Processing of Wet Preserved Natural Fibers with Injection Molding Compounding (IMC)

Hans-Jörg Gusovius, Gesine Wallot, Stefan Schierl, Roman Rinberg, Tobias Hartmann, Lothar Kroll and Ivonne Jahn

Abstract The paper shows selected results of a comprehensive national network activity related to the integrated value chain for new whole plant based fibrous raw materials. Therefore, both the supply of raw materials and the processing with the innovative one step technology injection molding compounding (IMC)—are optimized. As a result the fiber length in the product is longer compared to the standard two-step process with pelletizing. The new technology helps in improving the manufacturing of fiber reinforced composites by considering the integrated value chain.

Keywords Plant fibers · Injection molding compounding · One-step technology · Mechanical properties

List of Abbreviations

°C	Temperature in grade centigrade
ATB	Leibniz-Institute for Agricultural Engineering Potsdam-Bornim
BD/SM	Butadiene to styrene-monomer ratio
cf.	Compare
D	Diameter
DM	Dry matter content

H.-J. Gusovius (✉) · G. Wallot
Department of Post Harvesting Technologies, Leibniz-Institute for Agricultural Engineering Potsdam-Bornim, Potsdam, Germany
e-mail: hjgusovius@atb-potsdam.de

S. Schierl
KraussMaffei Technologies GmbH, Munich, Germany

R. Rinberg · T. Hartmann · L. Kroll
Faculty of Mechanical Engineering, Department of Lightweight Structures and Polymer Technology, Technical University Chemnitz, Chemnitz, Germany

I. Jahn
Fraunhofer Institute for Mechanics of Materials IWM, Halle, Germany

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FENAFA	Ganzheitliche Bereitstellungs-, Verarbeitungs- und FErtigungsstrategien von NAturFAserrohstoffen
Fig.	Figure
IMC	Injection molding compounder
IWM	Fraunhofer Institute for Mechanics of Materials IWM
KM	KraussMaffei
L/D	Length diameter ratio
μm	Micrometer
mm	Millimeter
rpm	Revolution in rounds per minute
R&D	Research and development
SEBS	Styrene-ethylene/butylene-styrene block copolymer
temp.	Temperature
TPE	Thermoplastic elastomer
WPC	Wood polymer composite
wt%	Weight percent

Introduction

Growing, processing and utilization of agricultural based fiber resources has a long tradition both in textile and industrial applications (Mohanty et al. 2005; Hänninen and Hughes 2010; Shahzad 2012; Faruk et al. 2012). Recently an increasing amount of such raw materials is produced and specifically used in industrial applications within the European Union (Carus et al. 2014). Even though only 6 % (15,000 t) respectively 2 % (2000 t) of forest or agricultural based raw materials are processed by injection molding in the plastics industry. There is a growing interest for natural fiber reinforced materials in several fields such as construction and automotive. Thus, several bottlenecks need to be solved for the utilization of natural fibers by such technologies.

For more than 5 years 15 industrial companies and scientific institutions have been working in the natural fiber network FENAFA under the leadership of Chemnitz University of Technology. The network focused on new supply chain strategies and processing methods for domestic fiber plants. This more efficient and less risky use of fibers should attract more farmers to increase the cultivation of these plants. Starting point of this cooperation network have been raw material supply procedures with significantly reduced efforts and costs. FENAFA focused its work on three primary fields: First, in the agricultural technology sector, field procedures were to be improved in order to provide competitive raw materials at reduced price and in high quality. A second area focused on the development of processes to manufacture natural fiber based components reinforced with nonwoven semi-finished products. The third core activity has been the utilization of

"TechnoFlax", wet preserved hemp, and wood fibers in injection molding processes. Along the entire processing chain, mass market products such as portable cases, toolboxes and biodegradable toys for children have been produced with the developed compounds reinforced by regionally grown fibers.

Novel Supply Chain for Fibrous Raw Materials

The conventional production of natural fibers from materials such as hemp or flax is based on field drying and retting of straw. Under the condition of usual harvest time, weather conditions are often problematical for the harvest of dry straw (Gusovius et al. 2000; Hoffmann et al. 2013; Liu et al. 2015).

A weather-independent post harvest technique is under investigation at the ATB (Idler et al. 2011). The harvest of fiber crop (e.g. hemp) by means of a chopper followed by an immediate anaerobic storage is favourable for the farmer because available machines can be operated. Additional steps are the same as for ensiling of fodder. Thus, a raw material on a homogeneous quality level is available for further processing and utilization throughout the following period until the next growing season and harvest.

Additionally, one of a few advantages of this novel technology is the processing of the whole plant material to final products such as fibers for plastic reinforcement or fiber boards on an economic viable level (Gusovius et al. 2010; Wallot et al. 2012). A pilot scale plant with a raw material input of up to 1 t h^{-1} (300 kg h^{-1} DM) was established at the ATB in order to investigate the processing and further utilization of the resulting fiber material in several industrial applications (Wallot et al. 2012). In recent years the main focus of R&D at ATB has been the specification of the storage process and the processing steps for the manufacturing of fiber boards (Hoffmann et al. 2009; Pecenka et al. 2009).

In contrast to that significant adoptions of harvest, preservation and fiber generation have been actual key aspects in order to serve products useful for plastics reinforcement. Prior studies have already shown that the reinforcement potential of natural fibers is highly dependent on the fiber fineness as well the aspect-ratio of fiber elements (Ruth et al. 2002; Stark et al. 2003; Specht 2007; Schirp and Stender 2010).

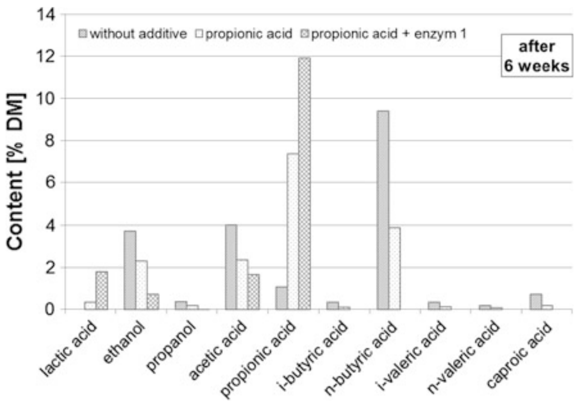
Specific investigations were carried out within the 5 year duration of the FENEFA network in order to improve the quality of the raw material beginning with an optimized preservation. Identical varieties (Fedora 17 and Santhica 27) were grown in each of the growing seasons at the experimental field of ATB in Potsdam-Bornim. Dependent on plant maturity and weather the yearly harvests have been realized by means of a forage chopper at particle size of approx. 10–20 mm between end of August and end of September. Subsequently the resulting plant material was properly densified and stored under anaerobic conditions in preserving bins or model silos (Fig. 1).

Fig. 1 Anaerobic storage in model silo bins (*left*) and chopped plant material (*right*)

Regularly, small sub-samples were taken from the stock for the biochemical analysis of preservation progress and resulting metabolism products of microorganisms. There is a substantial importance of such investigations because the desired stabilization of the preservation is a result of microbial activities and the conversion of carbohydrates into lactic acid. Furthermore the content of this and other organic acids as well as alcohols is influencing the organoleptic properties of the plant material. In particular the main focus of these experiments has been to determine the effect of different silage additives in order to reduce the content of odour relevant substances.

The results show that the addition of silage aid agents has a considerable influence on the content of odour relevant metabolism products in the raw material (Figs. 2, 3). Thus, the effect of propionic acid and as well in combination with a pectinase enzyme (Natuzym) results in a reduction of the content of both alcohols and the majority of organic acids. Organoleptic observations during the removal from the stock confirmed these measurements as the mentioned samples gave the most pleasant impressions.

Fig. 2 Content of metabolism products in wet preserved hemp after 6 weeks within the storage period 2012–2013



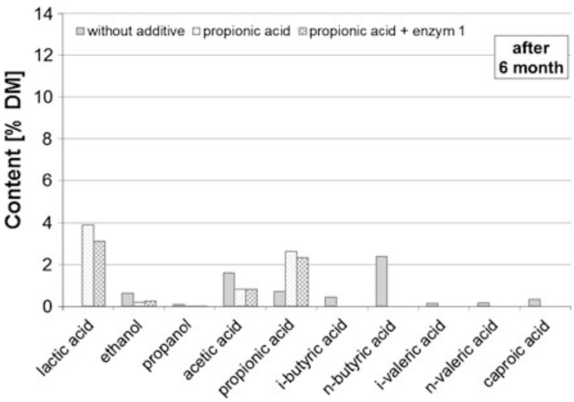
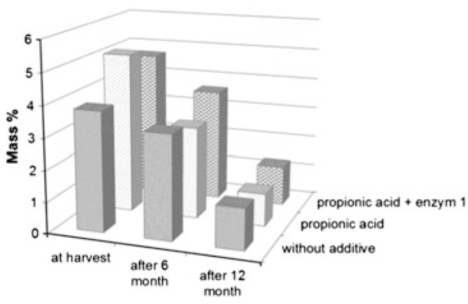


Fig. 3 Content of metabolism products in wet preserved hemp after 6 month within the storage period 2012–2013

Further analysis of the preserved plant materials were carried out to assess the stock composition and the change of the pectin content particularly. From previous investigations it is known that the microbiological activities in the course of wet preservation are comparable to those known as retting. The soluble part of the fiber gluing middle lamella is degraded by enzyme activities originating from anaerobic bacteria's (Fig. 4, right).

The content of hemicelluloses as an indicator of pectin's is noticeable reduced by up to 80 % independent of the type and variation of wet preservation (Fig. 4, left).

It can be summarized that the procedure of wet preservation is simple and low cost possibility to supply fiber crop raw materials for further processing and utilization. The use of silage aid additives can support the improvement of the odor characteristics but seems to be insignificant for a suitable reduction of fiber gluing substances. The latter is an important precondition in order to enable a purposeful fiber processing into an intermediate applicable for plastic reinforcement (Fig. 5).



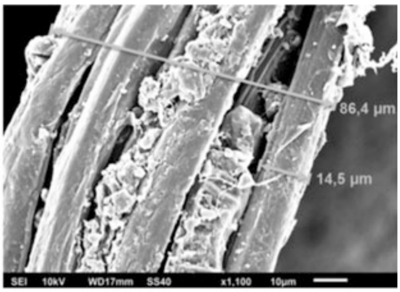


Fig. 4 Content of hemicelluloses in wet preserved hemp within storage period 2011–2012 (*left*) and REM-view of a fiber bundle after 12 month wet preservation (*right*)

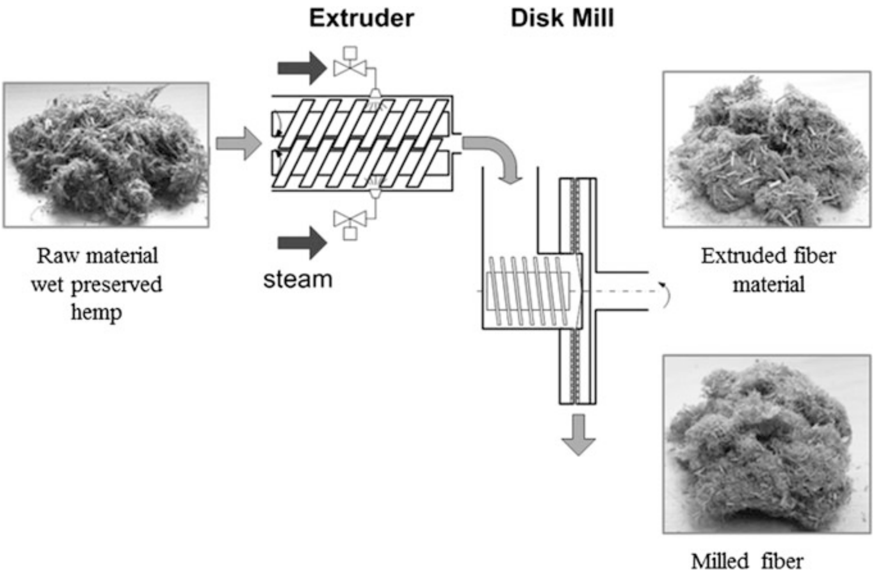


Fig. 5 Process sketch of fiber processing as part of ATB pilot plant

Raw material processing is carried out by means of a defibration extruder and a disc mill (Fig. 5). At both stages the resulting fiber quality can be influenced by the moisture content of the feedstock as well as operating parameters of both of the devices.

The processing of raw material with a lower dry matter content at the extruder results in preferred geometric fiber characteristics. A larger outlet gap of the extruder results in a similar effect (Table 1).

Further experiments were focused on the reduction of under and over sized particles in particular dust as well as remaining shive pieces. The former can already be influenced by a modification of the harvest process. Separate cutting and handling of the plant tops (flowers, inflorescence, seeds, leaves) and exclusive

Table 1 Particle size characteristics according to selected operating parameters of the extruder and dry matter content of wet preserved hemp

Test series		Mesh analysis	Image analysis by "FibreShape" (length weighted)		
Revolution/outlet gap (rpm/mm)	Dry matter content (%)	x_{50}-value (µm)	Aspect-ratio (−)	x_{50}-length (µm)	x_{50}-width (µm)
40/16	30	888	9.6	559	72
40/16	47	765	8.8	457	65
40/23	30	1055	10.6	702	80
40/23	47	761	9.5	530	70

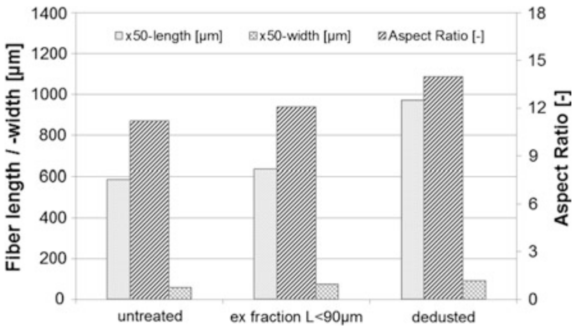


Fig. 6 Length weighted parameters of particle size distribution of a fiber material originated from processing of wet preserved hemp and different sizing treatments

chopping and storage of the remaining stem can result in a reduced dust content in the fiber fraction after processing. Within the described processing experiments simple sieves and a sieve drum separator (condenser) were used to improve particle size parameters of the intermediate (Fig. 6).

The resulting fiber material is characterized by an improved particle size distribution particularly regarding the fiber length as well as aspect-ratio which increased from 11 to about 14.

All experiments on harvest, storage and processing were always scheduled and carried out in order to supply intermediate fibrous materials to the cooperating partners in the network. Samples of different quantities were used to investigate if favorable fiber characteristics resulted in a good processability and composite properties as well.

Processing with IMC

The confection of customised solutions for raw materials with special combinations of their properties demands technological innovations with a high cost effectiveness. Therefore, the process of injection molding compounding (IMC) was investigated within the FENAFA network to enable a simplified processing of native fibrous raw materials into composites. The main goal was the realization of a consistent process chain from the raw material to the final product by a substantial improvement of material and energy efficiency with single step processing in comparison to conventional procedures. The IMC direct compounding provides specific advantages for the processing of heat-sensitive materials like natural fibers. The only singular thermal and mechanical treatment leads to better composite characteristics compared to the traditional two-step-processing. The preceding pelletizing is not applied and higher fiber length can be realized in the final product (Widmayer 2012).

Main advantages of the injection molding compounding process are the obvious reduction of the costs of the component manufacturing and the flexibility of the material combination. The continuous mode of an extruder is combined with the discontinuous function of an injection molding machine in order to produce parts consisting of different raw materials in one process step. The one time heating of the material leads to a tapering of the energy consumption as well as to a lower thermic strain of temperature sensitive materials like composites reinforced with natural fibers. Furthermore, there is no need for granulation and drying of granulate before further processing.

The Injection Molding Compounder (IMC) consists of a fusing and homogenization unit, a twin-screw extruder and an injection system (KraussMaffei 2013). These parts are connected by a diverter pipe. A material storage used as buffer is, in Y-setting, connected between. The material supply is made gravimetrical with an underfeed extruder to get a material throughput nondependent from the rotational speed.

Granulate, powder and additives are added over dosing units. Short-, intermediate-, and endless fibers are added downstream into the extruder. In the closely intermeshing, co-rotating twin-screw extruder the material (polymer, matrix, additive, fiber) is homogenized and conveyed isothermal over the diverter pipe in the injection unit, respectively during the injection process into the material storage. For the start-up there is a diverter valve between extruder and material storage, which is open to derive the melt into a container until the melt quality is in tolerance. Therefore, in normal operation the valve is closed. The material storage buffers the melt supplied from the twin-screw extruder during the injection process and holding pressure phase. This is necessary, to combine the continuous extrusion process with the discontinuous injection molding process. Additionally, the melt buffer applies an adjustable pressure on the melt to ensure a constant part quality. The composite is dosed dependent on throughput into the injection piston. Due to the dosing process the material storage is completely emptied. Configurable dynamic pressure works against the retreat of the injection piston. When the adjustable shot volume for the next injection cycle is reached the diverter valve is closed. This causes a separating of plasticize and injection unit while the injection and holding pressure phase. The shut-off nozzle valve is opened and the injection process starts. After completion of the holding pressure phase the cooling period starts followed by the mold opening time, the remove time and the closing time. Simultaneously the dosing phase starts by closing shut off nozzle valve and open the transfer valve. As soon as the expected threshold value is met the cycle starts again.

Initially different compositions of materials and methods were developed and proofed in a two-step process (1. Compounding, 2. Injection Molding) within the network activities of FENAFA. In order to scale-up and verify the practical feasibility these material systems were processed on an Injection Molding Compounder of KraussMaffei (KM 1300-14000 IMC) available in the Pilot Plant of the Fraunhofer Institute for Mechanics of Materials (IWM) (Fig. 7).

Fig. 7 Injection molding compounder at Fraunhofer Pilot Plant Center Schkopau (Michel 2015)

For the scale-up of the process a demonstrator part from the logistics sector (transport and storage box) was used. The trials are carried out without preceding pelletizing the flax and hemp based compounds.

The production process consists of the steps compounding, injection molding, automatic removal of the molded part from the tool and the drop off on a conveyor with a robot.

In the preliminary stage, the screw geometry of the compounding extruder (D = 84 mm, L/D = 37), the mold, the dosing device and process parameters (e.g. temperature of the cylinder, rotational speed of the screw, mass throughput) were adapted to the configuration of the KM 1300-14000 IMC due to knowledge found ex ante. The mixing areas consist of kneading and tooth—elements to get a homogenous fiber filled melt.

An innovative gravimetrical universal dosing unit developed during this project by the TU Chemnitz was used for the dosing and feed of hemp fibers, which have very poor flow properties.

Processing, Sampling and Characterization of Products

The demonstration of feasibility of the R&D results was performed by producing transport and storage boxes (shot weight approximately 1100 g) with the industrial sized injection molding compounder KM 1300-14000 IMC (Fig. 10).

All compounds consist of matrix polymer polypropylene (BE170MO from Borealis) with 30 wt% of hemp or flax and 3 wt% of a maleated polypropylene as coupling agent (Scona TPPP8112 GA from BYK Kometra). In Fig. 8, the coloring of the first trails is shown. In addition to a strong odor of burned fibers which occurred during processing, a dark coloring in comparison to the wood-filled part was noted. The odor and the darkening were caused by degradation of the agro-based natural fibers (flax and hemp). The frictional heat resulted in high melt

Fig. 8 Comparison of colouring of different fiber types (wood *left*; flax *middle*; hemp *right*)

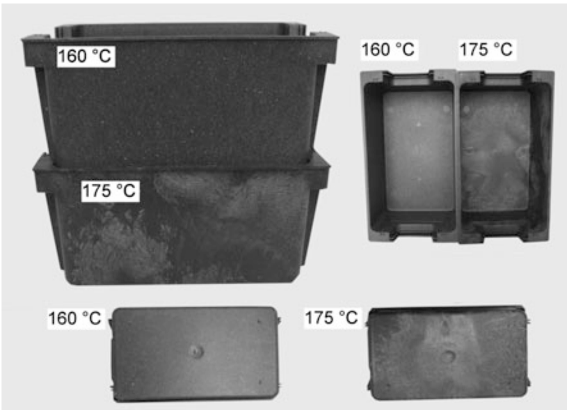
temperatures. Thus, in comparison to the wood-filled material (60 wt% of wood powder), the process parameters for the flax and hemp filled compounds had to be adapted.

Compared to wood fibers, natural fibers like hemp and flax are more sensitive in terms of thermal stability. A special run-up regime of the IMC for natural fibers as well the reduction of the barrel temperature below 160 °C (instead of 175 °C in Fig. 8) resolved that problem. Due to this, the main energy input for melting and compounding was caused exclusively by frictional heat. Furthermore, the throughput was adapted by decreasing the rotational speed of the screw from 200 to 400 rpm to 60 rpm.

In Fig. 9 the result of that processing adaption is shown. There is a significant difference between 160 and 175 °C, which is a remarkable progress in processing of preserved natural fibers.

The mechanical testing of the transport and storage boxes (cf. Fig. 9) with flax and hemp fiber-filling were performed at test specimens cut by waterjet. The specimens for tensile testing (according to DIN EN ISO 527-1 and 527-4) were obtained from the long side of the box and those for Charpy impact testing (according to DIN EN ISO 179-2) from the short side. Figure 10 provides an overview of results of mechanical testing of the transport and storage boxes. All samples are reinforced by a natural fiber content of 30 wt%.

Fig. 9 Comparison of coloring with different barrel heat of 160–175 °C



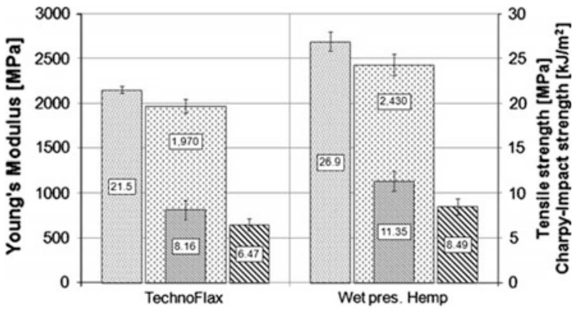


Fig. 10 Mechanical characteristics of test specimens cut out of natural fiber-reinforced boxes (30 wt% fiber content, flax *left*; hemp *right*)

Overall, the results of the hemp-filled (cf. Fig. 10, right) sample exceed the ones of flax-reinforced (cf. Fig. 10, left) sample in all tested mechanical properties, but the general properties are still less than those of common wood polymer composites (WPCs). An addition of a thermoplastic elastomer (TPE) as impact modifier was performed in the second step of experiments in order to improve especially the impact properties. Within the studies two types of TPEs were tested, TPE-1 Exxon Mobile Chemical's propylene-based Vistamaxx™ 6202 and styrene-based TPE-2 Kripoflex which is actually a dry-blend of 65 wt% linear high molecular weight styrene-ethylene/butylene-styrene block copolymer (SEBS with butadiene to styrene-monomer ratio BD/SM of 67/33) and 35 wt% of a low molecular weight oily component. TPE-2 showed especially excellent performance in context of processing WPC due to both mechanical properties and processability (Hartmann et al. 2014). In Fig. 11 the results of impact strength tests are shown in comparison to the unmodified composites. The addition of 20 wt% TPE-2 increased Charpy impact strength at room temperature by more than 100 % regarding the mean values or 50 % by taking the standard deviations into account. The addition of 20 wt% of TPE-1 leads to an increase of 70 % of Charpy impact strength regarding the mean

Fig. 11 Comparison of unmodified and modified natural fiber-reinforced composites (with 30 wt% flax and 20 wt% impact modifier)

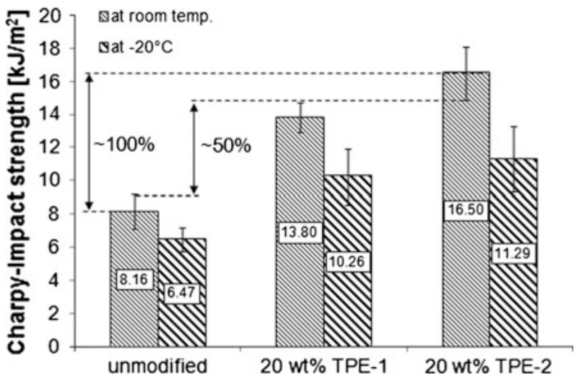


Table 2 Processing parameters of modified and unmodified composites (with 30 wt% flax)

Impact modificator	Torque of extruder—compounding (%)	Melt pressure—injection molding (bar)
Unmodified	39	500
20 wt% TPE-1	32	525
20 wt% TPE-2	27	480

values. The tests at −20 °C show an equal enhancement of impact properties. Moreover, the acceptable advancements for impact properties at the same time Young's modulus shows a decrease of almost 40 % for TPE-1 and 50 % for TPE-2. The tensile strength was also reduced by 25 % on TPE-1 and 32 % on TPE-2 addition. In summary, the mechanical properties still have a huge potential for optimization and will be a topic of further studies.

During the monitoring of the processing parameters for melt pressure and torque, another important fact was observed. The parameters for the torque of extruding section under stable processing conditions and the maximum melt pressure in injection unit are listed in Table 2.

The data show that an addition of TPE-1 and TPE-2 had opposing effects on the material behavior during processing compared to the unmodified sample. While an addition of TPE-1 leads to a small torque reduction of 7 % combined with an disadvantageously increase of melt pressure due to stress of the fiber content, an addition of TPE-2 reduces both, the torque by 12 % and the melt pressure. Thus, adding TPE-2 enables sensitive processing.

Summary

The supply chain for fibrous raw materials is optimized in order to supply particle morphology adapted to the processing requirements. Subsequently, an optimization of the process parameters and the screw configuration of the IMC are realized. Thus, the arrangement of kneading and mixing elements as well as the screw speed is investigated. Finally, downstream and upstream feeding of the fibers is examined. The results reveal that an optimal configuration is characterized by high friction and low heat stress on the material. Higher screw speed leads to lower variations of the resultant values which probably exist due to a better homogenization of fibers in the melt.

Products such as transport and storage boxes are produced on an industrial sized injection molding compounder KM 1300-14000 IMC to demonstrate that the R&D results are not only achieved under lab conditions but also on industrial scale machines. Using an innovative dosing system, Polypropylene, 3 % coupling agent and whole plant materials from hemp and flax at 30 wt% are applied.

The results indicate that there are substantial differences on the mechanical properties of different fibrous raw materials to be used as a reinforcing agent. The supply and processing of raw materials on an industrial and an economic viable scale and respective characteristics of the products is important both in practice and research.

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