MECHANICAL CHARACTERISATION OF SISAL FIBRES FOR REINFORCING OF COMPOSITE MATERIALS WITH SEVERAL DIFFERENT SURFACE TREATMENTS

C. Romão[1], P. Vieira[2], J.L. Esteves[3]

[2] INEGI – Institute of Mechanical Engineering and Industrial Management, Rua do Barroco, 174 , 4465-591 Leça do Balio, Portugal
[1] ESTV/IPV - Department of Mechanical Engineering and Industrial Management, Superior School of Technology of Viseu, Institute Polytechnic of Viseu, Campus Politécnico de Repeses, 3504-51Viseu, Portugal
[3] FEUP – Department of Mechanical Engineering and Industrial Management, Faculty of Engineering of University of Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal

ABSTRACT

The work described in this paper refers to the mechanical characterisation of sisal natural fibres composite materials.
Were made experiences with different surface treatments with the purpose of increasing the adhesion between the fibres and the matrix, and consequently to improve mechanical behaviour of the composite material.
A brief description of the production and test setups of the composite materials is made.

1. INTRODUTION

Today the search for new, recyclable and renewable materials is leading the researchers in new ways. Natural products are emerging and some research is starting in this matter. The work presented here shows the utilisation of sisal fibres with different surface treatments with the purpose of increasing the adhesion between the fibres and the matrix, and consequently to improve mechanical behaviour of the composite material [1-2]. The treatment used is called mercerization, and is described below. Before the treatment application the natural fibres were cleaned in order to remove contaminating agents. Some plates were made using sisal fibres as reinforcement and an epoxy resin as matrix.

2. SURFACE TREATMENT

A fibre surface treatment has been done to increase the fibre/matrix adhesion. This treatment (mercerizing) is made in some steps. First, the fibres were immersed in a bath of Sodium Hydroxide solution (NaOH), prepared with distilled water. During this process the bath was stirred continuously using a mechanical agitator. Finished the immersion stage the solution presented a yellow colour, because of the substances removed from the fibres. Next, the fibres were washed several times with distilled water, until the water pH came to neutral. To dry the fibres we left them 5 days at ambient temperature, and then exposed six hours at 60º C in an oven. [3-7]
The main objective of the treatment is the fibre superficial cleaning by the remove of some agents (grass, silica, etc.) that difficult the chemical reactions between the fibres and the matrix. Additionally, can remove the lignin and the hemicelluloses, responsible for some degradation mechanisms.
This treatment improves the interface fibre/matrix adhesion by increasing the chemical compatibility (exposing the hydroxyl groups of the fibres) and the mechanical anchorage.
Four different treatments of mercerization were made, with different volume percentage of Sodium Hydroxide (NaOH) and bath time immersion:

- 4% (NaOH) in volume and 1 hour bath immersion
- 4% (NaOH) in volume and 2 hour bath immersion
- 8% (NaOH) in volume and 1 hour bath immersion
- 8% (NaOH) in volume and 2 hour bath immersion

All treatments were made with a bath temperature of 20ºC.

3. FIBRE PREPARATION

The first step for the application of the sisal fibres was the superficial cleaning; because when they were acquired contained some contaminating agents on the surface. After this cleaning process and before the surface treatments (when applied), the fibres were cutted to pieces of approximately 30 mm length, with the aim of prepare the fibres for the production of an aleatory mat.

For the mat production the fibres (previously dried on an oven) were placed aleatory in a mould, afterwards the mould was closed and submitted to some pressing, resorting to a press. After 10 minutes the mat produced was removed. Fig. 1 shows the mould employed and the mat of sisal fibres.

Fig 1. The natural fibre mat

4. MANUFATURING A COMPOSITE PLATE

To produce the sisal composites plates, was used the epoxy resin Reapox WOOD RX8 from REA Industries with the following characteristics:

Tensile Strength (MPa) = 49,98; Modulus (GPa) = 2,91; Density (g/cm^3) = 1,13.

To manufacture the composite plates, with aleatory sisal fibre reinforcement, was used the compression moulding technique. The mould used, made of steel (Fig. 1), have a cavity of 150x100x4 mm^3 and was prepared with the application of the mould release agent QZ13 from Ciba.

For the production of 25% volume fraction of sisal fibre reinforced composite plates, we calculated the need of 20 grams of fibre for each plate. That amount of fibre before compression is in volume 4 times bigger than the final thickness of the plate. After the preparation of the resin, we introduce it in the mould (Fig. 2). The mat is then placed in the mould (Fig. 3) and the mould closed. At last, the mould is placed in the hot plate press, and submitted to a cure stage of 1 hour at 60 ºC.

Fig. 2. Introducing the resin

Fig. 3. Placing the mat

Void content and a non homogeneous distribution of the sisal fibres in the matrix are, apparently, the main problems that need optimisation in the production process used. For the plates produced, we estimate, by measuring the plate and weighing it, that the void content is around 10 %.

Fig. 4. Plate after the opening of the mould

5. MECHANICAL CHARACTERISATION AND RESULTS

For the mechanical characterisation of composite materials it was used an INSTRON 4208 universal testing machine. The tests were made according to the ISO 527-4 standard, using a 100 kN load cell and a 2 mm/min traction speed. Fig. 5 shows the setup.

Fig. 5. Test setup

The Tables 1 to 6 and Fig. 6 and 7 shows the results of the mechanical characterisation for the REAPOX WOOD RX8 resin and for the composite plates made with 25% volume fraction of sisal fibre with and without surface treatment.

Table 1. 25% sisal fibre without surface treatment

	Without treatment		
Id. Specimen	**σ_r [MPa]**	**E [GPa]**	**ε_r [%]**
0	36,48	4,23	1,02
1	43,61	4,63	0,89
2	47,16	5,61	1,14
3	52,97	5,02	1,22
Average	**45,05**	**4,87**	**1,07**
StDev.	6,90	0,59	0,15
Minimal Value	36,48	4,23	0,89
Maximum Value	52,97	5,61	1,22

Table 2. 25% sisal fibre (4% NaOH, 1 hour)

	4%(NaOH), 1 hour		
Id. Specimen	**σ_r [MPa]**	**E [GPa]**	**ε_r [%]**
4.1.0	47,34	6,80	0,97
4.1.1 *			
4.1.2	50,09	6,23	1,05
4.1.3	52,11	6,49	0,89
Average	**49,85**	**6,51**	**0,97**
StDev.	2,40	0,29	0,08
Minimal Value	47,34	6,23	0,89
Maximum Value	52,11	6,80	1,05

* Brake at the grips

Table 3. 25% sisal fibre (4% NaOH, 2 hour)

	4%(NaOH), 2 hour		
Id. Specimen	**σ_r [MPa]**	**E [GPa]**	**ε_r [%]**
4.2.0	58,38	7,16	1,00
4.2.1	62,33	6,21	1,21
4.2.2	69,71	7,44	1,18
4.2.3	60,81	5,75	1,37
Average	**62,81**	**6,64**	**1,19**
StDev.	4,88	0,79	0,15
Minimal Value	58,38	5,75	1,00
Maximum Value	69,71	7,44	1,37

Table 4. 25% sisal fibre (8% NaOH, 1 hour)

	8%(NaOH), 1 hour		
Id. Specimen	**σ_r [MPa]**	**E [GPa]**	**ε_r [%]**
8.1.0	49,75	5,47	1,10
8.1.1	63,71	6,93	1,20
8.1.2	59,48	5,97	1,28
8.1.3	64,92	6,00	1,43
Average	**59,47**	**6,09**	**1,25**
StDev.	6,88	0,61	0,14
Minimal Value	49,75	5,47	1,10
Maximum Value	64,92	6,93	1,43

Table 5. 25% sisal fibre (8% NaOH, 2 hour)

	8%(NaOH), 2 hour		
Id. Specimen	**σ_r [MPa]**	**E [GPa]**	**ε_r [%]**
8.2.0	47,90	5,96	1,01
8.2.1	49,00	5,96	1,19
8.2.2	54,45	6,82	1,21
8.2.3	46,71	5,94	0,94
Average	**49,51**	**6,17**	**1,09**
StDev.	3,42	0,43	0,13
Minimal Value	46,71	5,94	0,94
Maximum Value	54,45	6,82	1,21

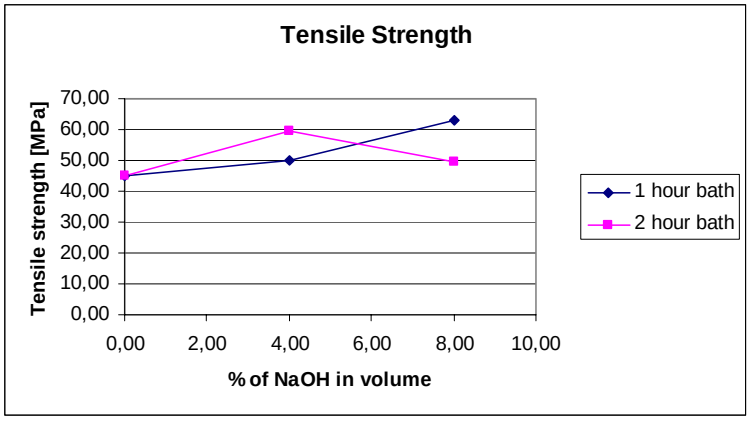

Fig. 6. Tensile Strength Graphic

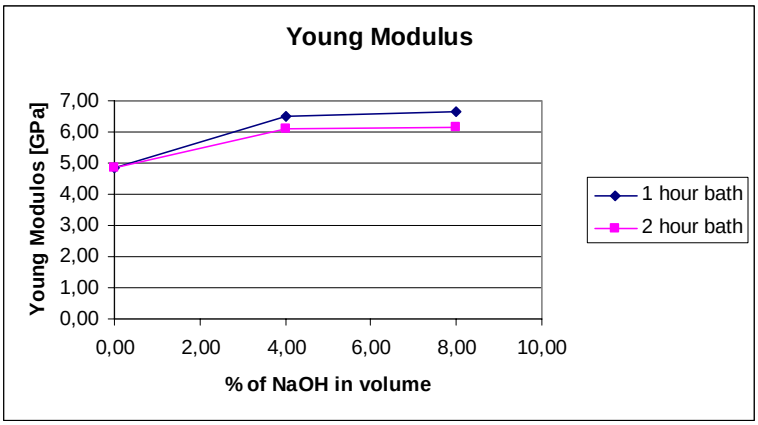

Fig. 7. Young Modulus Graphic

Table 6. Comparison between the mechanical characterisation of the different fibre surface treatment

	Composite	Tensile Strength σr [MPa]	Modulus E [GPa]	Deformation ε_r [%]
A	REAPOX WOOD RX8 +25% sisal fibre Without treatment	45,05	4,87	1,07
B	REAPOX WOOD RX8 +25% sisal fibre 4%(NaOH), 1 hour	49,85	6,51	0,97
C	REAPOX WOOD RX8 +25% sisal fibre 4%(NaOH), 2 hour	62,81	6,64	1,19
D	REAPOX WOOD RX8 +25% sisal fibre 8%(NaOH), 1 hour	59,47	6,09	1,25
E	REAPOX WOOD RX8 +25% sisal fibre 8%(NaOH), 2 hour	49,51	6,17	1,09
F	Resin REAPOX WOOD RX8	49,98	2,91	6,50

If we look to the tensile strength, the value obtained by the different natural sisal fibres composites is similar or inferior to the resin matrix for the composites A, B and E. If we look to the modulus, the sisal fibres composite present more than the double of the matrix modulus value, except for the non treated fibres.

A bad fibre/matrix adhesion could be responsible for these results even the surface treatments made to the fibres improve the adhesion between fibre and matrix, observed by the results obtained in comparison with the untreated fibres.

When analysing the crack surface, we could confirm that the adhesion fibre/matrix was, in some cases, bad. In some cracks surfaces, we could see small parts of fibre completely removed from the matrix. That shows that the adhesion is poor, and that the fibre is weakening the matrix instead of reinforcing it.

6. CONCLUSIONS

It is necessary to point the research in the surface treatment of the fibres. Only with good surface treatment we can obtain a good adhesion fibre/matrix, and that is one key-point to improve the mechanical composite properties.

In this study, we try to improve the mechanical properties of sisal natural reinforced plastics by the modification of the surface properties of the fibres. We made the study using the mercerizing treatment of the fibre surface with different concentration of Sodium Hydroxide solution (NaOH) and different bath time. The treatments give some results with some improvements of the mechanical properties. Future works should be made to study the improvement of the mechanical properties by the optimisation of the mercerization treatment parameters (concentration of NaOH solution, time and bath temperature).

The production process of the sisal fibres composites must also be optimised to promote a homogeneous distribution of the aleatory fibre reinforce and minimize the volume fraction of voids.

ACNOWLEDGEMENTS

This work was implemented in the scope of a research and development project "POCTI/40201/EME/2001 – Mechanical Behaviour of Natural Fibre Reinforced Plastics Composites", supported by "Fundação para a Ciência e Tecnologia", program POCTI and FEDER, Portugal.

References

1. **C. Romão**, "Study of the Mechanical Behaviour of Composite Polymeric Materials Reinforced with Natural Fibers", Dissertation presented for obtaining a Master's degree in Mechanical Engineering, from the Engineering Faculty in the University of Porto, April, 2003.
2. Project SAPIENS POCTI/40201/EME/2001, "Mechanical behaviour of Composites of Polymeric Matrix Reinforced with Natural Fibers", Report of Annual Progress, 1º year of activity, 2002.
3. **Peters R.H**. "Textile Chemistry I: The Chemistry of Fibres", Elsevier Publishing Company, 1963.
4. **Peters R.H**. "Textile Chemistry II: Impurities in Fibres, Purification of Fibres", Elsevier Publishing Company, 1967.
5. **Rowell R.M**. "Chemical Modification of Agricultural Fibers for Property enhanced Composites", Cap. V, Project: Modification of Lignocellulosics for Advanced Materials and New Uses, http://www.fppl.fs.fed.us, 1995.
6. **Rowell R.M., Han J.S. and Rowell J.S.** "Characterization and Factors Effecting Fiber Properties", Natural polymers and Agrofibers Composites, pp. 115-134, 2000
7. **Eichorn S.J., Baillie C.A., Zafeiropoulos N., Mwaikambo L.Y., Ansell M.P., Dufresne A., Entwistle K.M., Herrera-Franco P.J., Escamilla G.C., Groom L., Hughes M., Hill C., Rials T.G. and Wild P.M**., "Current International Research into Cellulosic Fibres and Composites", Journal of Materials Science, v36, pp.2107-2131, 2001.