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Revolutionizing fiber-reinforced thermoplastic composite recycling: The LightCycle project's journey towards energy-efficient upcycling and quality material regeneration

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ABSTRACT

The recycling of fiber-reinforced thermoplastic composites presents a significant challenge, leading to high waste generation and limited reuse options. The LightCycle project aims to address this issue by developing an energy-efficient and circular method for upcycling glass fiber composite waste and recycling polypropylene (PP). This work focuses on analyzing the material properties of PP regenerates (rPP) from post-consumer waste in Austria, as well as the compounds derived from it and glass fiber-reinforced thermoplastic composites. The goal is to establish material expertise and determine acceptable quality ranges for the LightCycle process. Thermal and rheological material data measurements to analyze the material input quality and the fluctuation range suggest a stable LightCycle process with a wide process window. Furthermore, the analyzis of first compounds with rPP and two different types of shredded glass fiber reinforced thermoplastic composites highlights the significant impact of the type of flake used on the compound's properties.

Index Terms – mechanical recycling, fiber-reinforced thermoplastic composites, circular economy, recycling challenges, waste reduction, limited reuse, circular-oriented approach, energy-efficient methods, upcycling, glass fiber composite waste, PP regenerates, post-consumer, material analysis

INTRODUCTION

Fiber-reinforced thermoplastic composite components, commonly known as lightweight components, offer significant benefits in reducing CO_2 emissions in transportation and mobility due to their low weight. However, the sustainability of these components is currently compromised by the unresolved recycling challenge. Despite regulations mandating a high percentage of recycling in end-of-life vehicles [9], the production of PP organic sheets or unidirectional (UD)-PP tapes for lightweight construction results in up to 40% waste, with only a fraction being effectively recycled. Additionally, the complex geometries of these components require trimming after forming, further complicating the recycling process. The recycling of secondary composites made from these materials is largely unexplored, impeding their use in industries like automotive, which have strict quality requirements. Moreover, recyclates from these components have a "heat history" that makes reprocessing challenging. As a result, fiber-reinforced composite components primarily end up as post-consumer waste and are incinerated. To address these issues and facilitate widespread market adoption, innovative recycling concepts and closed-loop process technologies are required.

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To address these challenges, the funded project LightCycle aims to advance the injection molding compounding process through the establishment of a LightCycle pilot plant. This plant will serve as a technologically advanced, circular-oriented, and energy-efficient method for upcycling glass fiber composite waste and regenerating polypropylene (PP). By doing so, it will close the material loop for glass fiber-reinforced thermoplastic composite components, enhance the efficiency of circular-oriented lightweight production, and open up new market opportunities. The development of a robust industrial manufacturing process for high-quality components made from regenerates-based thermoplastic composites is essential for the LightCycle project. This involves the implementation of recipe methodology, inline measurement technology for assessing glass fiber content and melt quality, and analysis of the interactions between process parameters and component properties. The ecological advantages of the one-step LightCycle process, which involves processing in a single heat stage, including energy savings and resource conservation through the utilization of regenerates, will be demonstrated through comparative life cycle assessments.

The focus of this project is primarily on the material analysis of two key aspects: (1) a PP regenerate produced from post-consumer waste in Austria, aiming to determine the variation in input quality, and (2) compounds derived from the PP regenerate and two different shredded glass fiber-reinforced thermoplastic composites. To process materials with varying qualities, which is often the case with such materials, it is crucial to generate material expertise through analysis. The determination of material data, encompassing rheological, thermo-gravimetric, dynamic, and mechanical properties, forms the foundation of the project. This offline analysis establishes acceptable ranges of material input quality for the LightCycle process and defines the quality expectations for regenerated materials from the compounding process.

EXPERIMENTAL

1-1- Materials

For the experiments of this work, a PP regrind, a virgin PP and two different flakes made from post-industrial glass fiber reinforced thermoplastic composites were grown. The rPP, type Skyplen KC, PreZero Polymers Austria GmbH, Austria, with MFR = 10.3 g/10 min (230 °C, 2.16 kg) and density of 0.93 g/cm^3 was kindly provided by the manufacturer. The material was delivered in a big bag with a filling quantity of 1 ton. The sampling for the material data determination was done at five different locations at 500 g each. The virgin PP, type HE125MO, Borealis A.G., Austria, with MFR = 12 g/10 min (230 °C, 2.16 kg) and density of 0.908 g/cm^3 was used as a reference and was also kindly provided by the manufacturer. In addition, two different post-industrial glass fiber-reinforced thermoplastic composites were shredded and kindly provided by LIT Factory (Linz Institute of Technology, Austria) in an average size of 10 mm. The flakes were from unidirectional fiber composites based on PP and glass fiber, produced by LIT factory and the other one was from technological composite (TEPEX®, Bond-Laminates GmbH (LANXESS Group), Cologne, Germany) kindly provided by ENGEL AUSTRIA GmbH containing PP, carboxylated PP, glass fiber, and minute quantities of stabilizer.'

1-2- Sample preparation

For the rheological analyses plates with a diameter of 25 mm and a thickness of 2 mm were prepared via compression molding in a vacuum press (P200 PV, Dr. Collin GmbH, Ebersberg, Germany). Pressing was conducted at 220 °C in 30 minutes.

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The samples for mechanical characterization were prepared by injection molding machine (Allrounder 470 A 1000-400, Arburg GmbH + Co KG, Germany) with a mold temperature of 45 °C, a nozzle temperature of 230 °C, an injection speed of 30 cm^3/s and a cooling time of 40 s. rPP granules were pre-dried for 2 hours at 80 °C in thermal dryer to eliminate any moisture in the samples.

For composite samples, rPP was compounded with flakes at different composition ration in a twin-screw extruder (ZSE27MAXX, Leistritz Extrusionstechnik GmbH, Nuremberg, Germany) with a twin-screw side feeder. The process was parameterized with a nozzle temperature of 220 °C, a compounder screw speed of 350 rpm, a side feeder screw speed of 250 rpm and a total throughput 6 kg/h.

To further investigate the effect of compounding on rPP, virgin PP and rPP were compounded alone in compounder. The sample codes listed on table 1.

The compounds were injected molded same as previous samples for the **mechanical characterization**.

Table 1- Sample coding of composites containing rPP and different types of flakes

Sample Code	rPP (wt.%)	PP (wt.%)	EN flakes (wt.%)	UD flakes (wt.%)
rPP Pos (1-5)	100	0	0	0
PP	0	100	0	0
rPP9UD	90	0	0	10
rPP7UD	70	0	0	30
rPP9EN	90	0	10	0
rPP7EN	70	0	30	0

1-3- Thermal Characterization

2-3-1- Differential scanning calorimetry (DSC)

Specific heat capacity measurements were performed using a differential scanning calorimeter (DSC Mettler Toledo, Greifensee, Switzerland) based on ASTM 3418. The measurements were carried out in the temperature range from 30 to 230 °C with a heating rate of 10 K/min, and a cooling rate of 20 K/min. The DSC was calibrated in the same temperature region before each experiment, using a sapphire sample as standard, with a well-known specific heat capacity. Furthermore, melting temperatures and fusion enthalpy values were calculated for virgin and recycled polymer samples from the calorimetric data obtained during the second heating ramp. The degree of crystallinity in each virgin polymer was calculated as follows in Equation 1:

$$X_c = \frac{\Delta H_m}{\Delta H_m^0 * w} * 100 \tag{1}$$

Where ΔH_m is the enthalpy of melting, ΔH_m^0 is the enthalpy of 100% crystalized PP which is 207 J/g, and w is the weight fraction of PP in the samples.

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2-3-2- Thermogravimetric Analysis (TGA)

In order to analyze the thermal stability of rPP and composites, TGA analysis was performed with (Mettler Toledo GmbH, Greifensee, Switzerland) based on ISO 11358. All the samples were heated from 25 to 600 °C with a heating rate of 10 C/min in nitrogen environment and from 600 to 800 °C with a heating rate of 20 °C/min in oxygen environment to further analysis inorganic additives in the samples.

1-4- Rheological characterization

2-4-1- Parallel plate rheometer (PPR)

The rheological properties of rPP were measured using a rotational rheometer, type MCR 702 MultiDrive (Anton Paar GmbH, Graz, Austria). The experiments were carried out in parallel plate configuration at 210, 220, and 230 °C in a nitrogen environment with a plate diameter of 25 mm. Strain sweeps were carried out in the strain range from 0.01 to 10 % at a fixed frequency of 1 rad/s to measure the zero-shear viscosity of rPP. In the evaluation, the complex viscosity at a frequency lower than 0.1 s^{-1} was used as an indicator for the zero-shear viscosity. All frequency sweep tests were conducted in the linear viscoelastic region, which was chosen at a strain of 1 %, as confirmed by the strain sweep tests. All the samples had the same recent past thermal history. At least two replicates were performed on fresh samples.

1-5- Mechanical characterization

2-5-1- Tensile and Charpy Test

The tensile properties of the samples were measured according to ISO527 using a Zwick Z001 machine with a 1 kN load cell from ZwickRoell GmbH & Co. KG, Ulm, Germany, at room temperature. The initial gauge length and displacement rate were 50 mm and 10 mm/min, respectively. Furthermore, Charpy impact test was conducted on the notched samples, based on ISO 179-2 with ZwickRoell GmbH & Co. KG, Ulm, Germany, at room temperature. The mechanical properties results were averaged over five samples of each composition.

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3- RESULTS AND DISCUSSION

3-1- Input Quality

Fluctuations in the recyclate stream, particularly in the post-consumer sector, are widely reported. These fluctuations arise from various factors, such as the material selection for packaging, which often includes challenging-to-separate multi-layer materials or combinations. Moreover, the inadequate separation practices of consumers hinder the attainment of high-quality recycled output. Additionally, the low purchase prices of sorted bales for sorting plants can lead to subpar output quality, thereby impacting the subsequent recycling process [1, 5-8].

3-1-1- Thermal properties - DSC

In order to investigate the fluctuation in thermal properties of rPP, five samples, taken from the big bag with 1 ton of material, were compared with the virgin PP. The measurements were repeated three times. Endothermic melting peaks (figure 1) are observed for the investigated samples. The first being associated with polyethylene (PE) melting (∼125°C) and the second with α crystals melting from rPP (∼162°C). This suggests that the recycled polymers represent a mixture of different polymers [2]. The obtained results indicate that the crystalline phase of PE remains separated in PP domains at microstructural level. If it is assumed that the crystalline phases from PE are the same with the amorphous ones [1-2], then the total amount of PE from recycled PP is ∼3-5%. Furthermore, the shape of melting curves is similar to each other which means similarities in lamellar thickness of PP crystallites and their degree of perfection. The crystallinity content of rPP samples were measured based on formula 1. It is good to mention that, the crystallization behavior and crystallization content directly affect shrinkage, warpage, and mechanical properties of the final product. Furthermore, the fluctuations in rPP thermal behavior in DSC was infinitesimal and can be neglected. For a comparison with the virgin PP, a mean value was calculated from the rPP results. The thermal properties are summarized in table 2.

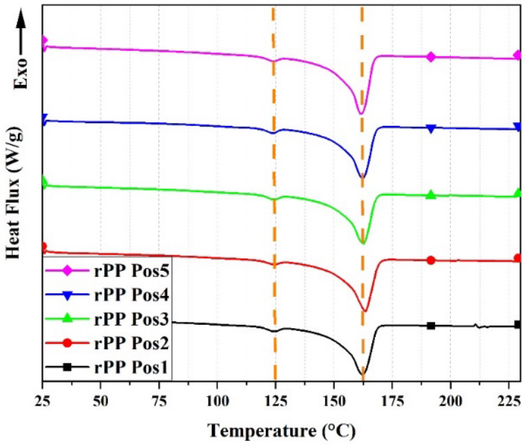

Figure 1- DSC thermograms of rPP samples

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Table 2- Thermal properties of rPP and virgin PP

Sample	**Heating**		**Cooling**		**Xc**
	Tm [°C]	**ΔH_m [J/g]**	**T_c [°C]**	**ΔH_c [J/g]**	**(%)**
rPP	162.5 ± 0.7	49.8 ± 2.6	121.1 ± 0.9	64.3 ± 3.3	31.0 ± 1.5
PP	167.0 ± 0.8	85.5 ± 1.9	109.5 ± 0.7	83.3 ± 0.6	40.2 ± 0.3

3-1-2- Thermogravimetric properties - TGA

To further investigate the thermal degradation behavior of rPP and to find the maximum operation temperature, TGA analysis was used. The results are depicted in figure 2a. All samples showed a nearly similar thermal degradation behavior, onset and end sets. The absence of a carbonaceous residue at the end of the degradation process indicates that the thermal degradation of rPP appears to be a one-step reaction, as represented by the single peak observed in figure 2. This can be attributed to the radical random scission mechanism of the PP thermal degradation, which is typical for polyolefins [3, 4]. For a comparison, TGA thermograms of the virgin PP are also depicted in figure 2-b. The virgin PP starts to degrade at a higher temperature than rPP, but it degrades faster (inset of figure 2-b), and the degradation end set of virgin PP is lower than rPP. This behavior is mainly reported in isotactic PP, and due to the presence of undefined PP types, like random or block copolymers, the degradation rate is different, and the presence of ethylene domains in rPP hinders the thermal degradation. In addition, it is not known which PP types and stabilizers the rPP regrind consists of. Furthermore, the maximum operating temperature is reported when around 25% of the sample has degraded, and after that, the material starts to degrade rapidly. Based on figure 2-b, the maximum operating temperature for rPP is around 430 °C, and for virgin PP is around 425 °C. It must be considered that the measurement was carried out in a nitrogen atmosphere and that the maximum processing temperature in reality will be at least 40 °C lower.

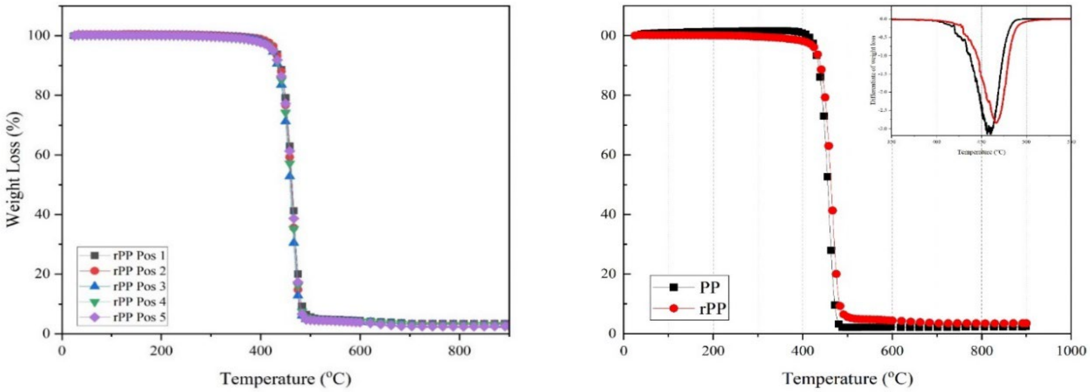

Figure 2- TGA thermograms of (a) rPP samples, and (b) comparison between rPP and virgin PP

3-1-3- Rheological properties - PPR

This characterization aimed to analyze the rheological properties and potential variations of rPP using a rotational rheometer. The complex viscosity of rPP samples was investigated, and no significant difference was observed, indicating consistent material flow behavior (Figure 3a). To qualitatively evaluate the flow behavior, the complex viscosity of rPP and virgin PP were compared at three temperatures (Figure 3b).

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Both materials exhibited similar shear-thinning behavior across the frequency range. However, at 210 °C, rPP displayed higher viscosity (in average 11 %) than virgin PP, suggesting that the presence of ethylene domains in rPP increases flow resistance and viscosity. Interestingly, at a temperature of 220 and 230 °C, the virgin PP exhibited higher viscosity than rPP. The differences are in average 5 % and 6 %, respectively. Further analysis is recommended, including examining different types of commercial PP, such as random or block copolymers, and comparing their rheological behavior with rPP. It's important to note that the investigated rPP was sourced from various post-consumer waste streams, where PP copolymers commonly used in packaging likely represent a significant fraction. Therefore, comparing the results with PP copolymers is crucial to establish a general understanding of rPP properties.

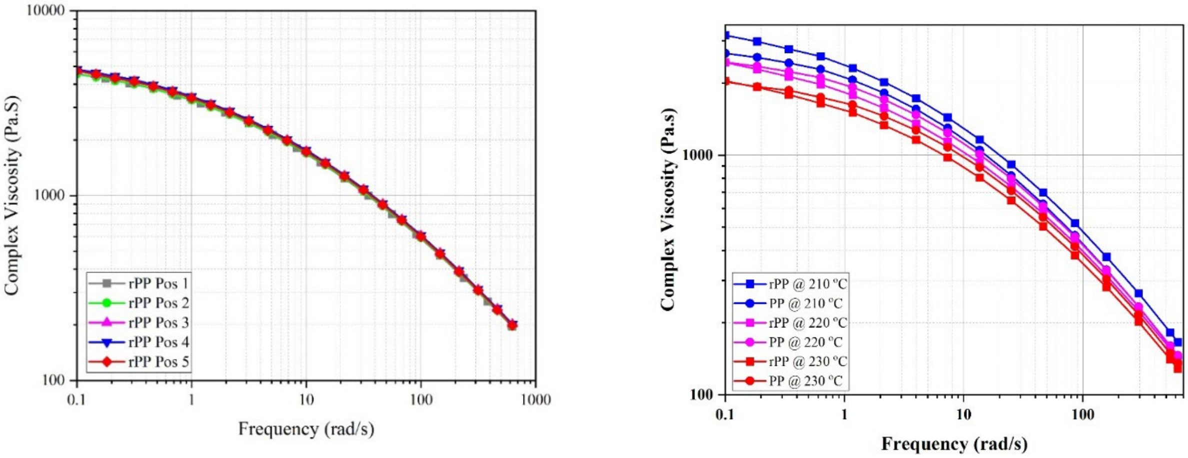

Figure 3- Rheological behavior of (a left) rPP samples, and (b right) comparison between rPP and virgin PP at three different temperature 210, 220, and 230 °C under nitrogen environment

3-1-4- Mechanical properties

To investigate the mechanical behavior of the injected molded parts, tensile and Charpy impact tests were conducted on rPP as well as virgin PP, and the results are summarized in table 3. It is good to mention that all the mechanical testing was performed 2 weeks after the injection to further consider the post-crystallization phenomenon in PP. As expected, higher tensile properties were observed in virgin PP compared to rPP. An average difference of 17 and 28% was observed in the elastic modulus and tensile strength of rPP compared to virgin PP. This would be mainly due to the higher crystallinity content of virgin PP. However, interesting results were observed in the Charpy impact test. The impact resistance of rPP was 44% higher than that of virgin PP. The probable reasons for this behavior are the presence of different PP copolymers in rPP and PE contamination, which is reported to be an impact modifier in rPP samples.

Table 3- Mechanical properties of injected molded rPP and virgin PP

Sample	Elastic modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)	Impact resistance (kJ/m2)
rPP	1.7 ± 0.0	34.0 ± 0.1	27.0 ± 18.8	5.0 ± 0.4
PP	1.4 ± 0.0	24.4 ± 0.0	15.1 ± 6.1	2.2 ± 0.2

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3-2- Compound quality (rPP/flakes)

3-2-1- Thermal properties (DSC)

To investigate the effect of the content and type of glass fiber on the thermal properties of rPP, DSC analysis were done, and the results are summarized in Table 4. Adding glass fiber flakes to rPP did not change the melting and crystallization temperatures of rPP; however, in both composite series, by increasing the flake content, crystallinity content (Xc) increased. This behavior is mainly due to the nucleation effect of the solid glass fiber in the rPP matrix, which provides a nucleation spot for PP chains to form a crystalline structure. The comparison of two series of composites suggests that a composite based on rPP/UD flakes has a higher crystallinity content than rPP/EN flakes at the same composition ratio. A likely explanation for this behavior would be the presence of carboxylated PP in the ENGEL flakes, which prevents the PP chains from forming a crystalline structure and reduces the growth rate.

Table 4- Thermal properties of rPP composites based on UD and EN flakes with different composition ratio

Sample	Flake Content [%]	Tm (PP) [°C]	Tm (PE) [°C]	T_c (PP) [°C]	Xc (%)
rPP9U1D1	10	163.4 ± 0.5	125.4 ± 0.2	120.9 ± 0.5	32.1 ± 0.4
rPP7U1D3	30	163.1 ± 0.4	124.9 ± 0.0	121.5 ± 0.8	38.8 ± 0.4
rPP5U1D5	50	162.7 ± 0.1	124.7 ± 0.0	122.1 ± 0.2	49.8 ± 0.3
rPP9EN1	10	163.3 ± 0.3	125.4 ± 0.2	120.9 ± 0.7	31.3 ± 0.6
rPP7EN3	30	164.6 ± 0.3	125.4 ± 0.0	120.4 ± 0.0	35.7 ± 0.4
rPP5EN5	50	163.8 ± 0.9	125.0 ± 0.1	121.8 ± 0.5	44.8 ± 0.7

3-2-2- Thermogravimetric Analysis (TGA)

To further investigate the effect of flake content and type on the thermal degradation behavior of rPP, TGA analysis was performed on the composites. The overall degradation behavior of composites was the same as that of rPP, showing a single-step degradation, mainly attributed to the radical random scission mechanism of PP thermal degradation. Regardless of the type of flakes used in the composites, by increasing the flake content, the onset of thermal degradation is reduced (table 5). However, the thermal degradation end-set increased with increasing fiber content. This behavior has previously been reported in nanocomposites and systems containing glass fiber, where a solid particle hindered thermal degradation and random chain scission by scarifying itself. The residues after each experiment were also measured. The residues in rPP are due to the presence of additives, and the residues after degrading the composite represent the amount of glass fiber and additives present in the granules. However, it is recommended to measure the ash content in order to validate the amount of glass fiber in the samples.

Table 5- TGA results of rPP and composites based on rPP and UD and EN flakes with different composition ratio

Sample	T onset [°C]	T endset [°C]	Residues [%]
rPP	449.6	475.0	3.8
rPP9U1D1	446.3	479.8	8.5
rPP7U1D3	442.8	480.5	19.0
rPP5U1D5	443.9	478.4	30.9
rPP9EN1	445.4	479.7	10.3
rPP7EN3	444.0	480.2	23.1
rPP5EN5	439.2	477.3	37.2

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3-2-3- Rheological Properties - PPR

In order to investigate the effect of the content and type of the flakes on rheological properties, a rotational rheometer was used at a set temperature of 220 °C. The dependency of complex viscosity on angular frequency is depicted in figure 4 (a). At the whole range of frequencies, the complex viscosity of composites of rPP/EN flakes is higher than that of rPP/UD flakes. This behavior is mainly due to the presence of carboxylated PP and minute stabilizers present in EN flakes. The presence of a compatibilizer like carboxylated PP would increase the interfacial attraction at the interface of glass fibers and the PP matrix. This would further reduce the slippage of PP chains on the glass fibers, increase the flow resistance, and further increase the complex viscosity. To investigate the effect of flake content on the complex viscosity of each composite series, the dependency of viscosity on angular frequency is depicted in a separate figure. In the rPP/UD composite (figure 4 (b)), three different dependencies were observed in each frequency range. First, at high frequencies (100–1000 [1/s]), rPP7U1D3 and rPP5U1D5 show nearly the same complex viscosity, higher than rPP9U1D1. This is a normal behavior in systems containing polymers and solid particles; by increasing the flake content, the system shows higher flow resistance and higher viscosity. However, at medium frequencies (0.3–100 [1/s]), totally different behavior was observed. rPP9U1D1 has the highest viscosity, followed by rPP7U1D3, and the lowest viscosity is rPP5U1D5. The probable explanation for this behavior is due to the absence of the compatibilizer or any additive, where the flakes exerted more shear stresses on the rPP matrix and lowered the viscosity. Finally, at low frequencies (0.1–0.3 [1/s]), rPP9U1D1 and rPP7U1D3 show the same viscosity, but a viscosity upturn is detectable in rPP5U1D5, which is mainly due to the networking of the glass fibers in the sample. The other possible explanation for the viscosity upturn is the orientation of the fiber under rotational forces exerted by the rheometer. To investigate the reason, in our future work, we will examine the effect of the orientation of the fiber on the flow behavior.

For rPP/EN composites (figure 4 (c)), in the whole range of frequencies, the samples containing more flakes showed higher viscosity. Here, one can notice the importance of the presence of the compatibilizer in systems containing glass fiber. Furthermore, at low frequencies (0.1–0.3 [1/s]), the same as in rPP5U1D, a viscosity upturn is detectable.

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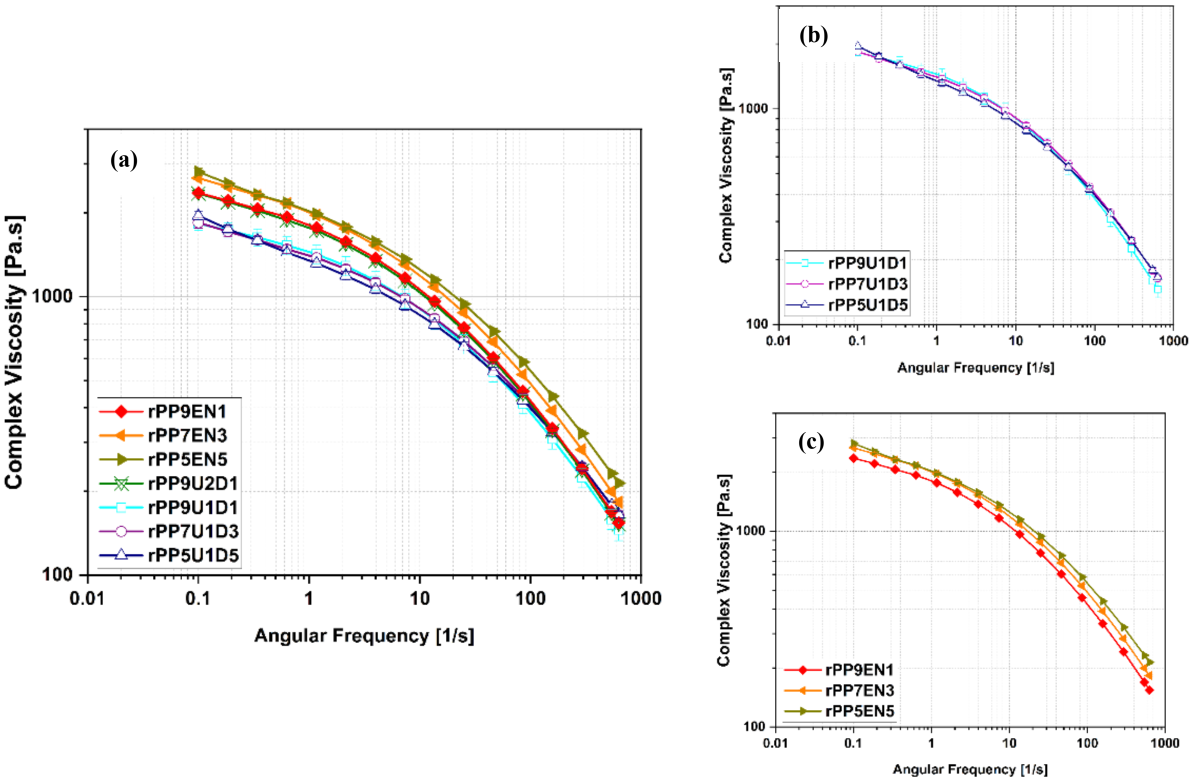

Figure 4- The dependency of the complex viscosity on frequency (a) in the whole composite series, (b) rPP/ UD flakes composites, and (c) rPP/EN flakes composites

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4- CONCLUSION

To identify the variation of the input quality of a PP regenerate, produced from Austrian post-consumer waste, for the new technological, circular-oriented, and energy-efficient LightCycle process, thermal, rheological and mechanical material analysis of these rPP as well as of a reference material (virgin PP) took place. Furthermore, compounds made from rPP and two different shredded glass fiber reinforced thermoplastic composites were produced and also characterized. This offline analysis was used to determine the ranges of variation for (1) the acceptable quality of the material input in the LightCycle process and (2) the quality of the regenerated material from compounding.

The analyses of the input quality were investigated by thermal, rheological and mechanical measurements comparing five samples taken from a big bag containing one ton of rPP. The comparison between these samples showed no significant differences. The DSC measurements showed contamination by PE. With the TGA measurements a maximum processing temperature of approx. 400 °C was determined. A comparison of the rPP with a virgin material with nearly the same MFR = 12 g/10 min (230 °C, 2.16 kg) showed a 9% lower crystallinity, as well as an increase of the flow resistance and viscosity at 210 °C, which is probably due to the presence of ethylene domains in rPP. In contrast, the virgin PP exhibited a higher viscosity at a temperature of 220 and 230 °C than rPP. The Charpy impact tests of the mechanical analysis showed a 44% higher impact strength of rPP compared to the virgin PP. This improvement is likely due to the presence of different PP copolymers in rPP and the impact-modifying effect of PE contamination. The analyses of the compound quality comparing two different shredded post-industrial glass fiber-reinforced thermoplastic composites showed that the increase of the flake content in both composite series resulted in higher crystallinity content. Comparing the two composite series, rPP/UD flakes exhibited higher crystallinity content than rPP/EN flakes at the same composition ratio. This difference may be explained by the presence of carboxylated PP in the ENGEL flakes, which hinders PP chains from forming a crystalline structure and reduces the growth rate. In the TGA analysis, performed on the composites to study the thermal degradation behavior of rPP, it was found that regardless of the type of flakes used, increasing the flake content reduced the onset of thermal degradation but increased the end-set of degradation. This behavior is consistent with previous findings in nanocomposites and systems containing glass fiber, where the presence of solid particles hindered thermal degradation and random chain scission. The residues after degradation indicate the presence of additives and the amount of glass fiber in the composites, although measuring the ash content is recommended to validate the glass fiber content. The complex viscosity of rPP/EN composites was higher than that of rPP/UD composites at all frequencies due to the presence of carboxylated PP and stabilizers in the EN flakes. These increase the attractive force at the interface and reduce the slippage of the PP chains on the glass fibers. The effect of flake content on complex viscosity varied in different frequency ranges, with a higher flake content leading to a higher viscosity at high frequencies and a different behavior at medium frequencies. At low frequencies, an increase in viscosity was observed due to crosslinking of glass fibers. The presence of a compatibilizer was critical in systems with glass fibers.

The first outcome of the material data measurements from the samples taken from the big bag thus indicates a stable process at the LightCycle plant, as there were no significant differences observed in terms of input quality variation. Additionally, the analysis of the compounds revealed that the presence of a carboxylated PP compatibilizer in EN flakes improves the interface between PP and flakes, reduces shear stresses on PP chains, prevents thermal degradation, affects the crystalline content of PP, enhances viscosity, and overall improves the mechanical properties of the composite. Based on these findings, it can be anticipated that rPP/EN flake composites will demonstrate superior mechanical properties compared to rPP/UD flake composites.

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