

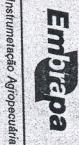
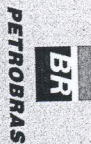
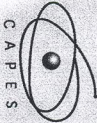
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XII INTERNATIONAL  
MACROMOLECULAR COLLOQUIUM

ISNaP<sub>2</sub>  
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7th INTERNATIONAL SYMPOSIUM ON  
NATURAL POLYMERS AND COMPOSITES

*Blangida*

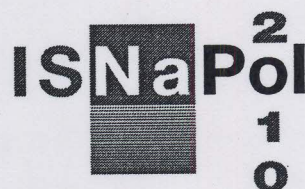
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XII INTERNACIONAL  
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7<sup>th</sup> INTERNATIONAL SYMPOSIUM ON  
NATURAL POLYMERS AND COMPOSITES

## FOREWORD

It is a great pleasure for us to welcome all the participants of the **XII International Macromolecular Colloquium and the 7<sup>th</sup> International Symposium of Natural Polymers and Composites**. We are very pleased with your contributions which are very important to the success of the Meeting. During this time, researchers will have the opportunity to initiate and enhance fruitful interactions among different institutions around the world working in the field of Polymer Science and Technology. We hope this Meeting will also offer a good opportunity to improve the research on the field of natural polymer-based materials and composites developed in Brazil.

Without your participation and specially the contribution of those presenting the 30 lectures, 34 oral sessions and 411 posters it would not be possible to organize this Meeting. We would like to acknowledge also the support from BRASKEM, CAPES, CNPq, FAPERGS, FAPESP, Petrobras and PROPESQ-UFRGS and the participation of the exhibitors dpUnion, Instrutécnica, Polimate and Reoterm.

We wish all the participants lots of interesting discussions and important stimulus for their further work and a pleasant stay in Gramado.

Organizing Committee





## TERMOPLASTIC STARCH REINFORCED WITH SUGARCANE BAGASSE FIBER

ISNaPol<sup>2</sup>  
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Thermoplastic starch is a biodegradable material that is not so strength with relation to mechanical properties. So there is important to seek reinforcements that may increase these properties without changing degradability properties or the low environmental impact of the starch. Sugarcane bagasse fiber has shown great characteristics and good results when used as load for the thermoplastic starch increasing the mechanics properties. In concentrations of 10% and 20% the sugar cane increases over 50% and 300%, respectively, the tensile strength and 7% and 21% the elongation at break when compared with the pure thermoplastic starch. Another concentration (30%) had been studied, however, in the torque rheometer tests, the torque of the 30% concentration was bigger than the 10% and 20% concentrations what can disturb the processing of the material in the extruder.

### Introduction

As the consumption of new products and materials has grown carelessly year after year, nowadays, the world is facing serious environmental problems related to pollution and lack of space for the waste. These problems are so serious that many efforts are being done to find solutions for them such as new ways of waste treatment and new materials that causes less environmental impact.

One material that is being studied exhaustively due to its biodegradation properties is the thermoplastic starch, which is a natural polymer made of, mainly, corn starch and causes almost no impact in the nature.

However, the mechanics proprieties are very low<sup>1,2</sup>, what makes hard to find an application for it and suggests that reinforcements may be done to improve its properties. On the other hand, these reinforcements can not disturb the degradability of the starch what lead us to seek natural reinforcements<sup>3,4</sup> such as sugarcane fibers.

In this work, it was studied the behaviour of sugar cane bagasse fiber as reinforcement of thermoplastic starch, evaluating mechanical, thermal, and rheometric properties of these materials.

### Experimental

#### Materials

Corn starch was kindly supported by Corn Products, and sugar cane bagasse fiber was kindly donated by

Edra Ecosistemas. Glycerol and stearic acid used were analytical grade.

#### Methods

##### *Thermogravimetric Analyses:*

As the fiber is subjected to heating in the process, it's important to understand how the heat affects the fiber and its chemistry structure. One good way to measure it is the thermogravimetric analyses, which consists in heating the material and checking the loss of the weight as the temperature rises.

Had been taken an amount about 7mg and put into the sample holder and set it on the TGA Q500, from TA Instruments. The heating rate had been adjusted to 10°C/min in two types of atmospheres, nitrogen and synthetic air, in different experiments in order to also study the behavior of the fiber in a reactive and inert atmosphere.

With the results, the graphs temperature against loss of weight of was constructed so the characteristics of the fiber could be analyzed.

##### *Torque Rheometry:*

Once the working temperature for the thermoplastic starch and for the fiber was set, the study of the behavior of the composite when submitted to shear could be done. This test was done in the Torque Rheometer, from HAAKE Thermo Electron Corporation.

For a better comprehension of the rheological behavior<sup>5</sup> the experiment was conducted in 4 different



rotations, 5, 100, 150 and 200 RPM. These rotations is important because the speed is direct related to the shear that the material is submitted and find the best rotation in the process helps to set the parameters in the extruder.

Thermoplastic starch was prepared with the following weight composition: corn starch, 60%; glycerol, 26%; water, 13.5%; stearic acid, 0.5%. Composites studied were obtained with 10, 20 and 30% of sugar cane bagasse fiber composites on basis of thermoplastic starch.

#### Tensile test:

After preparing thermoplastic starch and composites in torque rheometer, compositions were submitted to hydraulic press in order to prepare the tests samples for the tensile tests. This step consists in pressing the material placed in a mold with a hydraulic press heated in a temperature above the melt temperature of the thermoplastic starch. So the composite melts and will fulfill the mold in shape of the test sample.

As the tests samples are done, the tensile test can be done, following the ASTM D 638-90<sup>6</sup> standard, which consists in stretching the sample in proper equipment<sup>7</sup> at a rate of 5mm/min. The equipment used is an EMIC instrument and a 50kgf load cell.

It's important to say that this standard demands at least 4 test samples per formulation to have an average.

## Results and Discussion

### Thermogravimetric Analyses

Figure 1 and 2 show thermogravimetric analyses of the sugarcane bagasse fiber in synthetic air and nitrogen at heating rates of 10°C/min, respectively.

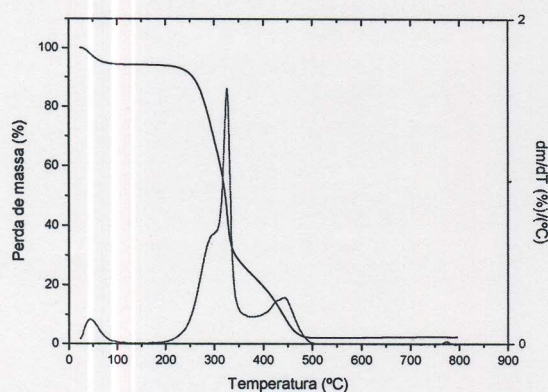


Figure 1: TG/DTG curve of the sugarcane bagasse fiber at 10°C/min in synthetic air.

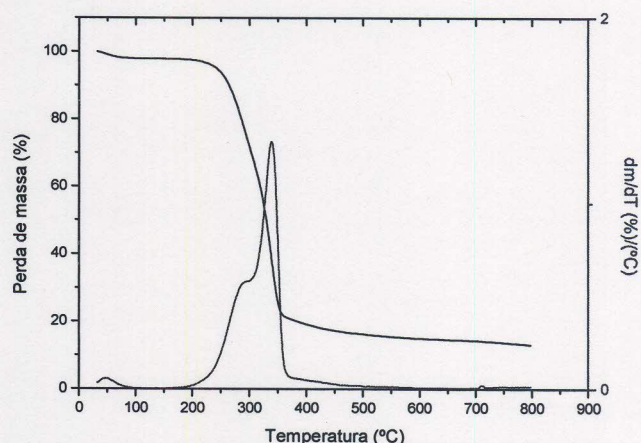


Figure 2: Curve TG/DTG of the sugarcane fiber at 10°C/min in nitrogen.

This curves permits identifying the initial temperatures of degradation (°C), volatile content(%) at 120°C and the residue(%) at 700°C for the fiber in both atmospheres. In synthetic air, the initial degradation temperature is 294,4°C, the volatile content is 5,8% and the residue is 2,3%. The same parameters in nitrogen atmosphere is 298,7°C, 5,1% and 13,5%, respectively.

### Torque Rheometry:

The figure 3 refers to the torque rheometry for the thermoplastic starch pure at different rotations.

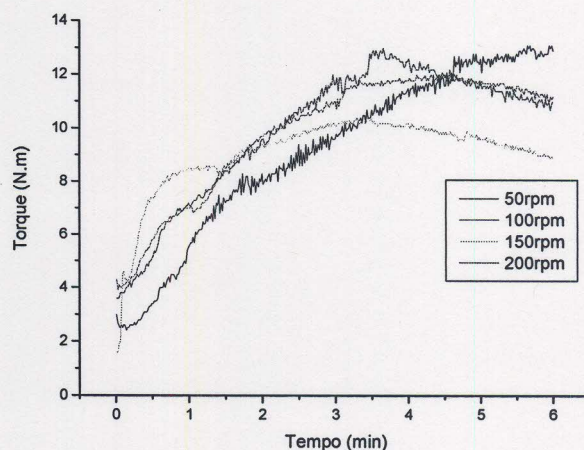


Figure 3: Torque curve against time for different rotations.

The test was made in 6 minutes because the material was almost already homogeny in this time, and in a longer time, such as 10min, the material presented a change of color perhaps caused by degradation. And the rotation chosen for the rest of the tests was the 200rpm because was the rotation that shown a better mixture. The chosen temperature for the process was 150°C because is very close to the melt



temperature of the starch, it is above starch gelatinization temperature and under its thermal degradation temperature. It's expected a decrease of the torque after the loading, raveling a pseudoplastic behavior, because is easier to process, however, if the torque do not decrease, showing a dilatant behavior, the polymer may face difficulty in other steps of the processing. With the curves obtained in the rheometer, the best composition and the best parameters could be obtained.

As these parameters were chosen, the fiber could be added and the rheometer test was realized. The figure 4 represents the torque against time for the composite at different concentration of the fiber.

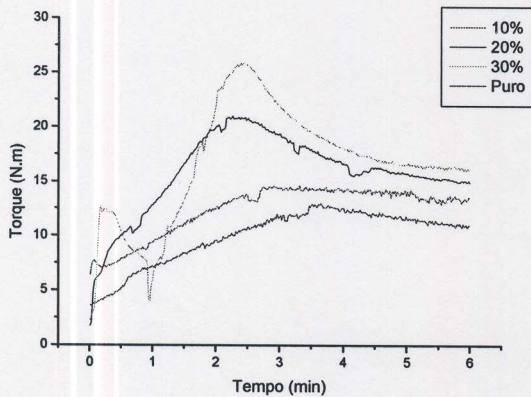


Figure 4: Torque against time for different compositions of the studied materials.

It's important to say that even though the 30% concentration had been tested, the volume of fiber is so high that is difficult to put the materials inside the mixture chamber and makes unviable to process this composition in large scale. Besides this fact, the torque, which is proportional to the concentration of the fiber, is bigger than the 10% or 20% concentrations.

*Tensile Test:*

Tables 1 to 3 show statistic analysis of the tensile tests for the composites following the ASTM D 638-90 standard.

Table 1: Statistic analyses of the tensile test results for the pure thermoplastic starch.

Sample test number	Elasticity Modulus, (MPa)	Tensile Strength, (MPa)	Tensile at break, (MPa)	Elongation at break, (%)
1	2,68	0,49	0,46	33,8
2	3,59	0,56	0,54	28,89
3	3,09	0,39	0,37	25,29
4	2,85	0,43	0,42	29,15
<b>Average</b>	3,05	0,47	0,45	29,28
<b>Standard Deviation</b>	0,40	0,07	0,07	3,49
<b>Average Standard Error</b>	0,20	0,04	0,04	1,74

It's able to see the average values for the pure thermoplastic test samples. These values are important to compare with the other composites.

Table 2: Statistic analyses of the tensile test results for the 10% fiber concentration composite.

Sample test number	Elasticity Modulus, (MPa)	Tensile Strength, (MPa)	Tensile at break, (MPa)	Elongation at break, (%)
1	5,33	0,52	0,51	14,97
2	4,16	0,83	0,81	41,77
3	4,82	0,86	0,84	30,02
4	4,32	0,79	0,78	38,55
<b>Average</b>	4,66	0,75	0,74	31,33
<b>Standard Deviation</b>	0,53	0,16	0,15	11,98
<b>Average Standard Error</b>	0,26	0,08	0,08	5,99

The results show an increase of the values, what suggests that the fiber is working as reinforcement for the starch.



Table 3: Statistic analyses of the tensile test results for the 20% fiber concentration composite.

Sample test number	Elasticity Modulus, (MPa)	Tensile Strength, (MPa)	Tensile at break, (MPa)	Elongation at break, (%)
1	6,73	0,89	0,87	33,29
2	8,27	1,11	1,07	38,98
3	12,02	1,344	1,32	32,15
4	10,76	1,25	1,19	38,31
Average	9,44	1,15	1,11	35,68
Standard Deviation	2,39	0,20	0,19	3,46
Average Standard Error	1,19	0,10	0,09	1,73

The values in the table 3 represent an important increase in the mechanics properties of the thermoplastic starch, indicating that the properties rise with the increment of more fiber.

### Conclusions

Composites of sugarcane bagasse fiber/thermoplastic starch (TPS) showed increase of tensile strength, elongation at rupture and elasticity modulus comparing with pure TPS, and these fibers are working well as reinforcement for the thermoplastic starch. However, the processing of the composite may be done with care, because the starch is sensitive to weather variations and degraded vary easy with the temperature. The window processing temperature is small, and the shear imposed to the polymer may be controlled as well to avoid degradation.

Also, it's important to remember that the fiber may interfere in the extrusion and injection process. So, the concentration of the fiber must be controlled following the equipments parameters.

### Acknowledgements

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