Mechanical properties of recycled PP/sugarcane bagasse fiber composites

Bachelor thesis

by

Adrián Segura Almengló

Supervisor: András Bartos

Laboratory of Plastics and Rubber Technology

Department of Physical Chemistry and Materials Science

Budapest University of Technology and Economics

2021

Contents

Abstract

In the last few decades, the consumption of polymers used on a daily basis has been increasing tremendously, and so with that, the necessity of obtaining the necessary raw materials, as well as the management of the generated waste. For this reason, the interest on finding new methods for decreasing the raw material quantity needed, as well as increasing the useful life of the polymers has increased significantly. Accordingly, the investigation and research of composites based on a polymeric matrix and natural fiber reinforcements have been generating a great interest since years ago, as well as the micromechanical deformation processes which occur during their use; without forgetting how the recycling processes affect to their mechanical properties.

This project presents the research of recycled polypropylene (PP)/sugarcane bagasse fiber (SCB). Different samples of composites were prepared, variating the SCB proportion and the application of a coupling agent. In first place, the extrusion of the raw materials was carried out for composite preparation, then injection molding was used to obtain dogbone specimens, from which 18-20 pieces were used and the rest was ground for recycling the composite. Mechanical properties like Young's modulus, yield stress, tensile strength and corresponding deformations, as well as impact resistance were determined. Simultaneously with the tensile testing, local deformation processes were detected using the acoustic emission (AE) technique.

The grinding steps of the materials were repeated twice in order to model the recycling process and to study its effect on the micromechanical deformation processes and on the macroscopic mechanical properties.

Key words: composites; polymers; natural fibers; recycling; micromechanical deformations

Resumen

En las últimas décadas, el consumo de polímeros empleados en el día a día ha ido aumentando considerablemente y con ello también la necesidad tanto de obtener la materia prima necesaria, así como gestionar adecuadamente los residuos generados. Es por ello que el interés en encontrar métodos que disminuyan la cantidad de materia prima necesaria, así como de poder alargar la vida útil de los polímeros se ha incrementado de manera notable. Debido a esto, la investigación y el estudio de composites formados por una matriz polimérica y refuerzos de fibras naturales lleva generando un gran interés desde años atrás, así como los procesos de deformación micromecánica que puedan producirse durante su empleo; sin olvidar el cómo afecta el reciclaje de estos composites a sus propiedades mecánicas.

Este trabajo presenta el estudio de composites formados por polipropileno H649 (PP) y fibra de bagazo de caña de azúcar (SF). Se realizan diferentes muestras de composite variando tanto la cantidad de SF como la inclusión de un agente enlazante. Se realizará en primer lugar la extrusión de la materia prima para la formación del composite, así como una segunda extrusión para obtener las probetas para los ensayos, de las cuales se utilizarán entre 18 y 20 y el resto se triturarán para reciclar el composite. Se realizan los ensayos para obtener las propiedades mecánicas como el esfuerzo a la tensión, el módulo de Young, la resistencia al impacto. Simultáneamente al ensayo de tensión, los procesos de deformación locales se detectan mediante el empleo de la técnica de emisión acústica (AE).

Los procesos de triturado del material se repiten dos veces con el fin de simular el proceso de reciclaje y estudiar el efecto de los procesos de deformación micromecánicos y las propiedades mecánicas macroscópicas.

Palabras clave: composites; polímeros; fibras naturales; reciclaje; deformaciones micromecánicas

1. Introduction

Since many decades, the use of polymers in our daily life has increased significantly until being an indispensable material in the way life is concerned nowadays. Only in 2018, the global plastics production almost reached 360 million tons [1]. This high demand on polymers induces a high demand on raw materials also. Accordingly, the need of finding ways for recycling polymers has increased in the recent years. The recycling rate for plastics today is approximately 25% although the recovery and recycling rates are increasing in many countries around the world, and an international market for recycled plastics is developing [2].

Also, there have been a lot of research dealing with the opportunities polymers can be produced with the less raw material proportion possible but also having the same or even better properties like increased stiffness, strength and/or impact resistance. This resulted in the birth of the composite materials. In the recent years, the aim has been to create composites by reinforcing the polymer with natural fibers, in order to be much more biodegradable and also for finding utility for the natural fibers, because most of the ones which are used are waste from other processes or materials.

The potential use of natural fibers is gaining interest in developing countries, as they often have tropical plants from where they can obtain the natural fibers. Plants like kenaf, jute, cotton, bamboo, or banana have great potential as reinforcements in polymer composites due to their excellent specific mechanical properties and, also for being environmentally friendly, renewable, cheap and being the principal alternative to synthetic fibers like glass or carbon [3].

Since the natural fibers have been a topic with great industrial interest in the previous years, the development has focused on the mechanical improvement and the reduction of the structural defects they have, as they are natural materials and not every sample is perfect. This aims to ensure durability, reliability, and increased production with less cost [4].

1.1 Polymers

Polymers can be defined as natural or synthetic substances composed of macromolecules, which are multiples of simpler chemical units called monomers [5]. Polymers can be classified in some different ways according to different factors:

- By the structure of the chain:

- Linear: all of the carbon-carbon bonds of the chain exist in a single straight line. These polymers can form a very strong mesh which will be hard to break when it suffers from external efforts [6].

- Branched: lateral chains bonded to the main one. If the polymer has multiple branches, it is called dendrimer.

- Crosslinked: there are bonds formed with close chains.

- By their origin:

- Natural: they can be found in the nature (cellulose, starch)

- Synthetic: they are created with processes of synthesis. E.g. nylon, Kevlar, neoprene.

- By the way the chains get ordered:

- Thermoplastic: chains are not chemically crosslinked. They get soft with heat and change their form (polyethylene, polypropylene)

- Thermoset: chains are crosslinked, forming a closed mesh. Their form does not change with heat (phenol – formaldehyde)

- Elastomer: chains are organized in mesh form with few bonds. Great elasticity and recuperation of the form when the effort stops.

- Depending on the stereoregularity (the way the atoms are organized in the repetitive unities of the molecule)

- Isotactic: substituents are placed in the same side of the chain.

- Syndiotactic: substituents are alternated.

- Atactic: substituents are placed randomly.

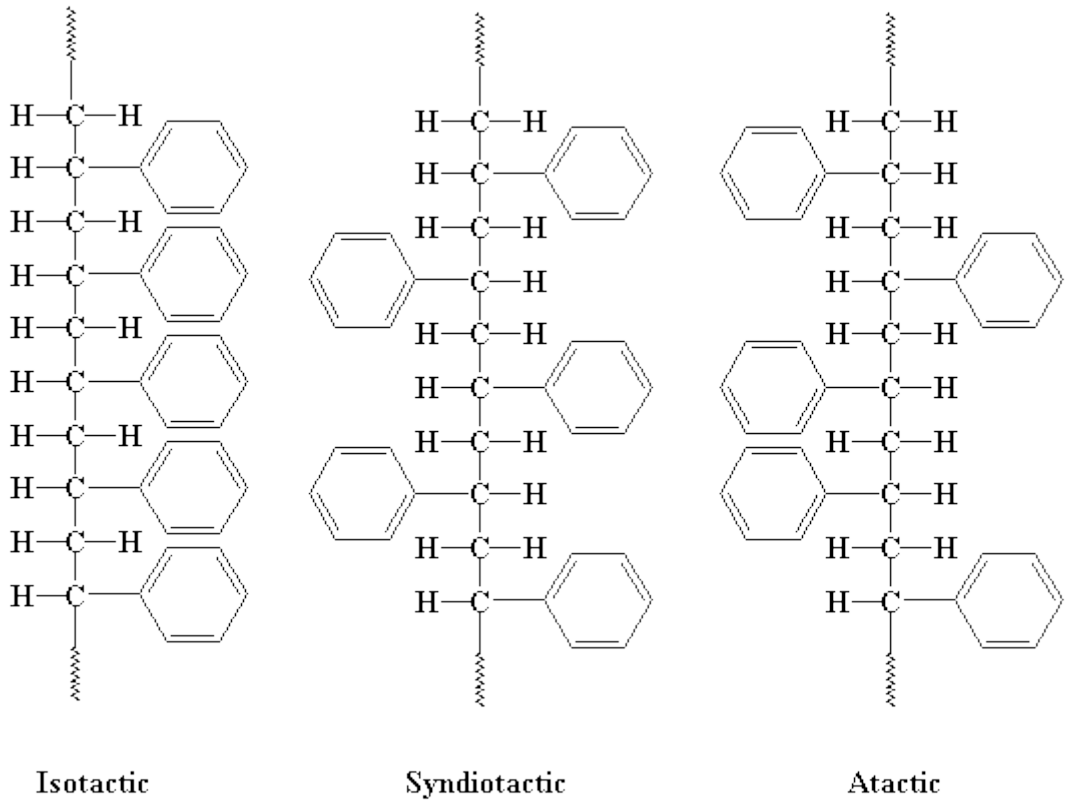

Figure 1 Tacticity of the polymers

- Attending to their composition:

- Homopolymers: all the monomers which form the polymer are the same.

- Copolymers: formed by two or more different monomers. They can be placed alternating, in blocks or randomly.

Talking about the properties of the polymers, there are some factors which are very important to know because, in front of an effort or a change in the properties of the environment like temperature or humidity, the material can experience several changes. The glass transition temperature (Tg) is the one which influences the behavior of the polymers. It is strongly related with the mobility of the chains: if the mobility is high, the Tg values are low; and if the mobility is low, the Tg values are high. It is a reversible transition, and the presence of large and polar groups greatly increases the value because it makes the mobility more difficult.

The melting temperature (Tm) is defined as the one at which the polymer suffers a transition from the crystalline state into the viscous flow one. The process of melting is carried out in a range of temperatures, not in a single one. This is due to the diversity of the chemical compounds which can be found in the polymeric chain [7].

The degradation temperature (Td) is the one from which the polymeric chains begin to suffer combustion reactions. It is an irreversible reaction which triggers the degradation of the polymer. The structure of the polymer determines the value of Td, because as the chains of hydrocarbons have high combustible properties due to the carbon and hydrogen groups, the presence of halogens hugely rises the Td value due to the low combustible properties [8].

All these values mentioned above are very important during the use life of the plastic, but also when it turns into waste. Nowadays, the industry has a huge concern about minimizing waste and being environmentally friendly and plastics are not commonly seen as a good option talking about recycling. The fact is that thermoplastic polymers are the ones which can be recycling due to the low melting temperature they have. This allows the industry to be more efficient while wasting less raw material. Pyrolysis is one of the recycling processes polymers can have, and it is defined as the heating of an organic material in absence of oxygen. This lack of oxygen means the material will not combust, but the chemical compounds will be degraded and decomposed in other combustibles like gases or charcoal.

1.2 Composites in the plastic industry

Since the early part of the twentieth century, the development and application of plastic and reinforced plastic composites has experienced an increase, principally due to the suitability of producing them in a massive way, the ability to be molded in several shapes and the great resistance to environmental factors. [9] As the use of polymers has dramatically increased within the years, it has also produced a large necessity on variating the mechanical properties that they have. As polymers formed by an only component can barely change their mechanical properties, the need of be able to variate them, like increasing stiffness, ductility, impact resistance or strength, in order to use them in a huge

quantity of applications; has produced an enormous popularization in the use of composites.

The main reason of the composites' popularization is the ability they provide of variating the mechanical properties in an easier and cheaper way than investing time and economical resources on finding new polymers with the desired mechanical properties. There are also many different ways in order to modify a polymer: filling it with particles, performing a polymer blend or reinforcing it with fibers. Although all of them are interesting and useful and will be discussed after, the main topic of this research project is strongly related with the natural fiber reinforced composites. Natural fibers are highly chosen instead of other types of reinforcement due to their low cost, the biodegradability, the high availability and the specific mechanical properties they have [10].

Regarding the composites, some factors have to be taken into consideration for obtaining the best performance possible with the desired characteristics:

-Component properties: the characteristics of the matrix have an enormous influence on the way the reinforcement works in the composite. The lower the stiffness of the matrix, the higher the reinforcing effect of the filler. Elastomers are highly improved with the use of fillers as both stiffness and strength are raised [11]. In the case of the matrix being weak, the filler will carry an enormous part of the load. Also, properties like the chemical composition, purity and the surface free energy will highly influence the way the reinforcement bonds with the matrix. It is very important to consider also the characteristics of the filler particles in wood flour composites, as they can change the way the composite gets deformed and the way the failure happens, which will determine the properties of the composite.

-Composition: the matrix and reinforcement ratios in the composite have a huge impact on the properties as well. The range of adding reinforcement is quite high, from a few ppm to 70% depending on the reinforcement type. As the reinforcement will provide composites of higher stiffness or stability, some other properties can get worse, so it is very important to be sure of what properties does the composite need.

-Structure: it is highly assumed that the structure of the polymers filled with small particles is homogeneous but that is quite rare to happen and often some structures related with particles merge in the composite. The most important topic about the structure is the different orientation the anisotropic reinforcement can have, so it can variate the

properties of the composite in an enormous way. Although all of this, the structure in composites is highly complicated and is in the field of the nanocomposite where it has an enormous relevance, where multiple structures of small particles can appear and hugely influence the properties of the material.

-Interfacial interactions: when adding the reinforcement to the matrix, the way they bond has a huge impact on the mechanical properties of the composite. If the interfacial interactions are not proper, the deformation processes will appear quite easier than in a composite with optimum interfacial interactions. It is known that in the case of composites formed by polyolefins reinforced with natural fibers, the interfacial interactions are not the best, causing that a lot of intermolecular bonds between matrix and reinforcement are not carried out and the voids in the composite will appear. For avoiding that phenomenon, it is necessary to use a coupling agent or perform chemical and physical techniques which eases the bonding process so we can be sure the composite has the best possible interfacial interactions.

The most common physical and chemical techniques are the following ones [12]:

-Alkali treatment: heating the fibers at 80ºC in 10% NaOH aqueous solution during 3-4h, washing and drying in a ventilated oven. Allows to obtain smaller and better-quality fibers and also improve fiber wetting.

-Acetylation: fibers are immersed in glacial acetic acid for 1h, then in a mixture of acetic anhydride and a little quantity of concentrated sulphuric acid for few minutes. After this it gets filtrated, washed and dried in a ventilated oven. It is an esterification method which stabilizes the cell walls in what humidity absorption involves. Together with alkali treatment, is the one which provides the best results for thermal and dimensional stability.

-Treatment with stearic acid: solution composed by stearic acid and ethyl alcohol. It is added by drops in the fiber which are then dried in the oven. It produces also an esterification process in the fibers.

-Benzylation: fibers immersed in 10% NaOH and stirred with benzoyl chloride for 1h filtrated, washed and dried, then immersed in ethanol for 1 h, rinsed and dried in oven. This method allows decreasing the hydrophilicity of the fibers.

-TDI treatment: fibers are immersed in chloroform with few drops of a catalyst based on dibutyltin dilaurate and stirred for 2 h after adding toluene-2,4-diisocyanate. After this process the fibers are rinsed in acetone and dried.

-Peroxide treatment: the fibers are immersed in a solution of dicumyl (or benzoyl) peroxide in acetone for about half an hour, then decanted and dried. It has been proved by some research that it produces a great improvement in mechanical properties of the fibers.

- Anhydride treatment: it is usually carried out by utilizing maleic anhydride or maleated polypropylene (or polyethylene) in a toluene or xylene solution, where the fibers are immersed for impregnation and reaction with the hydroxyl groups on the fiber surface. As a result, the water absorption of the fibers is significantly reduced.

- Permanganate treatment: the fibers are immersed in a solution of $KMnO_4$ in acetone (typical concentrations may range between 0.005 and 0.205%) for 1 min, then decanted and dried. The hydrophilic nature of the fibers gets hugely decreased.

- Silane treatment: the fibers are immersed in a 3:2 alcohol– water solution, containing a silane-based adhesion promoter for 2 h at pH around 4, rinsed in water and dried in the oven. This treatment improves the quality of the surface of the fiber. Together with the TDI treatment, it is the one which guarantees the best results for the mechanical properties.

1.3 Fiber reinforcements

The use of fibers, either natural or synthetic ones, as reinforcements has improved the way polymers can behave when their mechanical resistances have been tested. If we look in the history, natural fibers such as cotton and wool have been used since a lot of centuries ago. They could be found in the nature, and they provided strength when a light weight was required. The investigation with new analytical techniques such as X-ray diffraction, allowed to explain the reason of why the materials in fiber form have these unique properties. The molecules within fibers tend to align along the fiber axis, making the strength and the stiffness quite superior to the same material in a random form. Also,

when natural and synthetic polymers are extruded into fiber form, the structure is oriented along the fiber axis, resulting in increased strength and stiffness of the material. [13]

Talking now about the fiber reinforced composites, two different parts can be named: the matrix, which is the polymer, acts like a holder and has more presence in the composition; and the reinforcement, which gives the polymer the improved mechanical properties. [14] In the composites, the fibers are surrounded by a layer of matrix which holds them in the desires position, distributes the load over all the fibers and also protects them from any type of possible degradation due to environmental factors.

Regarding on the mechanical properties of the fiber reinforced composites, we cannot just pay attention to the chemical composition of the fiber. It is very important also to check its physical and geometric parameters, as they are highly dependent on the length and the volume fraction of the fiber [15], the fiber orientation and the interfacial interactions [16]. Studies have proved that varying the fibers angle of orientation from 0º to 90º turns out as a huge variation of the mechanical properties [17].

1.4 Sugarcane bagasse

As mentioned before, the huge advance in the industry has led to an enormous augment in raw materials needs and also in the way the wastes are treated, especially focusing on the recycling field. As polymerics materials need between 20 and 500 years for decomposing depending on the type and the structure [18], an important field of research has been the way polymers can be reinforced with natural fibers, conducting into desired mechanical performances and easing not only the way they can be decomposed after the useful life finishes, but also the price and the weight if we compare them to the synthetic fiber composites. Regarding to the natural fibers, quite interesting properties can be found in them like high strength and stiffness, also they are light, non-corrosive and flame retardants. [19] In this project, the focus is on sugarcane bagasse. Sugarcane is a popular cultivation in tropical and subtropical countries, finding the largest productions in Brazil (721), India (347), China (123) and Thailand (96) (millions of tons). When the sugarcane is processed for obtaining sugar and ethanol, the sugarcane bagasse is generated as a waste after sugarcane stalks are crushed (140 kg of bagasse is obtained from every ton of sugarcane processed) [20]. When sugarcane bagasse is produced, it is

stored wet in order to remove residual sugar remaining in the waste. Depending on the use it will have, sugarcane bagasse will remain wet but, for using it as a reinforcement of composites, it must be dried.

Figure 2 Sugarcane bagasse

Regarding the composition of the sugarcane bagasse, it is mainly composed by cellulose, hemicellulose, lignin, ash and wax [21] [Figure 3].

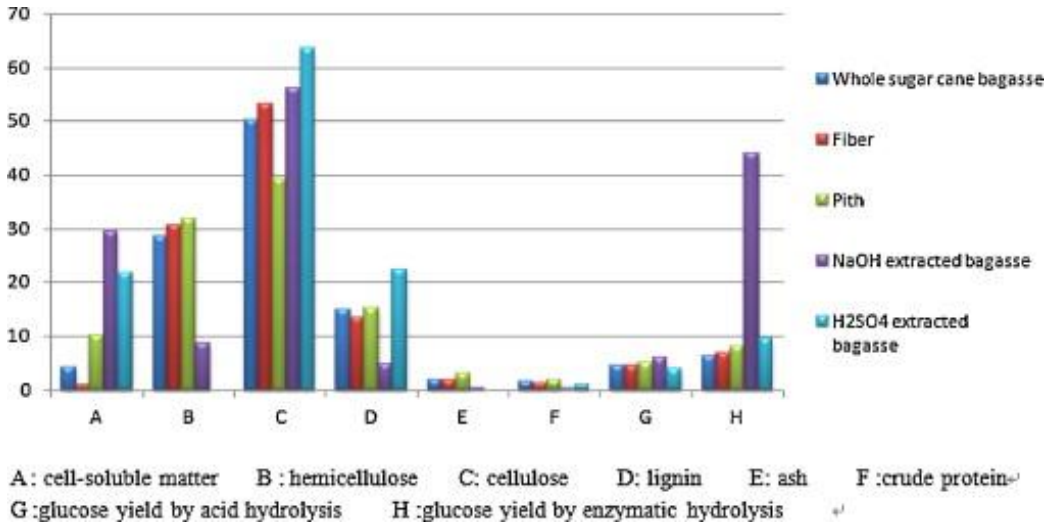

Figure 3 Chemical composition and sugar yield of sugar cane bagasse and its fractionated components (percent dry matter)

Cellulose is the main compound in natural fibers and so, in sugarcane bagasse. It is very important to consider its structure has both amorphous and crystalline parts. The properties are highly determined by the quantity of crystalline and amorphous material does the cellulose contain [22]. If we talk about the crystalline phase, hydrogen bonds can be found, and it makes the region quite stiff but this also privates the material of being too much reactive [23]. On the other hand, the amorphous phase has free -OH groups which make the region quite reactive, but the mechanical properties are poor [24]. When designing a composite reinforced with natural fibers, the ratio of the crystalline and amorphous phases is very important in order to add the coupling agent amount required for obtaining the desired mechanical properties [25].

When the desire is converting crystalline into amorphous phase so it becomes activated, the ball milling is the most common method [26]. The ball mill is a type of grinder formed by a cylindrical rotating shell which has balls inside it. It works based on the principle of impact and attrition, so the size reduction of the materials is carried on due to the impact the balls perform due to the rotation of the shell in its axis [26].

Sugarcane bagasse wasted have a huge window of applications apart from being used in the reinforcement of composites. It can also be applied mixed with gelatin, starch and agar to produce tableware packaging material [27]. Ceramic and refractory products can be produced if the sugarcane bagasse ash is mixed with Arabic gum and water [28]. Also, sugarcane bagasse ash and sugar cane straw ash can replace in a certain way cement and act as pozzolanic additive in manufacturing of concrete and ash block [29].

Comparing sugarcane bagasse fiber with other natural fibers [30] (Table 1), it is shown that sugarcane bagasse fiber does not have the best mechanical properties compared to other natural fibers but is great use is due to the high amount of fiber which can be obtained in a cheap way as a subproduct form the production of sugar.

Fibre	Density (g/cm^3)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Break (%)	Length (mm)	Diameter (µm)
Synthetic						
E-glass	2.5-2.59	2000 – 3500	70	2.5	-	-
Aramid	1.4	3000 – 3150	63.0 – 67.0	3.3 – 3.7	-	-
Carbon	1.4	4000	230.0 – 240.0	1.4 – 1.8	-	-
Natural						
Bamboo	0.6–1.1	140–800	11–32	2.5-3.7	1.5-4	25-40
Jute	1.3-1.49	320–800	8-78	1.5–1.8	1.5-200	20-200
Kenaf	1.4	223-930	14.5-53	1.5-2.7	-	-
Flax	1.4-1.5	345–2000	27.6-103	1.2–3.3	5-900	12-600
Sisal	1.33-1.5	363–700	9.0–38	2.0–7.0	900	8-200
Hemp	1.4-1.5	270-900	23.5-90	1.0-3.5	5-55	25-500
Coir	1.15-1.46	95-230	2.8–6	15-51.4	20-150	10-460
Ramie	1.0-1.55	400-1000	24.5-128	1.2-4.0	900-1200	20-80
Abaca	1.5	400-980	6.2-20	1-10	-	-
Baggase	1.25	222-290	17-27.1	1.1	10-300	10-34
Cotton	1.5 – 1.6	287 – 800	5.5 – 12.6	3.0 – 10.0	10-60	10-45

Table 1 Mechanical properties of natural fibers

1.5 Composite properties

There are multiple factors that have high impact in the way a composite will behave. They are fiber volume fraction, fiber dispersion, fiber aspect ratio, fiber orientation and interfacial adhesion.

Fiber volume fraction indicates how much fiber content has the composite. Depending of this fraction, the mechanical properties of the composite like tensile strength, impact resistance, elasticity or elongation at break in a tensile test will differ. This allows to variate the fiber quantity in order to have the desired characteristics.

Fiber dispersion refers to the way the fibers are displayed in the polymer. For good performances, a good dispersion is necessary, so the fibers are separated from each other which allows the polymeric matrix to surround them and giving as a result a homogenous composite. The homogeneous display allows the composite to have similar mechanical

performances in all parts of it, so there will not be weaker points which would allow the micromechanical deformation processes to emerge.

Fiber aspect ratio is defined as the relation between the length of the fiber and the diameter and is a critical parameter in a composite, as it can indicate the minimum value for which the maximum allowable stress can be resisted for an external load.

Fiber orientation is also important as the same orientation will allow to have improved mechanical properties. As the fibers are randomly orientated at the beginning of the processing of the composites, a progressive orientation is carried on (Figure 4).

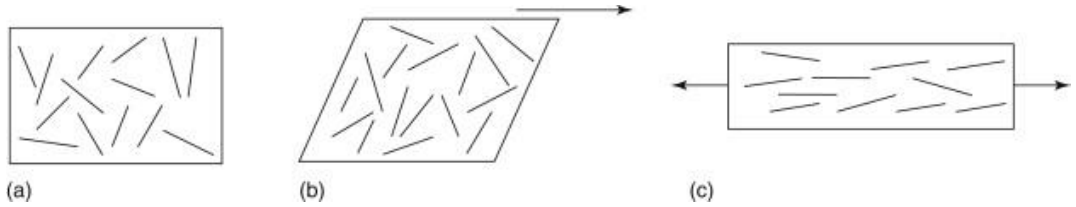

Figure 4 Orientation of individual fibers during processing: (a) initial random distribution, (b) rotation during shear flow, and (c) alignment during elongational flow.

Interfacial adhesion is also one of the most important factors in order to determine the composite properties [31]. Regarding on the chemical composition of the materials, it can be expected to find covalent bonds as intramolecular bonds and two types of intermolecular bonds: Hydrogen bonds and van der Waals forces, being the last one hugely weaker than the Hydrogen bonds [32]. Despite that, only van der Waals forces can be formed in polypropylene, and because of that, it is very likely to find debonding of larger particles or fibers from the matrix [33].

Research has been done willing to prevent the debonding from the matrix of the composites, giving rise to the appearance of the coupling agents [34]. In the case of the polypropylene and natural fiber composites, the maleated polypropylene (MAPP) is the most common coupling agent applied [35], which is a polymer grafted with maleic anhydride [36]. The use of a coupling agent enhances the mechanical properties as the interface bonding between the polymer and the natural fibers is improved due to the stress transfer amelioration [37] and the voids present in the composites are minimized [38]. MAPP contains both PP segments and grafted anhydric groups obtained from the maleic anhydride [39]. These anhydric groups can form ester bonds between them and the free -

OH groups contained in cellulose from sugarcane bagasse [40] and the PP segments are compatible and are able to embroil with the PP matrix which surrounds the natural fiber [41].

1.6 Micromechanical deformation processes

Composites reinforced with natural fibers are manufactured in order to improve the mechanical properties of the polymeric matrix. When an external load is applied on the material, micromechanical deformation processes emerge and the impact they have depends on the properties of the components and the interfacial adhesion and the structure [42]. Multiple deformation processes can appear in the composite and, as they are competitive processes, the strongest one will depend on the material properties and the stress conditions . As the natural reinforced polymers are heterogenous polymer systems, the elastic properties and stress concentration differs around the heterogeneities when the load is applied [43]. From all the micromechanical deformation processes which can be found in the composites, debonding is the most common and dominant one if there is not strong adhesion between matrix and reinforcement [44]. This phenomenon is due to weak interfacial adhesion between the polymer matrix and the reinforcement, so when the load is applied, the matrix and the filler are separated. This also includes the formation of voids which is the first step for the break of the material.

This phenomenon is very important and so, Farris investigated it in filled cross-linked elastomers, finding that almost all particles debond at large deformations being the volume increased by this phenomenon proportional to the volume fraction of the filler [45]. It is important to remark that there is not a predictive model in order to describe the way the composite suffer debonding and shear yielding, which is other type of deformation process that can emerge. The lack of the predictive model imbeds the difficulty of finding how the composite is going to respond to the external efforts.

Shear yielding is also one of the micromechanical deformation processes that can emerge in both amorphous or crystalline polymers and its effect is the slipping of large structural units. This process can lead to fiber breakage and/or fiber pull-out, both being competitive processes and having a great negative impact on the mechanical properties of the composite.

Crazing is another process highly related to yielding, defined as the formation of fissures in the polymer and indicates a localized failure in the chain network of the composite [46]. Cavitation is a micromechanical deformation process which is shown as the formation of voids as a result of the large negative hydrostatic pressure during deformation processes and it occurs before reaching the necessary stress for plastic deformation of the composite [47].

One of the main problems of studying micromechanical deformation processes is the few methods that exist in order to get reliable information about the local deformations. The measurement of volume strain as a method is based in the volume increase of the material when debonding, crazing or cavitation takes place. When these processes occur, the voids formed grow as the effort takes place. Measuring the specimen in a continuous way will show the volume increase as the deformation takes place.

Acoustic emission is one of the methods included in the Non-Destructive Evaluation (NDE) [48] and is defined as 'the class of phenomena whereby transient elastic waves are generated by the rapid release of energy from a localized source or sources within a material, or the transient wave(s) so generated [49]. Applied to composites and materials in general, acoustic emission gives information about the acoustic waves generated as a result of a microstructural changes. The parameters of the acoustic wave will be the font of information as they differ from each possible phenomenon. They are amplitude, duration, rise time, energy, counts and threshold. (Figure 5)

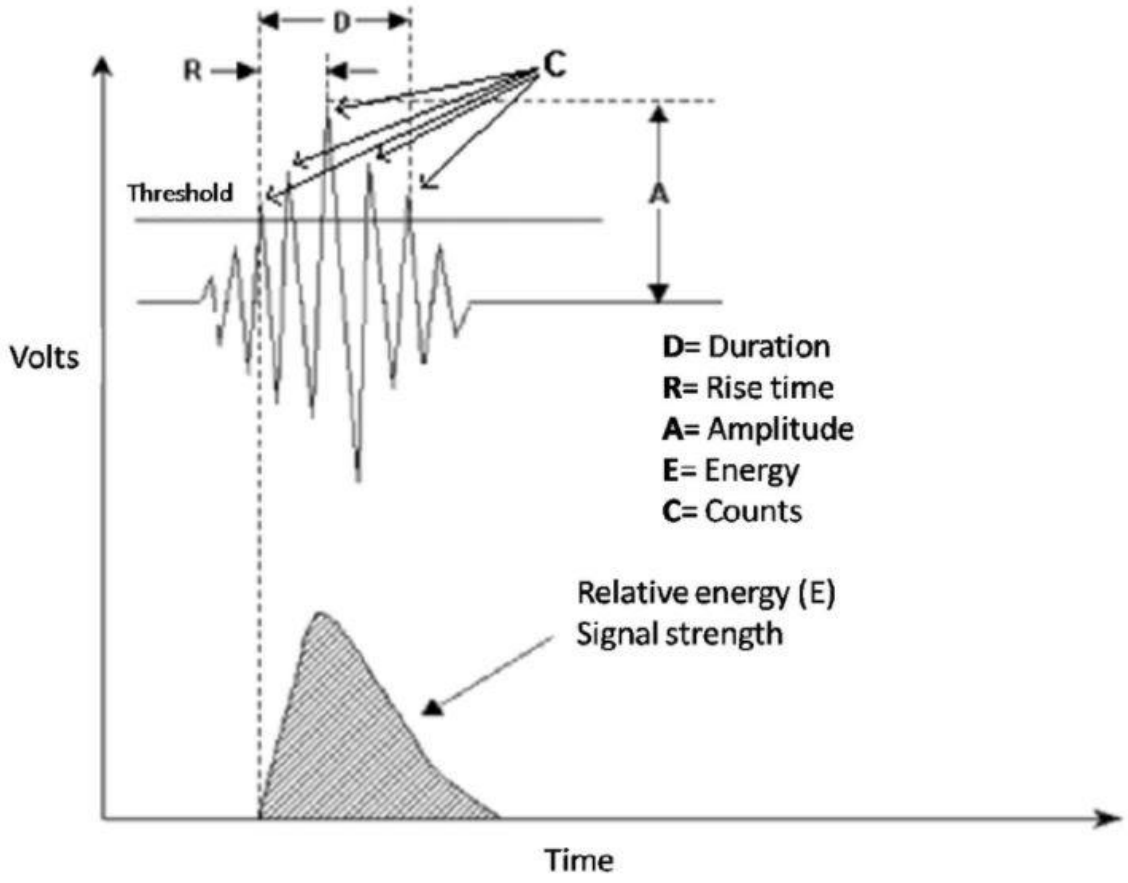

Figure 5 Parameters of acoustic emission signals

Two different types of acoustic signals can be detected using this method: burst or continuous signals. Burst signals are generally related with mechanism of short duration like fiber breakage, particle impact or fiber/matrix friction while continuous ones are related with external noise [50] (Figure 6). It is also important to say that the phenomenon mentioned previously cannot be detected with other different techniques which only detect geometrical discontinuities.

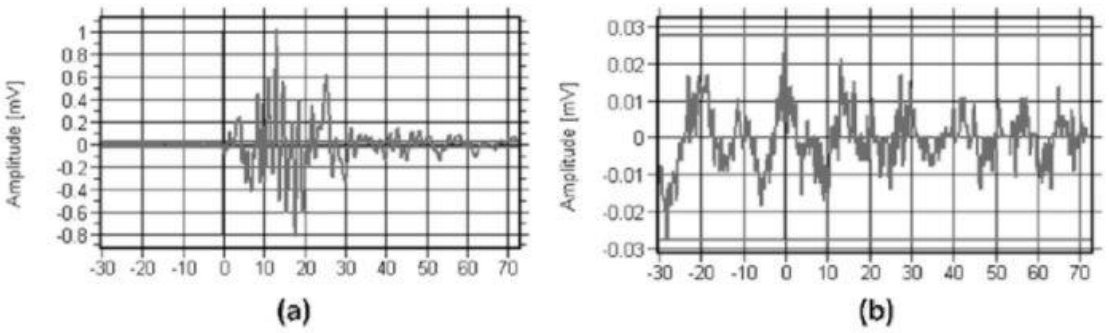

Figure 6 (a) burst signal and (b) continuous signal

Acoustic emission can be used at the same time a tensile test is being performed in order to monitor and identify the sequence of failure the composite has. As it was mentioned before, the shape of the acoustic wave will provide information about the different types of micromechanical deformation processes that can emerge (Figure 7).

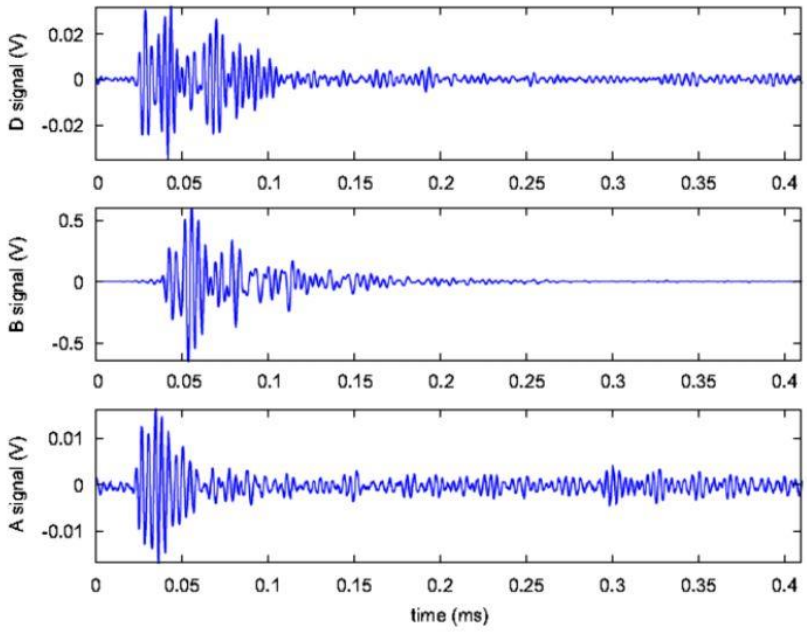

Figure 7 Matrix cracking (A signal), fibermatrix deboning (B signal) and delamination (D signal)

Applying AE testing, local deformation processes like debonding between fiber and matrix, fiber pullout and fiber fracture can be detected. Figure 8 shows, in the case A, a composite containing 16.5 wt% of fiber and no coupling agent; and in case B a composite with the same fiber content but with MAPP as a coupling agent.

Each small circle shows individual acoustic events, and the lines represent cumulative number of events vs elongation (right axis) and stress vs elongation (left axis). In the case A, there are not many signals, and they are grouped in two zones: one below 1% and other one along the rest of the test. From previous research it is known that the group below 1% is related to debonding due to this process usually appears at small deformations and stresses, while the second group could be identified as fiber fracture and/or pullout [51].

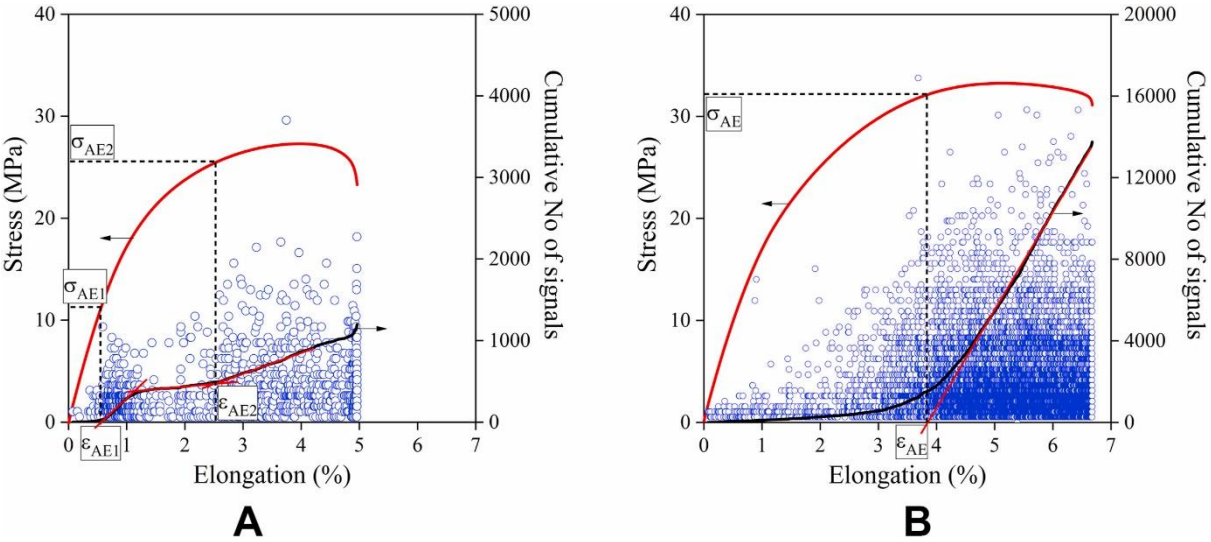

Figure 8 Results of the acoustic emission testing for samples of 16.5 wt% fiber content. A has not coupling agent. B has MAPP as coupling agent

In the case B, a notable difference can be appreciated with the addition of MAPP as a coupling agent. Number of signals have been increased hugely and the distribution is different. Most of the signals appear above 3% elongation, showing that the coupling agent has prevented many debonding events but this method does not give concrete information about what processes are emerging above 3% elongation, so other techniques are needed.

Scanning electron microscopy (SEM) is another technique which can provide useful information about the micromechanical deformation processes. Figure 9 shows two images obtained by the use of SEM: A is the 16.5 wt% fiber content composite with no coupling agent and B is the 16.5 wt% fiber content composite with MAPP as a coupling agent. Case A shows debonding as the dominant process and some fibers fractured but it can not be assured if this fracture is due to the tensile test, or it happened

when the material was homogenized. Case B shows the effect of adding MAPP as a coupling agent, as a large fiber fractured can be seen in the middle of the image. Although this, the large size of the fibers, the attrition suffered during processing and the rough surface does not allow to clearly identify the local processes emerged in the samples.

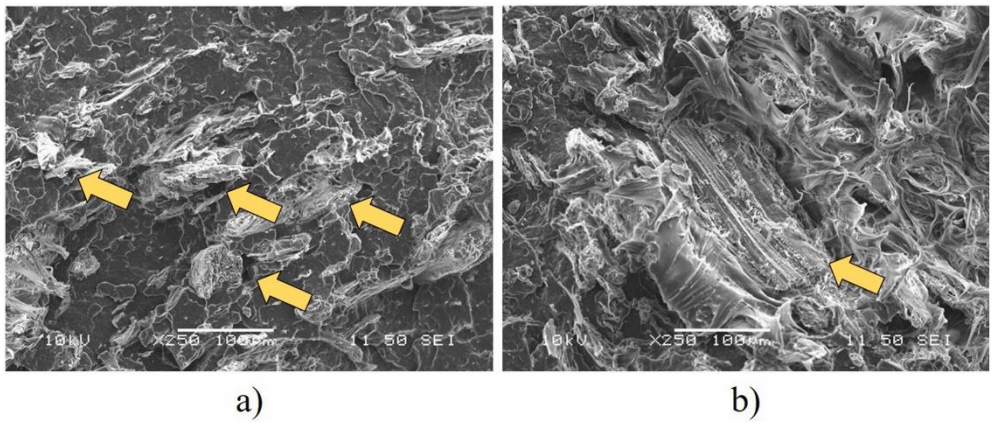

Figure 9 SEM micrographs recorded on the fracture surface of samples of 16.5 wt% fiber content. A has not coupling agent. B has MAPP as coupling agent.

1.7 Recycling

In the recent years recycling has become a very important step in every industrial process with the aim of damaging the environment the less possible and reducing the quantity of virgin raw material necessary for the final product. In the field of composites, the recycling methods can be variated depending on if the objective is to recycle the whole composite or, by other hand, separating the matrix and the reinforcement. It is very important also to know if the recycling process will affect in a negative way to the properties of the composite, as the polymer and/or the fiber could experiment degradation and mean a high downgrade in the mechanical properties as a result.

For composites formed by thermoplastic matrix and natural fiber reinforcement there are some processes for recycling. Remelting and remolding is the most common one if the aim is to recycle the composite itself. There is no separation between matrix and fiber and the process consists of the regrinding of the material for its extrusion molding. This process can lead to fiber breakage and the mechanical properties of the composite would differ in a negative way from the ones it had in the previous use. In that

case, a study needs to be performed in order to be sure that this breakage would still allow the composite to be used, depending on the mechanical requirements it need to have.

Chemical recycling can also be performed for separating matrix and reinforcement. It is performed by dissolving the matrix, but this process also leads to fiber breakage, and it has not been much studied [52]. Also, a thermal processing consisting of combustion or incineration can be performed not for getting the composite back, but for obtaining energy that can be used in the process of manufacturing new composites.

2. Experimental

2.1 Materials

For this project, composites were prepared with polypropylene used as matrix and sugarcane bagasse fiber for the reinforcement. Polypropylene was the Tipplen H 649 FH grade homopolymer, produced by the MOL Group Ltd. from Hungary. This polymer has a nominal density of 0.9 g/cm^3 and a melt flow rate of 2.5 g/10 min at 230 ºC and 2.16 kg load. Sugarcane bagasse fibers were provided by the sugar mill from Candi Baru Sugar Factory, Sidoarjo, Indonesia. For their use, the bagasse was washed with ethanol and dried. A polypropylene functionalized with maleic anhydride (MAPP) (Scona TPPP 2112 FA) supplied by Byk-Chemie GmbH, Germany, was used as coupling agent. It had a melt flow rate of 2-7 g/10 min at 190 ºC and 2.16 kg load and containing 0.9-1.2 wt% of maleic anhydride, according to the provider.

2.2 Sample preparation

Composites were prepared with polypropylene and sugarcane bagasse fiber, variating the content of fiber from 5 wt% to 30 wt% in 5 wt% steps. There were two batches prepared with these composition, one containing MAPP as a coupling agent and the other one without containing a coupling agent.

The fibers and the polymer were homogenized in a twin-screw compounder (Brabender DSK 42/7, Brabender GmbH, Duisburg, Germany) at the set temperatures of 170–180-185-190 °C, and at 40 rpm. The fibers were dried before extrusion at 105 °C for 4 h in an air circulating oven (Memmert UF450, Memmert GmbH, Schwabach, Germany). Extrusion was repeated once in order to increase homogeneity. The granulated composites were injection molded into standard (ISO 527 1A) tensile bars of 4 mm thickness using a Demag IntElect 50/330-100 machine (Demag Ergotech GmbH, Schwaig, Germany). Processing parameters were 40–170-180-185-190 °C set temperatures, 300–700 bar injection pressure, 50 bar back pressure, 50 mm/s injection speed, 25 s holding time, and 30 s cooling time. The temperature of the mold was set to 40 °C. The specimens were stored at ambient temperature (25 °C, 50% RH) for a week before further testing. From each composition, 20 samples were taken for testing and all

the rest of the specimens obtained from the extrusion molding were grinded and extruded again in order to simulate a recycling process. The extrusion molding was repeated with the same parameters and 20 samples were taken for testing and the rest ones grinded and extruded again. At the end, three extrusion molding series were performed: the first one for raw composite and the second and third ones simulating the recycling process.

2.3 Measurements

Tensile and impact testing were done. Tensile tests were performed using an Instron 5566 universal testing machine (Instron, Norwood, MA, USA) with a gauge length of 115 mm and 5 mm/min crosshead speed. For each composition with different fiber and MAPP content, 5 measurements were performed. Modulus, yield properties (yield stress and yield strain), tensile strength and elongation-at-break were derived from recorded stress vs. strain traces. Local deformation processes were followed by acoustic emission testing. Acoustic emission (AE) signals were recorded using a Sensophone AED 404 apparatus (Geréb és Társa Ltd., Budapest, Hungary). A single “a11” resonance detector with a resonance frequency of 150 kHz was attached to the center of the specimen. The threshold level of detection was set to 24 dB. Impact tests were performed following the ISO 179 standard at 23 ºC for Charpy impact strength. Samples had a 2mm notch depth and a 1 J hammer was used on 5 specimens for each composition. Fracture tests were performed with 2 specimens of each composition using a Zwick Pendulum Impact Tester HIT5.5P (Zwick Roell Group, Ulm, Germany).

3. Results and discussion

In this thesis, I aimed to investigate the effect of recycling on PP composites reinforced with sugarcane bagasse fibers, I examined the mechanical properties, also analyzed the micromechanical deformation processes that take place during deformation. The recycling was modelled by multiple injection molding steps. The results of the mechanical testing will be shown in the next section. It is important to note that the curves shown in most of the diagrams presented below are not fitted curves based on mathematical equations, they are only used to present trends and guide the eye. Furthermore, I examined the properties as a function of volume fraction of bagasse during the representation and evaluation of the results. I calculated the volume fraction from the weight and the density (1,20 g/cm^3) of sugarcane bagasse.

3.1 Mechanical properties

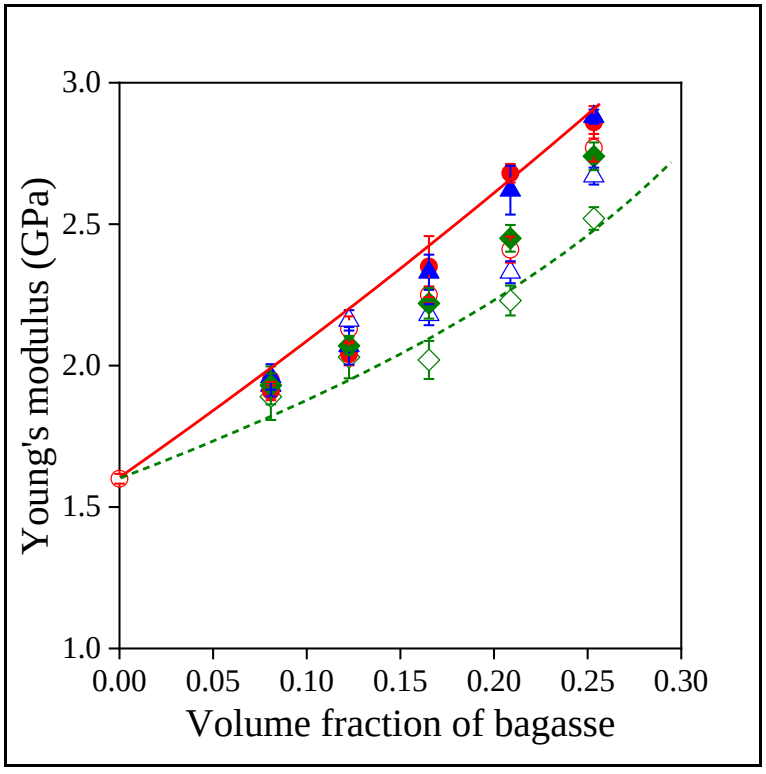

Figure 10 *Young's modulus as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

During the evaluation of the results, I plotted the values of all 6 series on a diagram for better comparison. First, I plotted the Young's modulus as a function of fiber content (V/V). The modulus is a value that characterizes the stiffness of the material, the resistance of the material to external deformation. It is an extremely important factor both in structural materials and in automotive or other applications where the product is subjected to some external load. Figure 10 shows that for each series, the modulus increased with increasing fiber content. This can be explained by the fact that the stiffness of the fiber is significantly higher than the matrix. The effect of recycling is less spectacular in Figure 10. However, we can say that the stiffness of the composites decreased after the repeated injection molding steps. One possible reason for this may be the degradation of the matrix or fiber breakage. It is well known that the attrition of natural fibers (similarly to traditional fibers such as glass or carbon) is significant during the processing. If the fibers break transversely, their aspect ratio will be smaller, so their orientation will be less significant, which may result in lower modulus.

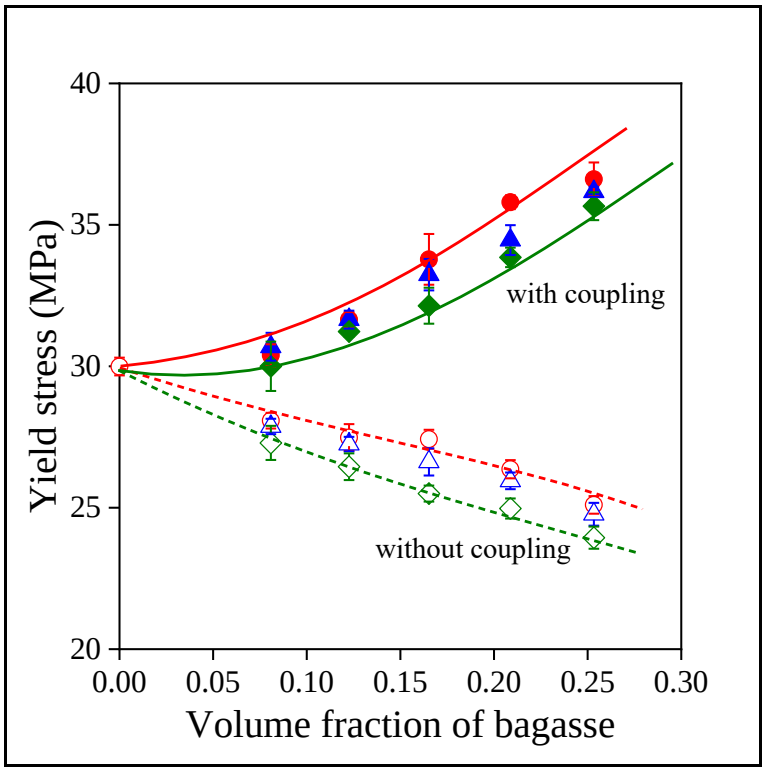

Figure 11 *Yield stress as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

Figure 11 shows the yield stress as a function of the fiber content, from which we can get information about the load-bearing capacity of the material. In the samples prepared without coupling agent (MAPP), the yield stress decreased with increasing fiber content, which is presumably due to weak interactions, which results in debonding (this assumption could be supported by the analysis of AE results later). In this case, the fibers are unable to carry the load because the continuity between the matrix and the fiber is broken. During debonding, voids form in the material, which reduces the effective cross-sectional area in the material. It is also worth noting that for the samples that are injection molded three times, the yield stress decreases to a greater extent as the fiber content increases. The reason for this is could be the same as in the case of Young's modulus: fibers with smaller aspect ratio (so they are more spherical) debond easier from the matrix. In the presence of coupling agent, the yield stress increased as a function of fiber content. The maleic anhydride group of the MAPP can form covalent bonding with the OH groups

of the bagasse fibers and the long PP chain can diffuse into the PP matrix. However, the effect of recycling is similar: multiple injection molding deteriorates the yield stress of the material.

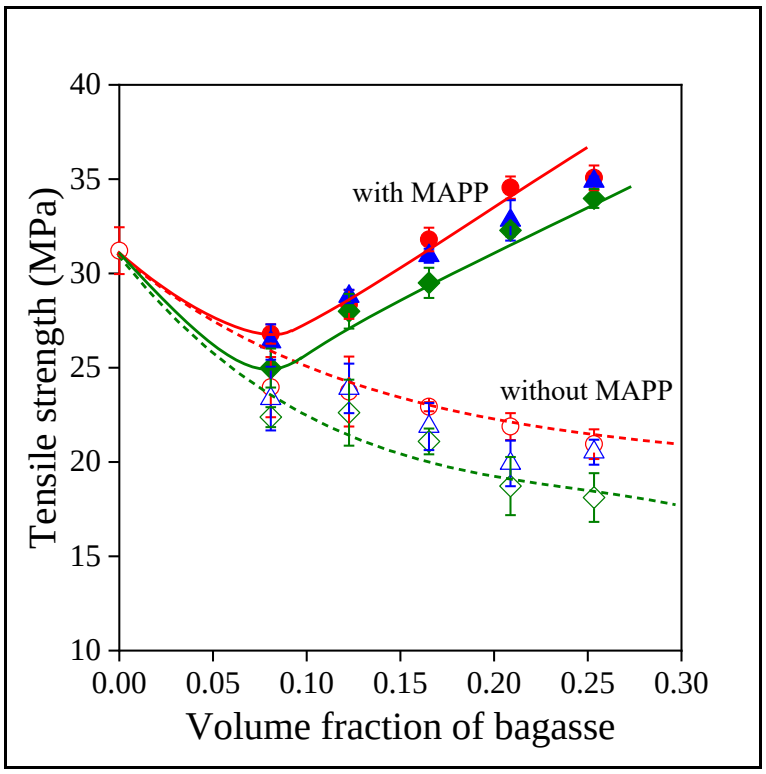

Figure 12 *Tensile strength as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

The change in tensile strength as a function of fiber content is shown in Figure 12. As in the figures shown earlier, I also indicated the tensile strength of the matrix as a reference. During the examination of the tensile strength, we can also say that due to the weak interactions, the load-bearing capacity of the fibers is not fulfilled in the samples without the coupling material, and the stress transfer does not take place. The effect of multiple processing is also somewhat more visible in the figure. In some compositions, the strength decreased by more than 10% after the third processing compared to the first injection molding. The tensile strength is determined on large deformation values, in which case the weak sports in the material also play a more significant role. Multiple

processing can lead to degradation of both the matrix and the fibers, that result in weak spots in the composites.

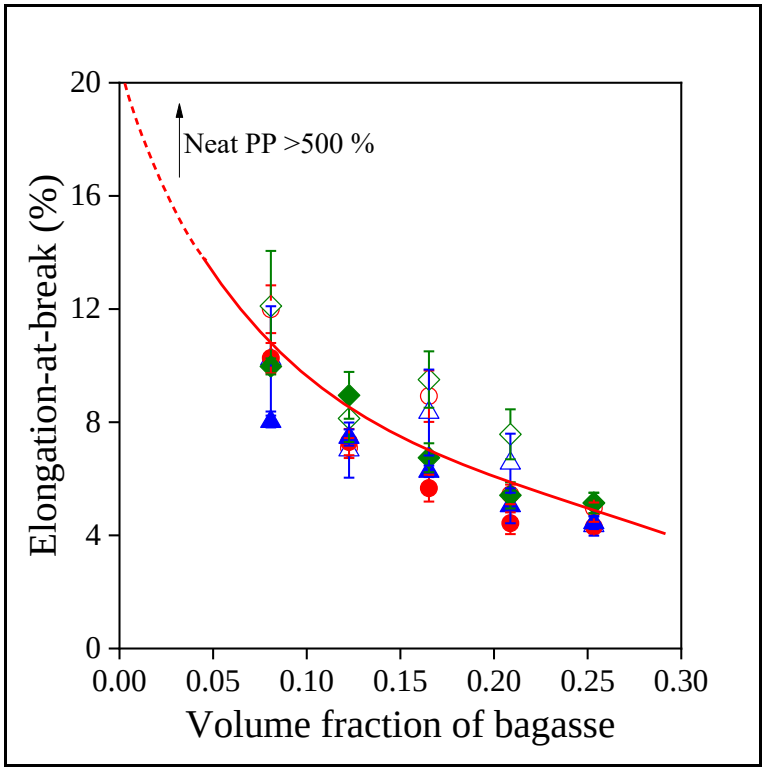

Figure 13 *Elongation at break as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

One of the major drawbacks of natural fiber reinforced composites is their low deformability. Accordingly, in this research, the elongation at break values decreased significantly with increasing fiber content (Figure 13). This is because the high fiber content inhibits the plastic deformation of the matrix. The samples without coupling agent showed somewhat higher deformation than the materials containing MAPP. The reasons are also to be found in the degree of plastic deformation, as previous research has shown that in the case of poor adhesion, debonding is followed by higher degree of plastic deformation.

Impact resistance is a measure of the ability of a material or structure there from to withstand the application of a sudden load without failure. The impact resistance of a structure is therefore a complex function of geometry, mode of loading, load application rate, environment and material properties. Since fracture resistance depends on so many parameters intro-duction of standard test methods was necessary. I also performed classical impact test on the samples. Like rigidity, impact resistance is an important feature of structural materials. The values were plotted as a function of fiber content in Figure 14. Examining the results of the composites, it can be observed that the impact resistance did not deteriorate with the addition of sugarcane bagasse fiber, moreover, minimal improvement can be observed in every cases. The samples without coupling agent show a slightly higher impact resistance, which may be due to the previously mentioned plastic deformation, which usually follows the debonding during the deformation of the composite.

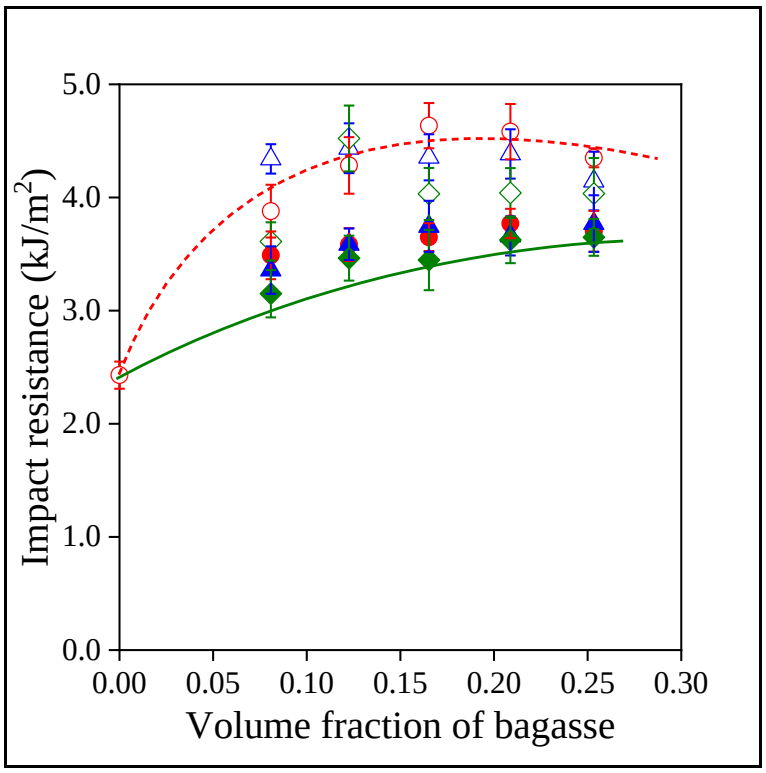

Figure 14 *Impact resistance as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

As for the effect of recycling, it can also be observed in the case of impact resistance that multiple processing steps have a detrimental effect on its value.

3.2 Local deformation processes

To understand the behavior of the natural fiber-reinforced composites, it is extremely important to monitor and determine micromechanical deformation processes. In parallel with the tensile tests, I registered the micromechanical deformation processes that took place during the deformation by acoustic emission (AE) measurement. The data registered during the measurement should be presented in one graph with the stress-strain curve (Figure 15 and Figure 16). Each event (debonding, fiber pullout of fiber fracture) has a unique sound effect, which is indicated by blue circle in the figures. Based on these, the black curve is the cumulative number of signals curve, from the shape of which we can deduce the type and amount of deformation processes that take place. The stress-strain curve is shown by red in the figures. However, deformation processes can occur not only one after the other, but also in parallel, so their determination is not an easy task.

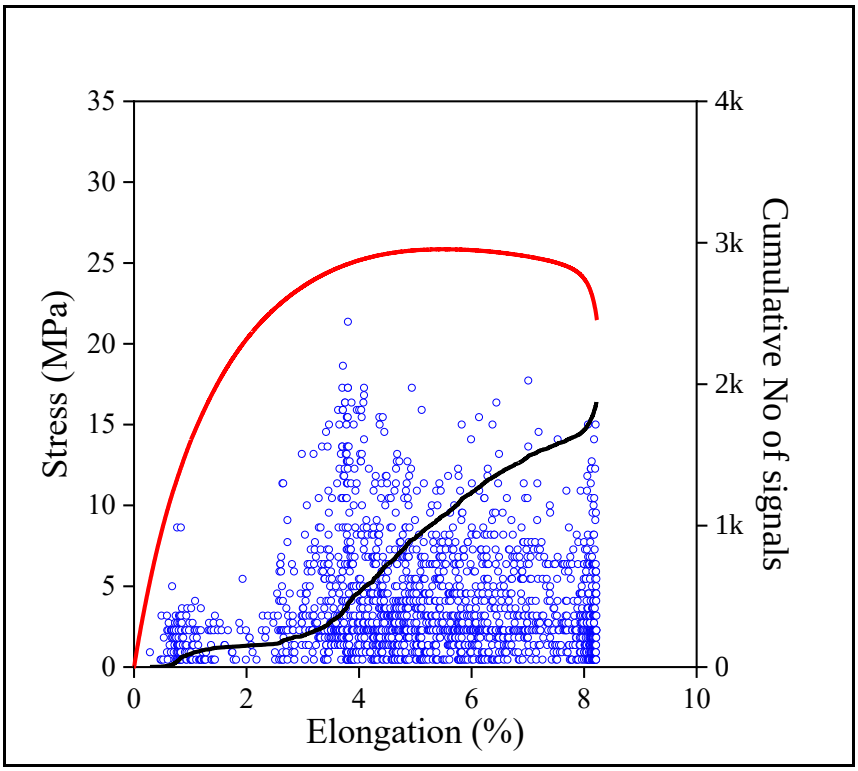

Figure 15 Results of the acoustic emission technique. 16,5 vol% sugarcane bagasse, poor adhesion, injection molded three times.

The figures (15 and 16) show the AE results of the three-times injection molded samples containing 16.5% sugarcane bagasse fiber. I obtained similar results to the figures presented in the introduction (Figure 8, 1. Introduction, 1.6 Micromechanical deformation processes): in case of poor adhesion a two-step process can be observed. The first can be related to debonding, the second to the fiber pullout and the fiber breakage. Due to strong adhesion, the fibers do not separate from the matrix, one dominant process can be detected, which is the fracture of the fibers. In this case, a longer "silent phase" can be observed in the initial section of the deformation. Since, at first glance, these results do not differ much from those of single-injection molded samples (Figure 8, 1. Introduction, 1.6 Micromechanical deformation processes), we need a more detailed analysis. Not only the shape of the cumulative number of hits curves is helpful, but also other information can be obtained from it. As it was shown in the introduction, initiation deformation (ε_{AE}) and stress (σ_{AE}) values can be determined that are characteristic of the system: they show the deformation and stress values at which certain micromechanical deformation mechanisms start.

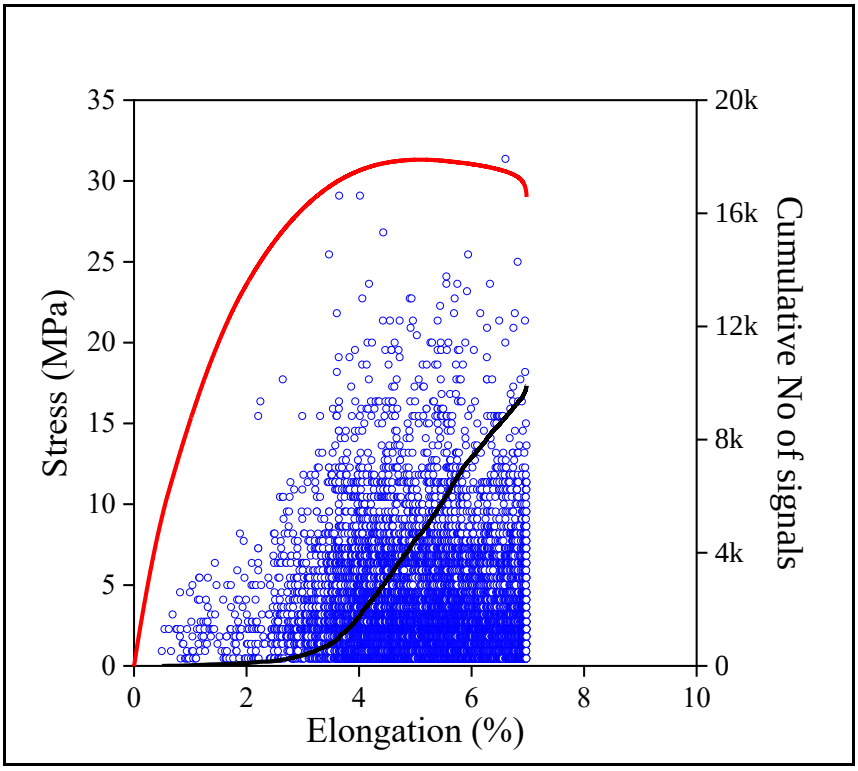

Figure 16 Results of the acoustic emission technique. 16,5 vol% sugarcane bagasse, good adhesion, injection molded three times.

In the Figure 17, I plotted the characteristic deformation values as a function of composition. Here, too, it is clear that poor adhesion results in a two-step process. The first appears at smaller deformation values, the second at larger. In the case of strong adhesion only one process can be seen, but for its initiation larger deformation is needed than for the second process in case of poor adhesion. It is important to note, that fiber fracture (strong adhesion, full symbols) starts at higher deformation in case of the three-times injection molded samples. This is pretty interesting, for further speculations also the characteristic stress values need to be checked.

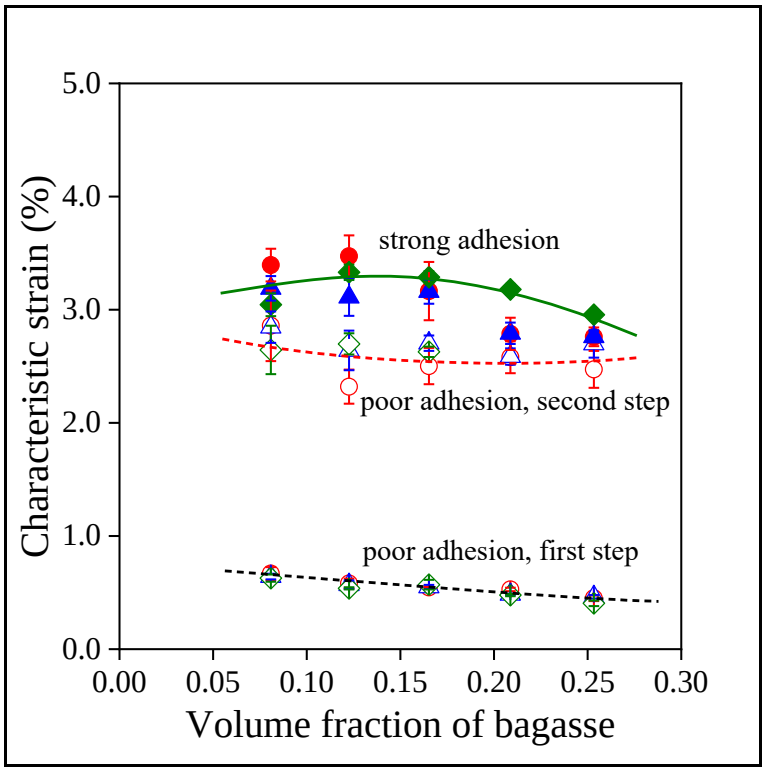

Figure 17 *Characteristic strain as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

Figure 18 shows the characteristic stress values against the fiber content. In the case of poor adhesion, the required stress for initiation does not depend on the fiber content, however, in the case of strong adhesion (using MAPP), the value increases with the fiber content. If we examine the effect of recycling, we cannot see significant differences between the composites. Based on the above mentioned, I think that also other factors should be examined to find the answer to the reasons for the different mechanical properties as a result of multiple processing. Such factors are particle characteristics, or matrix properties, like MFR of crystallinity. These factors need to be examined in the future.

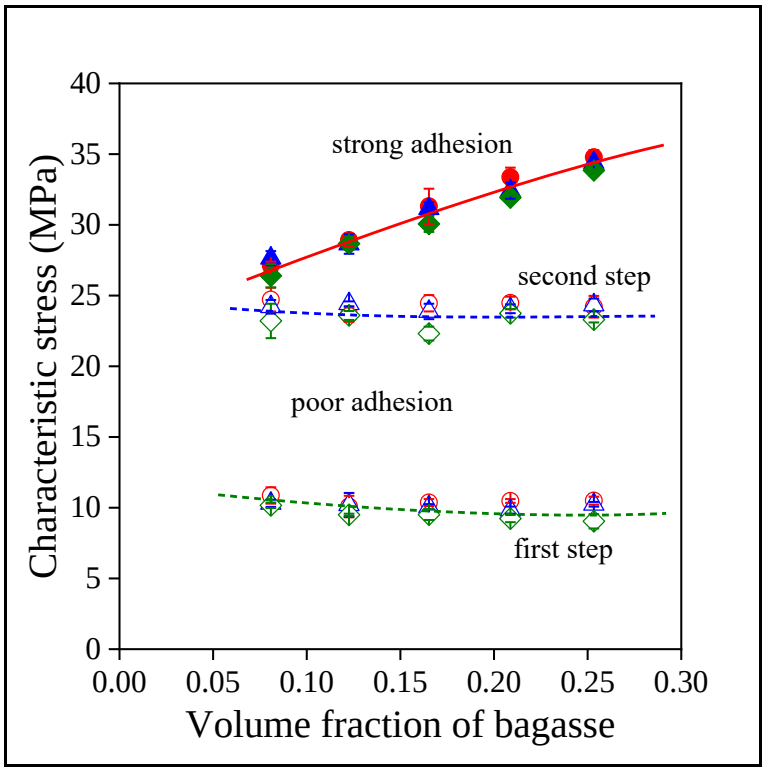

Figure 18 *Characteristic stress as a function of sugarcane bagasse content. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

3.3 Correlations

I have previously mentioned that in the case of composite materials it is very important to achieve a sufficiently high impact resistance and stiffness at the same time. In order to examine the two properties together, I plotted the impact resistance of the composites as a function of modulus (Figure 19). It can be stated that with the improvement of the modulus, the impact resistance of the PP/sugarcane bagasse fiber composites increased especially without the coupling agent. The composites showed a maximum curve without coupling agent, while they increased with the use of coupling agent. Although the impact resistance values are significantly lower than the values requires for the structural parts used in the automotive industry (~15 kJ/m^2), overall it can be said that the stiffness of the material has been significantly increased compared to the pure matrix without a significant deterioration of the impact resistance. It is also important

to emphasize that even the third injection molding step has no particular effect on the mechanical properties. Impact resistance and stiffness do not deteriorate significantly either. Sugarcane bagasse fiber reinforced composites have a good chance of being used in areas where high stiffness and strength are required, but good impact resistance is not a criterion. In addition, it is important to note that reasonable mechanical properties were achieved by using an agricultural by-product that previously could only be burned in the process of sugar production.

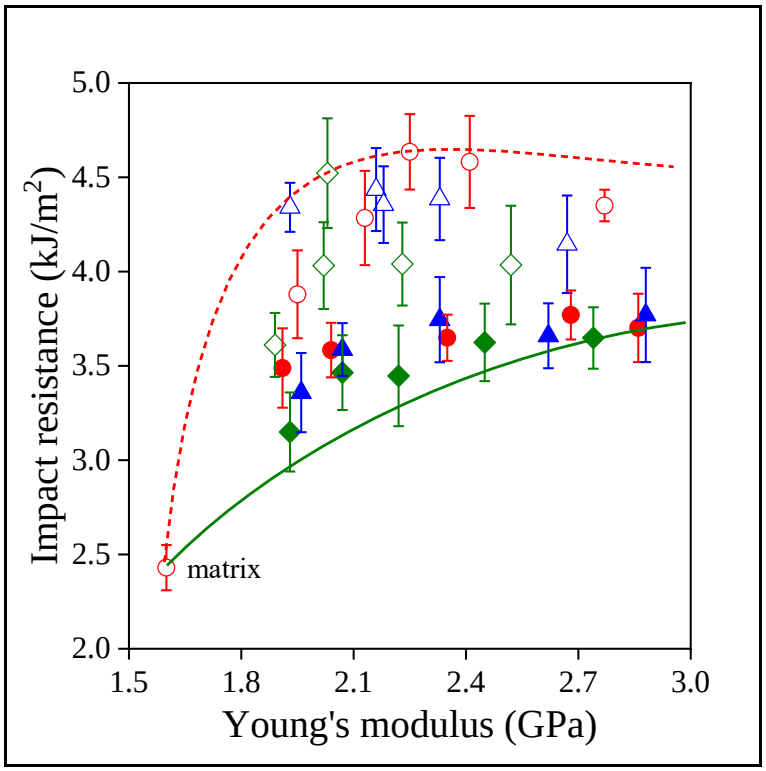

Figure 19 *Impact resistance as a function of Young's modulus. Symbols: ○ 1x injection molded, without MAPP; ● 1x injection molded, MAPP; △ 2x injection molded, without MAPP; ▲ 2x injection molded; MAPP; ◇ 3x injection molded, without MAPP; ◆ 3x injection molded, MAPP.*

4. Conclusions

Tensile and impact testing and acoustic emission testing have been performed for different composites with variating fiber content, coupling agent addition and recycling steps in order to study how the variation of the parameters affects to the mechanical properties of the material.

It has been shown that performing repeated injection molding steps for recycling the material has a negative influence in the mechanical properties of it. Stiffness, impact resistance and yield stress show decreased values with each injection molding step performed. Degradation of the matrix or fiber breakage could be the reason of the decrease of these properties, as it is known that fiber attrition of natural fibers is significant during the processing of the material, conducting into the apparition of weak spots in the composite.

The variation of the fiber content in the composite also has an impact in the mechanical properties of the material. It has been shown that elongation at break is highly decreased as the fiber content is increased. This because the high fiber content inhibits the plastic deformation of the matrix. In this case, samples without coupling agent showed higher deformation than the ones with no MAPP, due to the poor adhesion between matrix and reinforcement, and so the debonding emerged due to this. Impact resistance showed a minimal improvement with the addition of sugarcane bagasse, and the samples without MAPP showed slightly higher impact resistance than the ones with. This can be due to the debonding previously mentioned, which increases the plastic deformation of the composite. The modulus increased with increasing fiber content. This can be explained by the fact that the stiffness of the fiber is significantly higher than the matrix. Yield stress decreased with the addition of fiber content in the samples with no coupling agent probably due to the debonding resulted as weak interactions between matrix and reinforcement. In the ones with MAPP, yield stress increased as the fiber content did.

References:

[1] https://www.plasticseurope.org/application/files/9715/7129/9584/FINAL_web_version_Plastics_the_facts2019_14102019.pdf at 15/06/2021

[2] L. Shen, E. Worrell: *Plastic Recycling*, in: E. Worrell, M. A. Reuter: Handbook of Recycling, Elsevier, 2014, pp. 179-190

[3] Y. Pan, Z. Zhong: *A micromechanical model for the mechanical degradation of natural fiber reinforced composites induced by moisture absorption*. Mechanics of Materials, 85,2015, pp. 7-15

[4] A. Hemnath, G. Anbuchezhiyan, P. Nantha Kumar, N. Senthilkumar: *Tensile and flexural behaviour of rice husk and sugarcane bagasse reinforced polyester composites.* Materials Today: Proceedings (2021).

[5] https://www.britannica.com/science/polymer at 15/06/2021

[6] K.H.J. Buschow, R. Cahn, M. C. Flemings, B. Ilschner, E. J. Kramer, S. Mahajan, P. Veyssiere: *Encyclopedia of Materials Science and Technology.* 10th ed., Pergamon, 2001

[7] J.R. Collier, M.B. Lu, M.B. Fahrurrozi, B.J. Collier: *Cellulosic reinforcement in reactive composite systems.* Journal of Applied Polymer Science, 61, 1996, pp. 1423-1430

[8] A.L.Rusanov, T.N.Kolosova, R.M.Aseyeva, N.Z.Makharashvili: *Aspects of the thermal and combustible properties of halogen-containing aromatic polyamides.* Polymer Science U.S.S.R., 32 (1990), pp. 1100-1105

[9] C. Vasile: *Handbook of Polyolefins.* 2nd ed., CRC Press, 2000

[10] A.K. Bledzki, J. Gassan: *The influence of fiber-surface treatment on the mechanical properties of jute-polypropylene composites*. Composites Part A: Applied Science and Manufacturing, 28, 1997, pp. 1001-1005

[11] National Research Council (U.S.). (1992). *High-performance synthetic fibers for composites: Report of the Committee on High-Performance Synthetic Fibers for Composites*. Washington, D.C: National Academy Press, 1992.

References

[12] F.P. La Mantia, M. Morreale: *Green composites: A brief review.* Composites Part A: Applied Science and Manufacturing, 42, 2011, pp. 579-588

[13] *Natural fibers and their composites,* in: Tribology of Natural Fiber Polymer Composites, Woodhead Publishing Limited, 2008, pp. 1-58

[14] A. Shalwan, B.F. Yousif: *In state of art: mechanical and tribological behaviour of polymeric composites based on natural fibers.* Materials & Design, 48, 2013, pp. 14-24

[15] G. Faludi, Z. Link, K. Renner, J. Móczó, B. Pukánszky: *Factors determining the performance of thermoplastic polymer/wood composites; the limiting role of fiber fracture.* Materials and Design, 61, 2014. pp. 203-210

[16] A. Lotfi, H. Li, D.V. Dao, G. Prusty: *Natural fiber–reinforced composites: a review on material, manufacturing, and machinability.* Journal of Thermoplastic Composite Materials, 34, 2019, pp. 238-284

[17] D. Bhattacharyya, A. Subasinghe, N.K. Kim: *Natural fibers: Their composites and flammability characterizations.* Materials Science, 2015

[18] D. Yao, H. Li, Y. Dai, C.-H. Wang: *Impact of temperature on the activity of Fe-Ni catalysts for pyrolysis and decomposition processing of plastic waste.* Chemical Engineering Journal, 408, 2021

[19] Z. Ling, T. Wang, M. Makarem, M.S. Cintrón, H.N. Cheng, X. Kang, et al.: *Effects of ball milling on the structure of cotton cellulose.* Cellulose, 26, 2019, pp. 305-328

[20] S. Tyagi, K.-J. Lee, S. I. Mulla, N. Garg, J.-C. Chae: *Production of Bioethanol from Sugarcane Bagasse: Current Approaches and Perspectives,* in: Applied Microbiology and Bioengineering. Academic Press, 2019, pp. 21-42

[21] S. Dumitriu, E. Chornet: *Polysaccharides as support for enzyme and cell immobilization,* in: A.M. Martin: Bioconversion of Waste Materials to Industrial Products. Springer, Boston, 1998, pp. 29-102

[22] Alain Dufresne: *Cellulose-Based Composites and Nanocomposites,* in: M.N. Belgacem, A. Gandini: Monomers, Polymers and Composites from Renewable Resources, Elsevier, 2008, pp. 401-418

[23] P.V. Joseph, K. Joseph, S. Thomas: *Short sisal fiber reinforced polypropylene composites: the role of interface modification on ultimate properties.* Composite Interfaces, 2002, pp. 171-205

[24] F.H. Forziati, W.K. Stone, J.W. Rowen, W.D. Appel: *Cotton powder for infrared transmission measurements.* Journal of Research of the National Bureau of Standarde, 45, 1950, pp. 109-112

[25] L. Takacs: *Self-sustaining reactions induced by ball milling.* Progress in Materials Science, 47, 2002, pp. 355-414

[26] W. Qiu, T. Endo, T. Hirotsu: *Interfacial interactions of a novel mechanochemical composite of cellulose with maleated polypropylene.* Journal of Applied Polymer Science, 94, 2004, pp. 1326-1335

[27] D. Lokhat, I. Sewlala, D. Hooblal, B. Paul: *Beneficiation of Sugarcane Bagasse Boiler Ash via Production of Syntactic Foams and Thermal Refractory Materials*. Key Engineering Materials, 779, 2018, pp. 89–96

[28] A. Goyal, A.M. Anwar, H. Kunio, O. Hidehiko: *Properties of sugarcane bagasse ash and its potential as Cement – Pozzolana Binder.* Twelfth International Colloquium on Structural and Geotechnical Engineering, 2007, pp. 10-12

[29] R.B. Melati, A.A. Schmatz, F. Pagnocca, J. Contiero, M. Brienzo: *Sugarcane bagasse: Production, composition, properties, and feedstock potential,* in: Sugarcane: Production Systems, Uses and Economic Importance. 2017, pp. 1-38

[30] M.I. Misnon, M.M. Islam, J.A. Epaarachchi, K.T. Lau: *Textile material forms for reinforcement materials – a review.* 3rd Malaysian Postgraduate Conference, 2013

[31] D. Maldas, B.V. Kokta: *Interfacial adhesion of lignocellulosic materials in polymer composites: an overview.* Composite Interfaces, 1, 1993, pp. 87-108

[32] R.H. Petrucci, G.F. Herring, C. Bissonnette, J.D. Madura: *Química general.* Pearson Alhambra, 2011

[33] A. Bartos, J. Kócs, J. Anggono, J. Móczó, B. Pukánszky: *Effect of fiber attrition, particle characteristics and interfacial adhesion on the properties of PP/sugarcane bagasse fiber composites.* Polymer Testing, 98, 2021

[34] L. Dányádi, K. Renner, Z. Szabó, G. Nagy, J. Móczó, B. Pukánszky: *Wood flour filled PP composites: adhesion, deformation, failure.* Polymers for Advanced Technologies, 17, 2006, pp. 967-974

[35] R.G. Raj, B.V. Kokta, D. Maldas, C. Daneault: *Use of wood fibers in thermoplastics. VII. The effect of coupling agents in polyethylene–wood fiber composites.* Journal of Applied Polymer Science, 37, 1989, pp. 1089-1103

[36] P. Mutje, M.E. Vallejos, J. Girones, F. Vilaseca, A. Lopez, J.P. Lopez, J.A. Mendez: *Effect of maleated polypropylene as coupling agent for polypropylene composites reinforced with hemp strands.* Journal of Applied Polymer Science, 102, 2006, pp. 833-840

[37] C. Shuai, L. Yu, P. Feng, C. Gao, S. Peng: *Interfacial reinforcement in bioceramic/biopolymer composite bone scaffold: The role of coupling agent.* Colloids and surfaces B: Biointerfaces, 193, 2020

[38] N. Vaneewari, D.V. Saranya: *Effect of silane coupling agent on the mechanical steel of sugarcane bagasse and polypropylene composites.* Int J Biol Macromol,74, 2015, pp. 428-432

[39] M. Kazayawoko, J.J. Balatinecz, R.T. Woodhams: *Diffuse reflectance Fourier transform infrared spectra of wood fibers treated with maleated polypropylenes.* Journal of Applied Polymer Science, 66, 1998, pp. 1163-1173

[40] H.S. Kim, B.H. Lee, S.W. Choi, S. Kim, H.J. Kim: *The effect of types of maleic anhydride-grafted polypropylene (MAPP) on the interfacial adhesion properties of bio-flour-filled polypropylene composites.* Composites Part A: Applied Science and Manufacturing, 38, 2007, pp. 1473-1482

[41] K. Oksman, C. Clemons: *Mechanical properties and morphology of impact modified polypropylene–wood flour composites.* Journal of Applied Polymer Science, 67, 1998, pp. 1503-1513

[42] T. Kowalewski, A. Galeski, M. Kryzewski: Polymer Blends. *Processing, Morphology and Properties.* British Polymer Journal, 19, 1987, pp. 418-419

[43] A. J. Kinloch, R. J. Young: *Fracture Behaviour of Polymers.* British Polymer Journal, 16, 1984, pp. 114-114

[44] K. Renner, J. Móczó, B. Pukánszky: *Deformation and failure of PP composites reinforced with lignocellulosic fibers: effect of inherent strength of particles.* Composites Science and Technology, 69, 2009, pp. 1653-1659

[45] R. J. Farris: *The Influence of Vacuole Formation on the Response and Failure of Filled Elastomers*, Transactions of the Society of Rheology, 12,1968

[46] S.Q. Wang, S.W. Cheng: *Experiments-inspired molecular modeling of yielding and failure of polymer glasses under large deformation.* Polymer Glasses, 2016, pp. 395-424

[47] A. Pawlak, A. Galeski: *Cavitation during tensile drawing of annealed high density polyethylene.* Polymer, 51, 2010

[48] G. Barber: *An overview of NDT technologies and systems.* Proceedings of the Asia-Pacific Conference on NDT, 5th, Auckland, New Zealand, 2006

[49] Standard Terminology for Nondestructive Examinations, West Conshohocken, ASTM International.

[50] I.M. De Rosa, C. Santulli, F. Sarasini: *Acoustic Emission for Monitoring the Mechanical Behavior of Natural Fiber Composites: A Literature Review.* Composites Part A: Applied Science and Manufacturing, 40, 2009, pp. 1456-1469

[51] R. Várdai, M. Ferdinánd, T. Lummerstorfer, C. Pretschuh, M. Jerabek, M. Gahleitner, G. Faludi, J. Móczó, B. Pukánszky: *Effect of various organic fibers on the stiffness, strength and impact resistance of polypropylene; a comparison*, Polym. Int., 70, 2021, pp. 145-153

[52] Y. Yang, R. Boom, B. Irion, D.-J. van Heerden, P. Kuiper, H. de Wit: *Recycling of composite materials.* Chemical Engineering and Processing: Process Intensification, 51, 2012, pp. 53-68

Figures

1. https://www.pslc.ws/macrog/tact.htm at 15/06/2021

2. http://blog.pulpandpaper-technology.com/why-bagasse-should-take-over-paper-pulp-industry/ at 15/06/2021

3. Y. W. Han, E. A. Catalano, A. Ciegler: *Chemical and physical properties of sugarcane bagasse irradiated with gamma rays,* Journal of Agricultural and Food Chemistry, 31, 1983, pp. 34-38

4. *Handbook of Biopolymers and Biodegradable Plastics*, 1st ed.,William Andrew, 2012

5. C. R. Ríos-Soberanis: *Acoustic Emission Technique, an Overview as a Characterization Tool in Materials Science.* Journal of Applied Research and Technology, 9, 2011, pp. 367-379

6. C. R. Ríos-Soberanis: *Acoustic Emission Technique, an Overview as a Characterization Tool in Materials Science.* Journal of Applied Research and Technology, 9, 2011, pp. 367-379

7. S.Z. Gholizadeh, Z. Lemana, B.T.H.T. Baharudin: *A review of the application of acoustic emission technique in engineering.* Structural Engineering and Mechanics, 54, 2015, pp. 1075-1095

8. A. Bartos, J. Kócs, J. Anggono, J. Móczó, B. Pukánszky: Effect of fiber attrition, particle characteristics and interfacial adhesion on the properties of PP/sugarcane bagasse fiber composites. Polymer Testing, 98, 2021

9. A. Bartos, J. Kócs, J. Anggono, J. Móczó, B. Pukánszky: Effect of fiber attrition, particle characteristics and interfacial adhesion on the properties of PP/sugarcane bagasse fiber composites. Polymer Testing, 98, 2021

10-19. Obtained from the own data evaluation

Tables

1. M.I. Misnon, M.M. Islam, J.A. Epa Arachchi, K.T. Lau: *Textile material forms for reinforcement materials – a review.* 3rd Malaysian Postgraduate Conference, 2013