67º congresso 2012 abm
67th abm international congress
ISSN 1516-392X

THERMAL CHARACTERIZATION OF JUTE FIBERS BY TGA/DTG AND DSC[1]

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Abstract
Several natural fibers are increasingly being considered as viable alternatives to substitute glass fiber in polymer composites reinforcement. In practice, lignocellulosic fibers extracted from plants have shown a real potential for this substitution. Their comparative advantages are lower density an cost as well as renewability, biodegradability, recyclability and Neutrality with respect to CO_2 emission, which is responsible for global warming. By contrast, the thermal resistance of the lignocellulosic fibers is restricted and may affect their application in engineering composites. Jute fibers are worldwide used in many single items and is now considered for composite reinforcement. The mechanical properties of the jute fiber has been extensively investigated but only limited works have been devoted to its thermal characterization. The present work investigated these characterization in terms of thermo-gravimetric analysis TGA/DTG and differential calorimetry, DSC. It was found that the jute fiber starts deteriorating around 150ºC and undergoes total degradation at 500ºC.
Key words: Jute fibers; Thermal analysis; Thermal decomposition.

CARACTERIZAÇÃO TÉRMICA DE FIBRAS DE JUTA POR TGA / DTG E DSC

Resumo
Várias fibras naturais estão sendo cada vez mais consideradas como alternativas viáveis para substituir as fibras de vidro. Na prática, as fibras lignocelulósicas extraídas de plantas têm mostrado um potencial real para esta substituição. Suas vantagens comparativas são: um custo menor, menor densidade, bem como renovabilidade, biodegradabilidade, reciclagem e neutralidade em relação à emissão de CO_2, que é responsável pelo aquecimento global. Em contraste, a resistência térmica das fibras lignocelulósicas é restringida e pode afetar a sua aplicação em compósitos de engenharia. Fibras de juta são mundialmente usadas em muitos itens únicos e agora também esta sendo considerada como reforço para compósitos. As propriedades mecânicas da fibra de juta têm sido extensivamente investigadas, mas poucos estudos têm sido dedicados à sua caracterização térmica. O presente trabalho investigou esta caracterização em termos de TGA análise termo-gravimétrica/ DTG e calorimetria diferencial, DSC. Verificou-se que a fibra de juta começa a deteriorar-se em torno de 150°C e sofre degradação total a 500ºC.
Palavras-chave: Fibras de juta; Análise térmica; Decomposição térmica.

[1] *Technical contribution to 67th ABM International Congress, July, 31th to August 3rd, 2012, Rio de Janeiro, RJ, Brazil.*
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ISSN 1516-392X

1 INTRODUCTION

Some measures are currently being taken in order to benefit the environment, such as reducing energy consumption, the promotion of clean systems, the control of CO_2 emissions and the use of natural materials.[1] In particular, the substitution of synthetic materials similar nature also contributes to other environmental protection measures.[2] A relevant example is the use of natural fiber extracted from vegetables rich in cellulose, which are called lignocellulosic fibers.[3] Because of this new global trend, many studies have been conducted for the use of natural fiber composites in industries such as construction and automotive. The advantages of these fibers over synthetic ones also include ecological and social aspects, because of improving recyclability, biodegradability and improved quality of life for residents of regions where it is made the cultivation of this plant.[4,5]
Among the lignocellulosic natural fibers that have great potential for application, highlight the jute (Corchorus capsularis), grown in tropical and humid climates in the northern region of the country, your plant produces a high strength fiber, which when mixed with polymers, can give rise to products with lower density, and may have great applications. The jute fiber has a low cost, abundance, and provide flexibility, isolation, a good set of properties, such as high specific strength and module.[6]
The lignocellulosic natural fiber jute has been extensively investigated in several studies, such as its use in the manufacture of polymer composites reinforced by it.[7-9] In addition to not cause problems to the environment, the composites reinforced with this fiber type residual can replace wood products, bringing several advantages, such as avoiding deforestation. In addition, these polymer composites can also be used in building materials and auto parts.[10] However, in order to have quality materials derived from jute fiber is necessary to study properties of this fiber has when exposed to various environments.[11,12] To this end, this study aims to evaluate the behavior of lignocellulosic natural fiber jute at different temperatures. The thermogravimetric behavior of jute fiber was investigated by thermogravimetric analysis (TG) and its derivative (DTG), as well as differential scanning calorimetry (DSC).

2 EXPERIMENTAL PROCEDURE

The jute fibers investigated in this study were supplied by the Brazilian firm Sisalsul. Figure 1 illustrates a typical jute plant fibers extracted from the stem.

Figure 1. (a) Typical of jute plant, (b) fibers extracted from the stem of the plant.

ISSN 1516-392X

The thermal stability of the fibers was evaluated by thermogravimetric analysis (TGA) and differential thermal (DTA) with the objective of measuring the mass variation of a sample as a function of temperature. It was also made a differential scanning calorimetry (DSC).
For the implementation of the analysis, the jute fiber was macerated to produce particles of around 1 mm. The equipment was a TA Instruments SDT Systems 2960, shown in Figure 2a.The maximum temperature of the tests was 650°C and three repetitions were made. The parameters used in the analysis of jute fiber were: heating rate of 10°C/min, starting from room temperature to 650°C in an oxygen atmosphere.

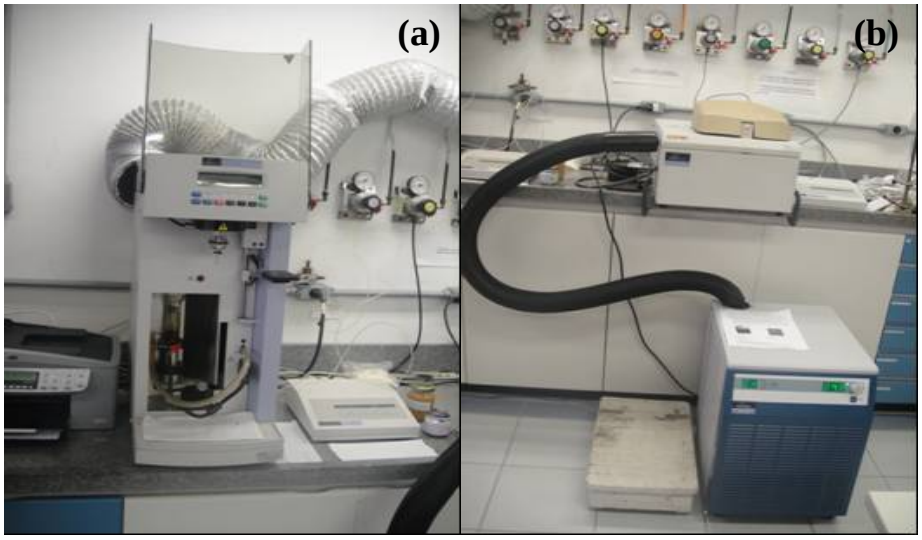

Figure 2. Thermal analysis equipments: (a) TGA/DTG, and (b) DSC.

DSC tests were also conducted on samples of macerated fibers of jute, using a TA equipment. Figure 2b illustrates DSC model 2910 Laboratory of Advanced Materials (LAMAV/ UENF) used in this work. The test parameters used for the test were as follows: the maximum temperature of the tests ranged being -10°C and 190°C. Heating rate of 10°C/min.

3 RESULTS AND DISCUSSION

The thermal stability of jute fiber is measured by weight loss by TGA performed with increasing temperature is presented in Figure 3. In the Figure, it should be noted, that after a small initial drop in weight from 25°C to 80°C, this decrease is usually attributed to the release of water related to the moisture absorbed on the surface of a hydrophilic lignocellulosic structure.[13]
After the first step constant, there is a large decrease of weight in the approximate temperature then occurs until about 301,87°C under an atmosphere of O_2. This sharp decrease can be associated with decomposition of jute fiber structure due to rupture of macromolecular chains.[13] With increasing temperature, a constant in the second step of TGA curves (Figure 3), there is even a limit of 687°C set for analysis. The intercept of the horizontal extension of the first constant stage with the straight line, at the inflexion point, corresponding to the sharp decrease gives the onset of the structure decomposition. As shown in Figure 3, this onset occurs at the relatively close temperatures of 302°C.
Taking into account the variation of weight loss with the temperature in the TGA

ISSN 1516-392X

curves, an approximately 9% smooth loss occurred up to 200°C. Then a greater loss of about 69% occurred from 200°C to 400°C. Afterwards, a continuous loss around 23% takes place from 400 until 687°C, when less than 21% of mass remained apparently unchanged. This is possibly due to ashes resulting from oxidation reactions. The temperature dependence of the DTG, i.e., the derivative of the TGA curves, provided additional information on possible events responsible for the distinct stages of mass loss in the jute fibers.

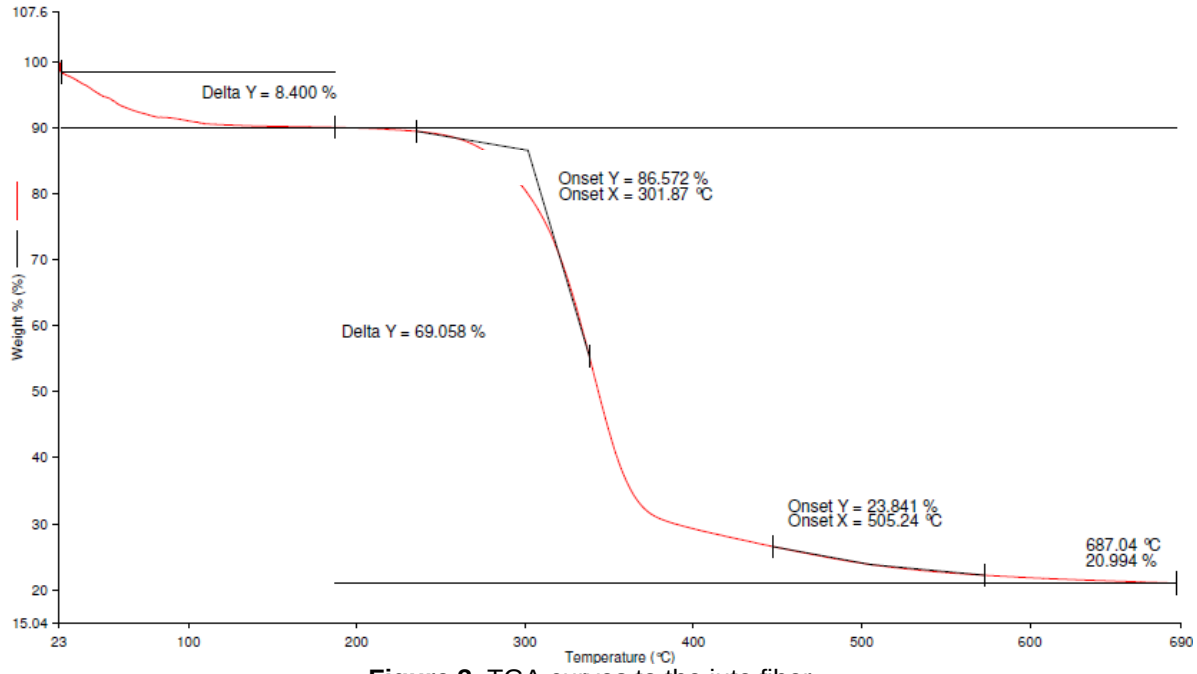

Figure 3. TGA curves to the jute fiber.

TGA curves of Figure 3 to show the important levels of weight loss associated with events occurring to the jute fiber thermal degradation. The small first drop in weight due to the loss of absorbed humidity is relatively close, 9%. This result together with the close decomposition onset temperatures (Figure 3), indicates that the initial release of humidity is not affected by the existent atmosphere. By contrast, the sensible differences in weight loss during the structure decomposition, 69%, together with corresponding significant differences between the decomposition temperatures, Figure 3, indicate an effect of the atmosphere. This will be better understood in conjunction with the DTG curves.

Figure 4 shows the DTG curves for the jute fiber. In fact the carbon and nitrogen that constitute the lignocellulosic structure of the jute fiber are expected to react with the oxygen releasing CO, CO_2 and H_2O. Additionally, other free elements in the structure, like Ca, K, Na, Fe, etc, may also react with the O_2 to form oxides associated with inert ashes.

A feature in Figure 5 is the existence of small peaks to 100°C for O_2 atmosphere. This is apparently an indication of another effective loss of weight taking place at temperatures higher than that associated with the first peaks. In fact, the existence of two peaks in the DTG curve of a natural fiber and its polymer composite was reported[13,14] at similar intervals of temperature. The lower temperature peak was attributed to the decomposition of more rigid molecular segments while the higher temperature peak would be related to the decomposition of more flexible segments.[14]

ISSN 1516-392X

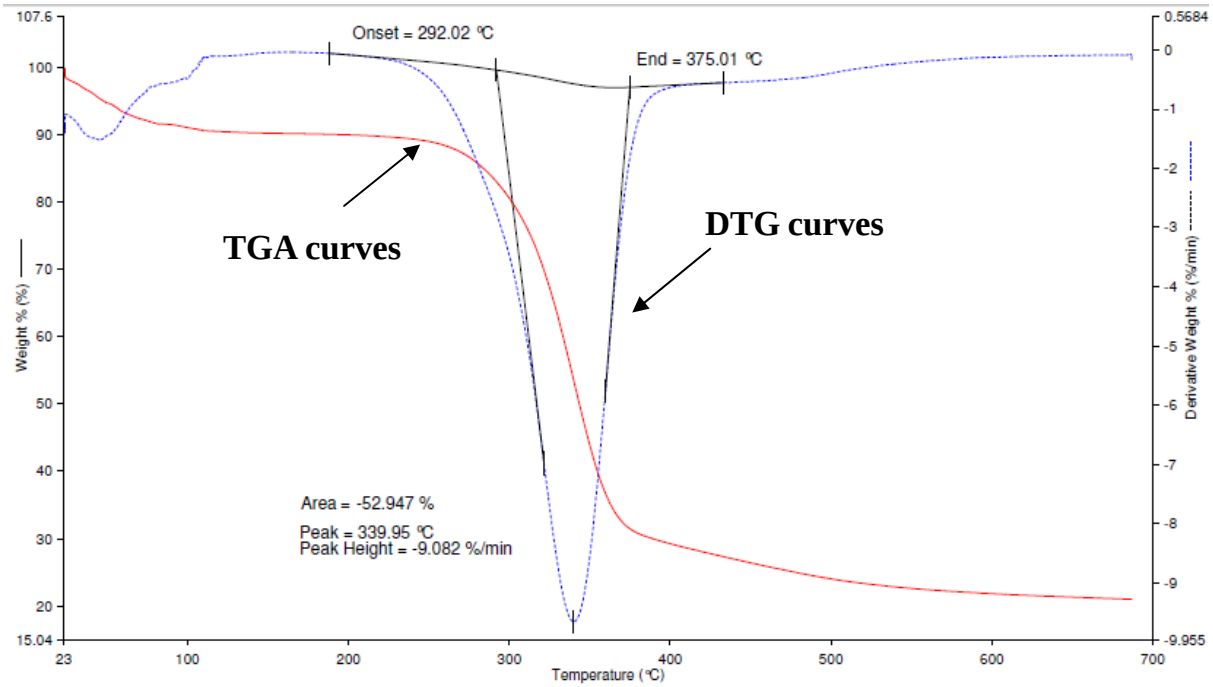

Figure 4. TGA curves for the jute fiber.

Figure 5 shows the DSC curve for the jute fiber. In this Figure an exothermic event occurs in the interval of temperature from approximately 112°C to 159°C with a peak at 125ºC. The exothermic heat released in this event was 63 J/ g. Its macromolecular chain structure begins the thermal degradation process around 100°C and, under an oxidizing atmosphere, leaves a solid residue of less than 10% of its total weight. These characteristics establish the limits for engineering applications of the jute fiber, especially as an insulating material. Apparently, peaks found for lignocellulosic fibers around this temperature correspond to the loss of constitution water of the cellulose structure

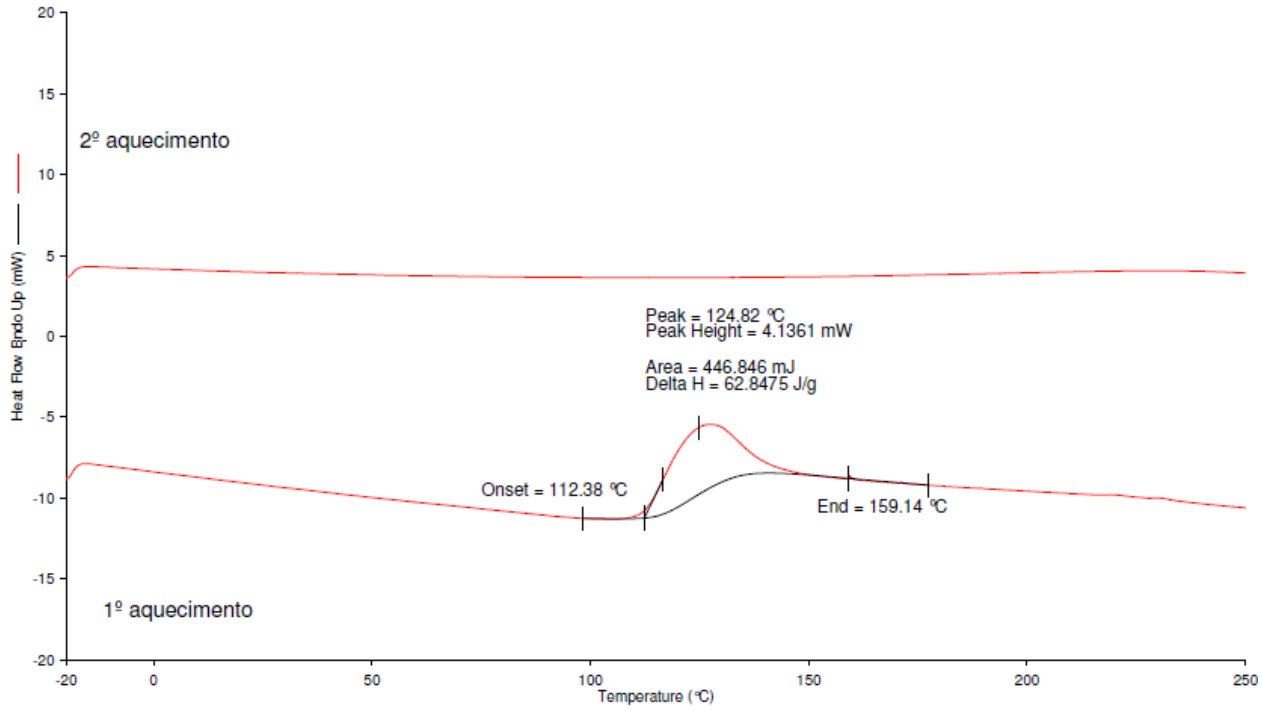

Figure 5. The DSC curve for the jute fiber.

ISSN 1516-392X

4 CONCLUSIONS

- The thermal behavior of a jute fiber analyzed by TGA, DTG and DSC revealed evidences of water loss and structural decomposition. Up to 80ºC approximately 10% of humidity is released while the lignocellulosic water of hydration is lost around 100°C.
- A structural decomposition displayed maximum weight loss associated with clear peaks at 339,95ºC on oxygen atmosphere.
- A constant amount around 10% of stable solid residue is formed as inert ashes from 400°C up to 687°C. This residue is composed of oxides from trace metals such as Ca, K, Na and Fe as contents of the jute fiber lignocellulosic structure.

Acknowledgements

The authors thank the support to this investigation by the Brazilian agencies: CNPq, Capes, Faperj and Tecnorte/Fenorte.

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