PP-Hemp increased by 1.4 cm with standard deviations of 0.6 and 0.3, respectively.

Figures 3 and 4 show the empirical formula describing the trend lines of the spiral flow lengths. Raising the injection pressure from 500 to 1000 bar leads to an almost 50% increase in the flow lengths of both compounds. Table II shows the trend line equations in the form of ($Y = aX + b$), where X and Y represent the temperature (°C) and spiral flow length (cm). The trend lines cover the different processing conditions and the different fibers. From the graph, it is also noticed that the best flow behavior for both compounds is obtained at 220°C melt temperature, 1000 bar and 80°C mould temperature. Temperatures higher than 220°C are avoided because of the thermal degradation of natural fibers.¹⁷

Table II. Trend Lines ($Y = aX + b$) of Flow Length Versus Temperature for Different Fiber Types, Mould Temperature and Injection Pressure

Fiber	Mould Temperature [°C]	Injection pressure [bar]	a	b
Sisal	80	1000	0.731	-55.283
Sisal	23 (RT)	1000	0.749	-67.253
Sisal	80	500	0.5622	-58.723
Sisal	23 (RT)	500	0.5568	-61.793
Hemp	80	1000	0.7098	-62.51
Hemp	23 (RT)	1000	0.6168	-49.927
Hemp	80	500	0.5392	-61.097
Hemp	23 (RT)	500	0.4278	-42.017

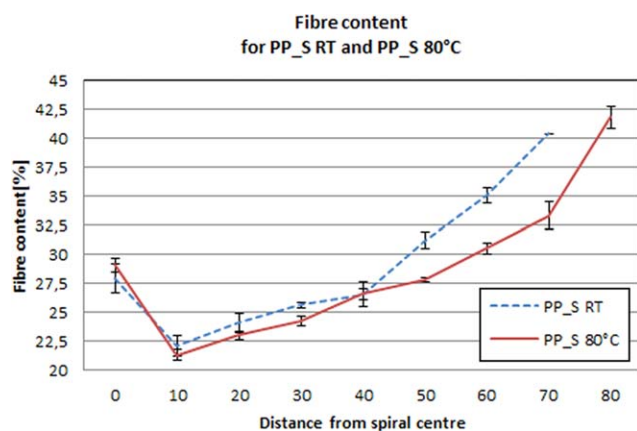


Figure 5. Fiber content over spirals length; PP-S. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

It was also proven that with these parameters, at least double the initial spiral flow length can be achieved. The worst conditions for injection are by the lowest melt temperature 180°C, lowest pressure 500 bar and without mould tempering, while the best conditions are by 220°C melt temperature, 1000 bar injection pressure, and 80°C mould temperature. By comparing the worst with the best conditions in terms of spiral flow lengths, the average flow length of the PP-S at the worst condition increases almost twice with the best conditions. The average increase in flow length of the compounds PP-S and PP-H by setting the worst to the best conditions is 67.2 and 59.1 cm, respectively, with corresponding standard deviations of 1.2 and 1.6.

To check the significance of the results in figures at each point under study along the spiral, *T*-test is applied considering the following parameters (single tail study with a confidence level of 95%, number of spiral samples equals 10). The two trends of sisal and hemp at both die temperatures (RT and 80°C) proved to have significant difference.

Effect of Fiber Type and Mould Tempering on Fiber Content Distribution along the Spiral

The actual fiber content of the original granules before injection was investigated by the same method mentioned in section (Fiber Extraction). For the compound PP-S, the actual fiber content was 26.7% with a standard deviation of 0.5, which is around 3% less than the given nominal value by the producer. The PP-H compound showed conversely more reliable fiber content of 31.3% with a standard deviation of 0.8.

Figures 5 and 6 show the fiber content development along the spiral length. *T*-test proves the significance of die tempering after the middle point—40 cm position—in case of sisal, Figure 5, by considering number of samples equals 3 and the level of confidence is 95%. The *T*-test for hemp composite, Figure 6, proved its significance except at the middle point where close values are observed. In Figures 5 and 6, the first measurement point corresponds to the gate (reference segment, Figure 2) just before flowing into the spiral mould. However, directly after the gate the fibers undergo turbulent flow conditions which might lead to the formation of fiber clusters at this specific point. In

comparison with single fibers (non-clustered), the fiber clusters suffer less frictional resistance force to the motion along the injection path. This difference in the frictional forces is attributed to the form of the fibers where accumulated cluster of fibers (agglomerate) have lower polymer interfacial areas and hence the resistance to flow is decreased in comparison to the single fibers where their areas are totally contacting with the host polymer. Consequently, according to the principal of energy conservation, the agglomerate clusters of fibers can flow longer distances. This explains why the fiber content at the end of the spiral length is higher than that at the beginning; i.e. 10–40 cm from the zero/reference point.

The intensity of this phenomenon is affected by the fiber type as well. A different behavior between the hemp compound and the sisal compound can be easily noticed. This is attributed to the fiber shape characteristics for each as will be later discussed in this article. When comparing the fiber wt % of the sisal compound at point 0 and at the end of the spiral, an increase of 140% of the fiber content can be proved for the 80°C mould. On the other hand, the hemp compound proved an increase of 130%. The concentration pattern of the fiber wt % of the two compounds is identical, where the fiber wt % content at point 0 (center of the spiral) shows a value higher than that after 10 cm.

By comparing the two Figures (5 and 6), a very interesting point can be observed, which are the midpoints of the spirals. It was observed that the weight content at this specific point (distance in relation to the length of spiral), always corresponds to the average wt % of the original compound granulates. Moreover, the fiber wt % is proved to be identical in value for both 80°C and RT moulds for the two compounds at exactly the same point. Here, it is important to mention that for PP-H the wt % (30.5%) at the intersecting point (32.7 cm) was interpolated from the graph. The reason for that is the analysis that took place at specific distanced segments as mentioned before, where the required point was not included. This matches the findings of Kubat et al.⁹ where the same phenomena is reported when injecting glass fibers and micro-particles in a polymer matrix in a spiral mould. Same pattern of fiber content increase

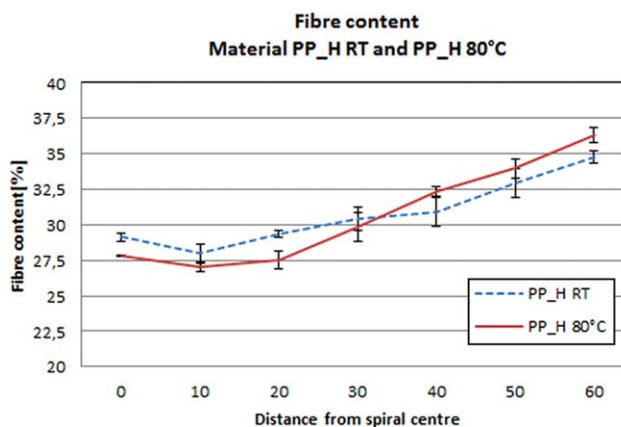


Figure 6. Fiber content over spirals length; PP-H. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

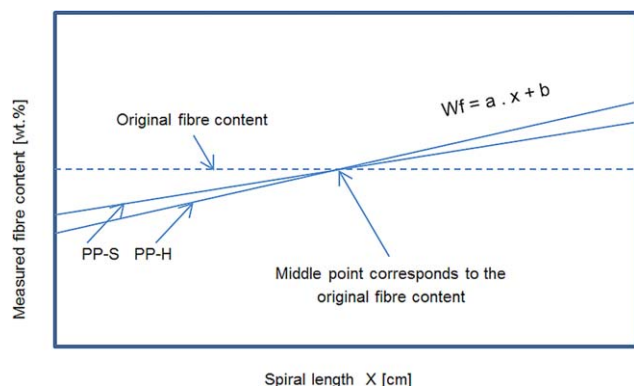


Figure 7. Relation between original and actual fiber content along spiral length. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

over the length in the direction of injection was also reported in other researches but in injected plates.^{10,19}

On the other hand, Danes et al. proved the same observation as well when injection moulded filled polymer into plates.²⁰ A reduction of the wt % at the prefatory field of the plates of 0.9 of the original wt % was reported. Passing this field nearing the middle of the plate, the fiber concentration continues to increase passing by the original wt % around the middle point of the plate till reaching 1.3 of the original wt % at the end.

This can be attributed to the following explanation; the average fiber content out of the different measurement points along the spiral corresponds to the original fiber content (original granulates) and the relative velocity between the flowing fibers and the host matrix results in a linear trend of fiber content deviation with respect to the spiral length (regardless the input point at the zero position). And hence, the deviation in the measured fiber contents balances around the middle point as shown in Figure 7.

Fiber Shape after Extraction

Table III illustrates the extracted fibers shape after injection. The sizes of the sisal fibers are larger than the hemp fibers. This affects presumably the fiber content distribution along the spirals. The fiber content of sisal ranges are [21.3–41.8%] and [22.1–40.4%] for 80°C and RT moulds, respectively. For hemp fibers, the ranges of fiber content are [27.0–34.8%] and [28.0–36.3%] for the same mould temperatures.

By subtraction, the deviations for sisal are 20.5% and 18.3% and for hemp are 7.8% and 8.3% for the both 80°C and RT mould conditions, respectively. In other words, the deviation in the case of sisal is greater than that in the case of hemp.

Recalling the relative speed between the fibers and the host matrix theory, the greater sized sisal fibers will acquire higher momentum and hence higher deviation in fiber content along the spiral length as presented in Figures 5 and 6. Additionally, at certain fiber content the fibers with larger diameter will have lower ratio of contact surface to the volume of fibers and hence will suffer less resistance during flow. This result is in

Table III. Structure of Extracted Fibers after Injection Moulding

	Zero point	End point
Hemp @ RT		
Sisal @ RT		

accordance with the observation of Kubat⁹ who reported that greater glass particles have greater deviation with respect to the original content.⁷ As shown in Table III, the change in the length of sisal fibers along the spiral length is more significant than that of hemp. This is attributed to the higher strain to failure (low flexural stiffness) of hemp fibers.²¹

Hemp fibers, like other bast fibers, exhibit low flexural stiffness on contrary to the sisal fibers, which are easily broken.

Quantitative study is carried out using the QICPIC apparatus and the frequency histograms of lengths and diameters (L&D) are calculated. Hence, the accumulated frequencies are calculated and the main values at 10%, 50% (median), and 90% are defined. See Figures 8 and 9 for the results of the extracted fibers length/diameter at end tips of the two types of spirals. As reported previously, Weibull distribution fits well with the strength of the flax single fibers.²² In this study as well, Weibull distribution seems to be the best fit of the fiber length as well as the fiber diameter.

For illustration, Figures 10 and 11 show the change of fiber length and diameter along the spiral length where only the median values (50%) are presented. As shown in Figure 10, length of hemp fiber sticks almost to 400 μm . This behavior does not change when the mould is at 80°C. This may be explained in terms of the energy needed to detach the hemp fibers, which requires most likely higher temperatures.

For the hemp fiber diameter, Figure 11, the fibers' diameters are not constant along the spiral length. The median of the fiber diameter increases from 15 μm at the spiral beginning to 25 μm at the middle of the spiral. By comparing the extracted original hemp fibers and the extracted hemp fibers after injection, it is obvious that there is a great reduction in diameter from 55 to 15–25 μm . That is not the case where sisal diameter is only reduced from 71 to 45–62 μm . This is attributed to the difference in fineness between hemp and sisal. Hemp proves fineness between 0.3 and 3 tex for single fibers, whereas sisal single fibers values lie in the range 1–4.6 tex.^{4,22} Also it is noticed from Figure 11 that hemp fibers suffer more fibrillation at the spiral beginning, hence higher aspect ratios (fiber length divided by fiber diameter) are found at the spiral start. Otherwise, fibers

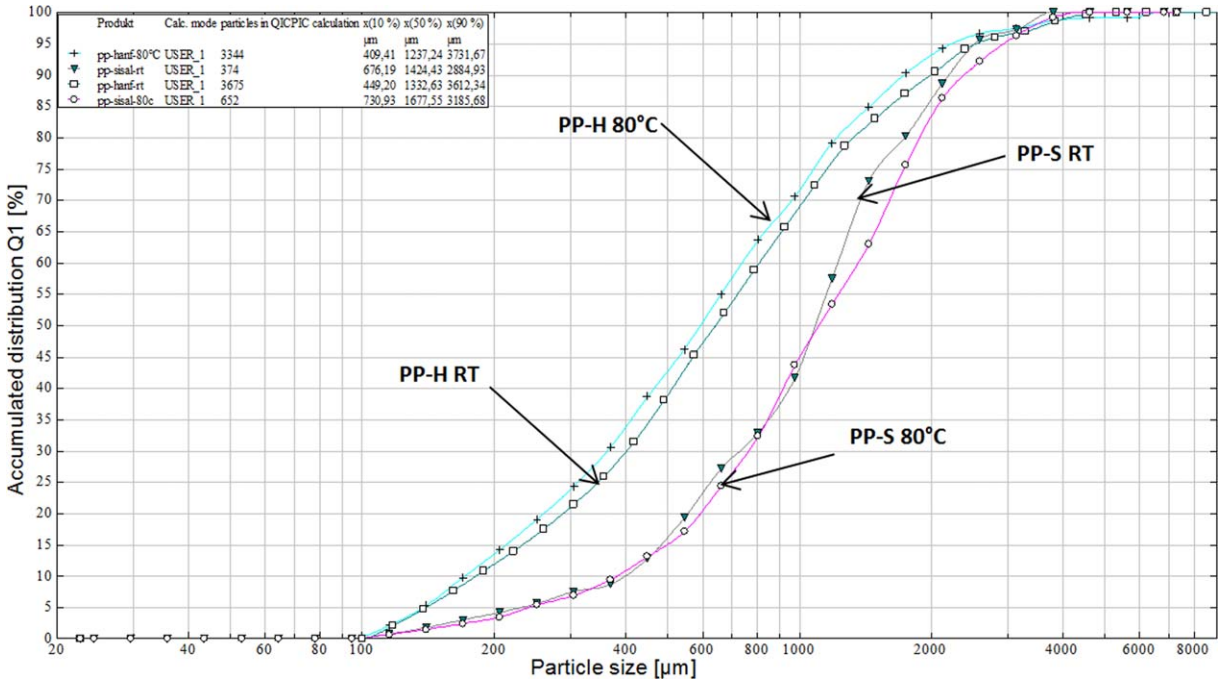


Figure 8. Accumulated frequencies of length distribution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

have relatively greater diameter and hence lower aspect ratio. Aspect ratio is believed to affect positively on the composite strength.^{5,23}

Sisal fibers on the contrary, show sensitivity to mould temperature. For instance, the room temperature mould shows an almost similar behavior like hemp fibers. Figure 11 presents

smaller diameter and shorter length for sisal fibers at the spiral beginning. But at 80°C mould temperature, the short thin fibers seem to have more flow-ability to settle in the spiral middle region as seen in graphs. Similar findings were also reported by in other researches.^{24–26} The diameter of sisal fibers at 80°C mould tempering is larger than that without tempering. This

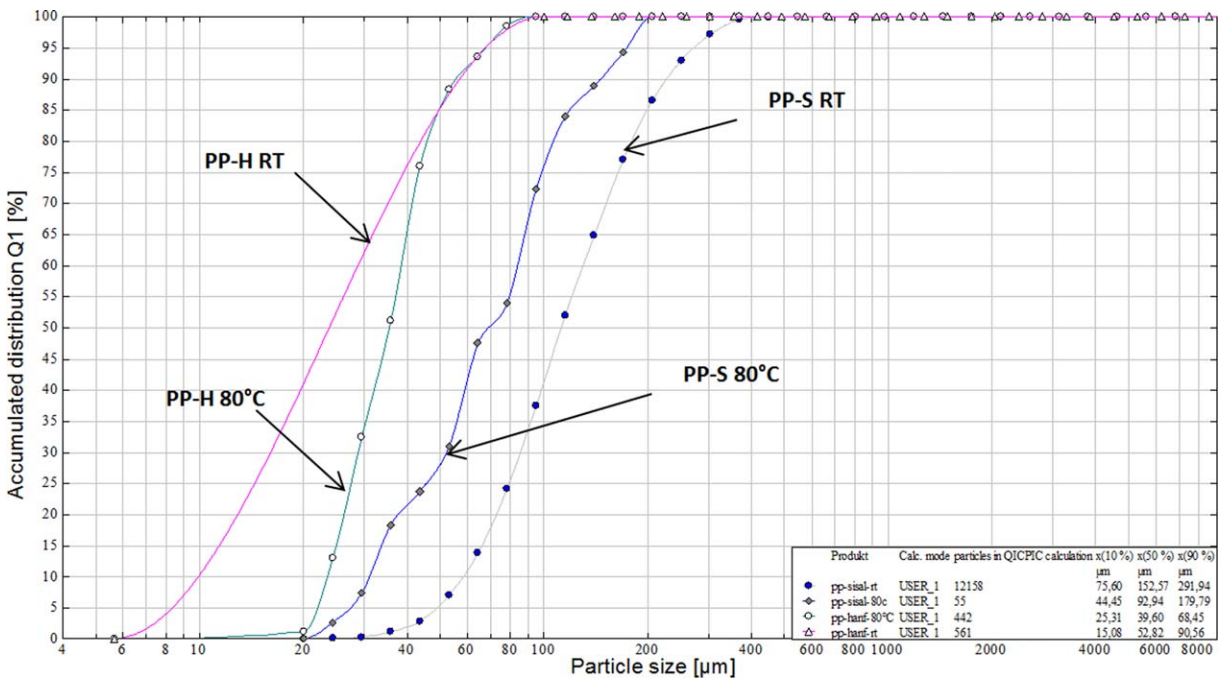


Figure 9. Accumulated frequencies of diameter distribution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

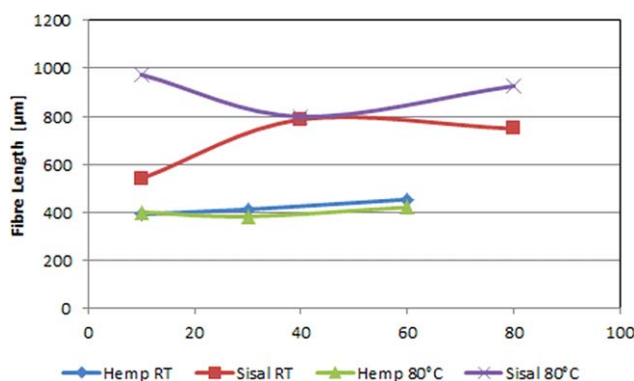


Figure 10. Change of the extracted fiber length – median value \times 50% – versus length over spiral. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

can be attributed to the detachment of sisal fiber cluster at higher temperatures. Whereas at room temperature; defibrillation is more observable than fibers detachment because of the friction between sisal fibers.

Effect of Injection Rate at Different Injection Pressures

For a better understanding of the behavior of the flow in the spiral with the variation of pressure and temperature, a flow length ratio is calculated for both compounds at 180°C, 200°C, and 220°C melt temperatures. Both materials were injected into a spiral mould heated to 80°C spiral mould with an injection rate of 20 cm³ min⁻¹. Perfect linear relation can be distinguished for both materials that can be linearly fitted with a very accepted coefficient of determination as shown in Figure 12.

Figure 12 shows that at a melt temperature of 180°C and 1000 bar injection pressure, the flow length is 180% longer than at 500 bar pressure at the same melt temperature. This great influence of pressure decreases with higher melt temperature where a melt temperature of 200°C and 220°C at 1000 bar proves a longer flow length by around 170% and 160%, respectively, as at 500 bar. In other words, the change in flow length with respect to pressure ($\delta l/\delta p$) is affected negatively with the increase of melt temperature.

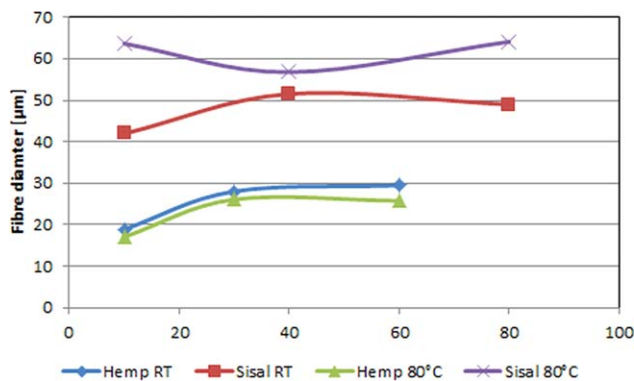


Figure 11. Change of the extracted fiber diameter – median value \times 50% – versus length over spiral. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

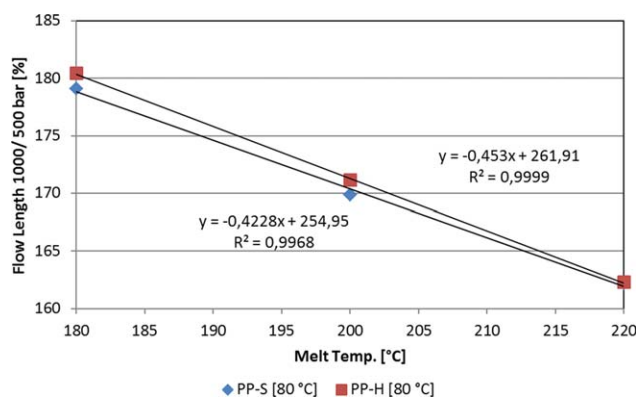


Figure 12. Effect of melt temperature on the change in flow length at different pressures for PP-S and PP-H compounds using low injection rate of 20 cm³ min⁻¹. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

It is noticed that by this low injection rate of 20 cm³ min⁻¹, the results of the two compounds are homogeneous and nearly the same. This can be attributed to the required time given to the fibers when flowing inside the spiral to orient without acting as an obstacle against the flow of the matrix.²¹ Hence, the improvement of spiral flow length by increasing the applied injection pressure is not significantly dependant on fiber type.

In Figure 13, it is noticed that increasing the injection flow rate from 20 to 50 cm³ min⁻¹ has a negative effect on the homogeneity of the results especially for the sisal fibers. This can be easily recognized from the fitting line which fits with all points of hemp compound, but not the sisal. Where the coefficient of determination for hemp and sisal compounds are $R^2 = 0.9939$ and 0.8225, respectively. This effect of injection speed on the homogeneity of the injected part was also investigated in other research, but a different change in the homogeneity was observed.¹⁹ This might be subjected to testing parameters where only two very low speeds were experimented 8/16 cm³ min⁻¹ in which both lie below the speeds used in this research. Also, it is obvious that the melt temperature has the same negative effect on ($\delta l/\delta p$), but not as strong as in case of 20 cm³ min⁻¹.

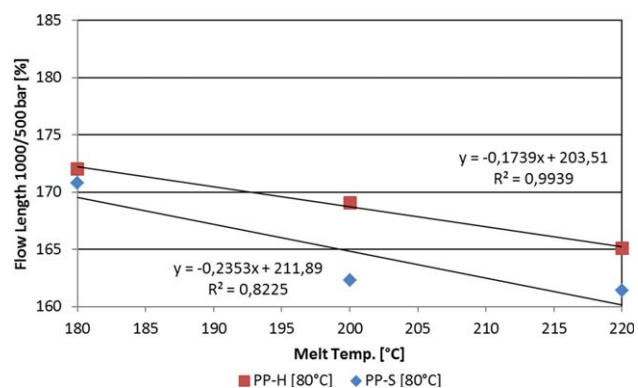


Figure 13. Effect of melt temperature on the change in flow length at different pressures for PP-S and PP-H compounds using high injection rate of 50 cm³ min⁻¹. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSIONS

The presented study discusses the effect of the natural fiber type and processing parameters during injection moulding on the flowability of the material as well as the homogeneity of fiber content, which in turn affects the physical and the mechanical properties of the composite. The following points are concluded:

1. The flow length of the injected spirals: Spiral flow length increased with the increase of the compound melt temperature, mould tempering temperature, and the injection pressure. On the other hand, the significance of the change in flow length in relation to change in pressure ($\delta l/\delta p$), proved to be lessened by increasing the melt temperatures.
2. The homogeneity of fiber content along the injection path: fiber content increases along the spiral flow length. This is explained in terms of:
 - a. Frictional resistance to flow of fibrillated fibers more than that suffered by agglomerates.
 - b. Hemp fibers—which have a high tendency to agglomeration (longer length and thinner diameter)—are always found at the end of the spiral length, on the contrary, sisal fibers of higher flexural strength and low strain to failure do not show such behavior.
 - c. Low injection rate of 20 cm³min⁻¹ allows the fibers to be homogeneously distributed and a linear relation between melt temperature and the improvement in spiral flow length because of pressure rise (1000 bar/500 bar) is attained. Conversely, high injection rate of 50 cm³min⁻¹ affects the fiber content distribution negatively especially with stiff fibers like sisal.

It is expected in the near future to develop this work by generalizing the spiral test results (one dimension) on real multi-dimension injection moulded products. Then it is also required to consider the fiber orientation variation and their effect on the mechanical properties. Not only this, but also the outcomes of this work can give a push for modelling such new composites.

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