

MECHANICAL CHARACTERISATION OF WOOL FIBRES FOR REINFORCING OF COMPOSITE MATERIALS

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ABSTRACT

The work described in this paper refers to the mechanical characterisation of Portuguese wool fibres, for the production of composite materials.

Single filaments, yarns, woven fabrics and composite plate's mechanical characterisation are made.

The comparison between a reference glass fibres reinforced composite is also made.

A brief description of the production and test setups of the fibres composite materials is made.

The use of these animal fibres is pointing also to the ecological problem of composite materials recyclability.

1. INTRODUCTION

This project starts from the observation of some less explored potentialities, in the inland region of Portugal, and which are related with the local handcraft production.

On this matter, one of the factors is the production and weaving of sheep wool.

This sheep's product can be considered a surplus of the main purpose of the sheep's rearing which is to obtain milk for cheese production, one of the main revenue sources of the populations in "Serra da Estrela" region.

In many cases, and even if people use part of this wool to semi handcraft production of high quality wool tissues, a great part is useless since it isn't enough demand for this product.

One of the possibilities to reinvigorate this activity is to take advantage of this material in the production of new objects that continue to be handcrafted, and would take the maximum profit of the local know-how, opening new perspectives of business to the local undertakers. That would contribute a little to the growing of the productivity of this most economically disfavoured region.

Following this principle, it comes forward the idea of connecting this activity to the composite materials sector. Using wool, in natural state or manufactured, as a reinforcement in a composite material, we could, with less difficulty, and without big investments, increase some sector's small businesses activities that, besides the fact of maintaining their productive methods (hence the fact that, for this experience, we use products now obtained with this production, as well as base products similar to those produced in that units, as natural wool), could enlarge their range of products, creating a non-existent offer in Portugal and less explored internationally. This technique would allow, for instance, the creation of a variety of domestic articles as furniture, accessories, etc.

On the other hand, we notice the growing interest by issues related with sustainable development, which should be also the interest of the researchers in a variety of

scientific areas, namely the composite materials and, in this case, composites with thermoset polymeric matrix.

It is known that this kind of materials has an ecological problem that is the fact of not being able to be recycled (besides charges, we don't recognize other means economically practicable to process products in thermoset composites of polymeric matrix).

As far as it concerns, and as we can't recycle this material, we should search ways of reducing the environmental impact. One of these ways could be the replacement of the synthetic reinforcement fibres by natural fibres which, besides the obvious advantage of not being harmful to the environment in the final of the life cycle of the product, are originated in non pollutant and renewable sources.

There are many experiences developed in this field, above all the ones related with reinforcement of vegetables fibres. Nevertheless, there is still a huge range of possibilities in what concerns animal fibres, which are still less explored.

The investigation in course is related with this potentiality.

2. WORK DEVELOPMENT

In a first phase, and as a starting point, we decided to make some thermoset composites of polymeric matrix, having as reinforcement a mat of wool fibres, without treatment (just with a first producter's washing) and wool tissue provided by the enterprise "Ecolã", whose head-office is in Manteigas – Portugal, in a way that would allow us to have the first idea of the characteristics of the material.

We made three different composites:

- 1 – Wool tissue with polyester resin
- 2 – Wool tissue with epoxy resin
- 3 – Wool fibres Mat (30% in volume) with epoxy resin

We used resins in the following formulations:

Epoxy – Reapox 520 / D526

Polyester – Quires 272 THV + 0,2% cobalt accelerator (1% volume) and 2% catalyser MEK50

The two first composites, with wool tissue were made by hand lay-up and the third, with wool mat by compression moulding. They were made at ambient temperature (more or less 16 °C) and without fibres treatment. In the three cases, composites were subjected to enough pressure to guarantee homogeneity. The fibre volume percentage in the final composite of the wool mat was of 30%.



Fig. 1 - Lay up of the composite reinforced with tissue



Fig. 2 - Composite under pressure



Fig. 3 - Introduction of wool fibres in the mould

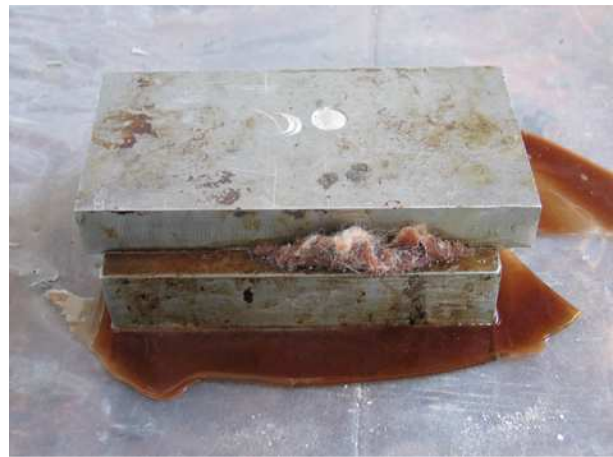


Fig. 4 - Wool fibres with epoxy resin in the mould

During the process, we noticed some difficulty in fibre impregnation, both in the tissue and in the mat. Nevertheless, the final result presented a very satisfactory impregnation, from which resulted an extremely homogeneous composite.

After a 8 days cure at ambient temperature, the plates obtained were cut in samples (Fig. 5). Traction and flexion tests were made for the tissue reinforced composites and flexion tests for the mat reinforced composite.



Fig. 5 - Traction test samples

From these tests, the following results were achieved:

Table1. Mechanical tests results

	Modulus (GPa)	STDev (%)	Rupture Strength (MPa)
Traction tests results			
Tissue + Epoxy	3,26	0,093	35.22
Tissue + polyester	1,746	0,044	23.88
Flexion tests results			
Mat + Epoxy	2,13	0,34	37.89
Tissue + Epoxy	3.13	0,12	51,05
Tissue + Polyester	1,83	0,078	44,38
Matrix values from the producer			
polyester 272	3.5		75
epoxy reapox 520	3.5		70



Fig. 6 - Traction test - composite tissue/epoxy



Fig. 7 - Flexion test - composite mat/epoxy

The traction samples were tested at a speed of 2 mm/min and the flexion samples at a speed of 2 mm/min

The results were below the resin values without reinforcement, which can invalidate this material as reinforcement. Nevertheless, some articles of other investigators show us that wool could effectively be used with that aim. [5]

It is necessary to study possible treatments to the fibres, so that the properties can be improved to the needed ones.

In the rupture crack we could observe a possible degradation of the fibres.

3. YARNS MECHANICAL CHARACTERIZATION

We decided to make traction tests with wool yarns, in which part of the yarns would be subjected to a thermal treatment, as a way to guarantee the lack of humidity.

We made 12 samples:

- 3 samples with polyester and wool yarns without treatment
- 3 samples with polyester and heated wool yarns treated at a constant temperature of 100° for 20 minutes
- 3 samples with epoxy and wool yarns without treatment
- 3 samples with epoxy and heated wool yarns treated at a constant temperature of 100° for 20 minutes
- 2 samples with wool yarns without treatment

We used resins in the following formulations:

Epoxy – Reapox 520 / D526

Polyester – Quires 272 THV + 0,2% cobalt accelerator (1% volume) and 2% catalyser MEK50

The yarns were impregnated with the respective resin after being slightly tensioned (manually, with the help of adhesive tape) and were subjected to an 8 days cure at ambient temperature.



Fig. 8 - Wool yarns with epoxy resin



Fig. 9 - Traction test of the wool yarn

Table2. - Traction tests results:

Specimen	Material	F (N)	σ (Mpa)	Average(Mpa)	Relation between s/a and c/a
1	Wool	6.12	15.91	17.515	
2	Wool	7.35	19.12		
3	wool s/a + epoxy	24.13	30.74	29.097	Improvement of 20%
4	wool s/a + epoxy	23.48	29.92		
6	wool s/a + epoxy	20.91	26.63		
7	wool c/a + epoxy	27.48	35	34.930	
9	wool c/a + epoxy	26.28	33.47		
10	wool c/a + epoxy	28.5	36.31		
11	wool s/a + polyester	19.67	25.06	22.097	Improvement of 27%
12	wool s/a + polyester	14.89	18.97		
13	wool s/a + polyester	17.47	22.26		
15	wool c/a + polyester	19.75	25.16	29.350	
16	wool c/a + polyester	26.33	33.54		

s/a – Without heating treatment

c/a – With heating treatment

The samples were tested at a speed of 10 mm/min. The achieved values are merely of reference, allowing us to compare the fibres performance in function of the thermal treatment.

We assume that the wool yarn has an initial section of 0.7 mm and for the impregnated resin yarns an initial section of 1 mm. The previous heated treated yarns results are clearly better in average.

4. GEL TIMER

As far as we didn't saw degradation in the fibres after they suffered the thermal treatment, we have done a test to examine if the temperature was exceeded by the resin at the moment of the polymerization.

We also measured the amount of time during which the composite was subjected at temperatures above 100° C.

To this effect we made gel timers with the epoxy and polyester resins, in which we achieve the following results:

Results from the polyester resin 272

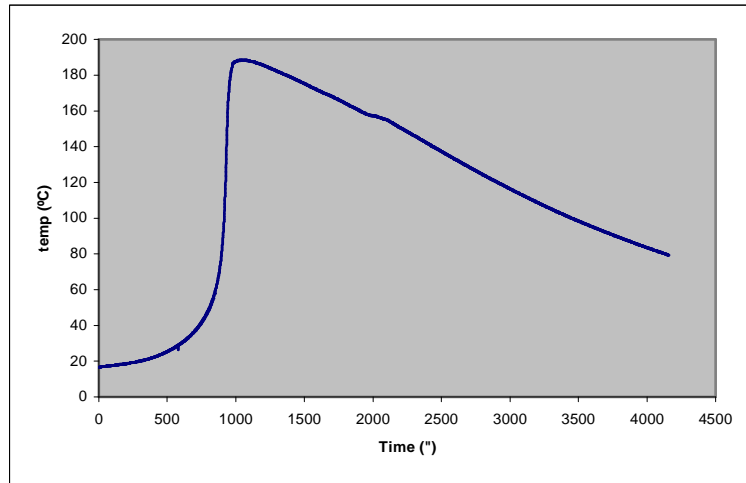


Fig.10 – Polyester resin 272 Gel Timer

Polyester resin– 272

Catalyser – 2%

Accelerator – 0,2% (1% volume)

Ambient temperature – 16.5° C

Gel Timer – 13.38'

Time to reach 100° C - 915''

Maximum C - 188.492° C in 1033''

Time to reach 100° C in the descending line - 3447''

From 100° C to the maximum Exothermic reaction temperature – 118''

From the maximum Exothermic reaction temperature to 100° C in the descending line – 2402''

Time between 100° C ascending and descending – 2520'' (42.0')

Results of the epoxy resin Reapox

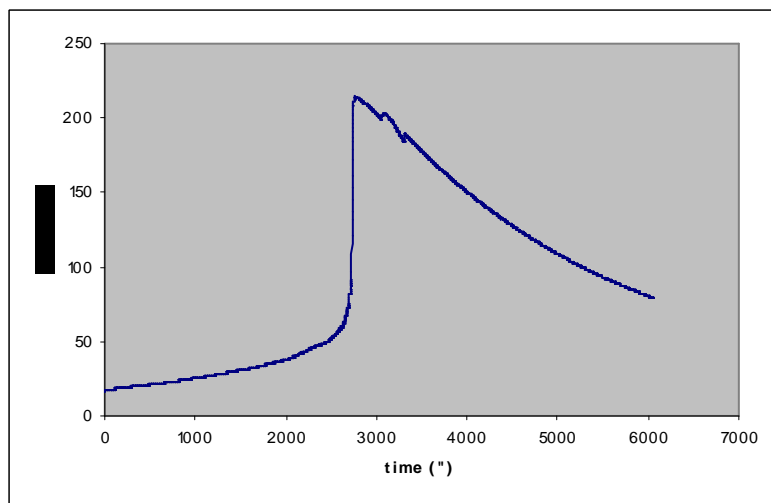


Fig. 11 – Epoxy resin Reapox 520 / D526 Gel Timer

Resina epoxy - reapox 520 – 100 parts in weight
Hardener - endur D526 – 30parts in weight
Ambient temperature – 16° C

Gel Timer – 46.42'

Time to reach 100° C - 2727'' (45.21')

Maximum Exothermic reaction temperature – 214.428° C in 2767'' (46.11')

Time to reach 100° C in the descending line - 5259''

From 100° C to the maximum Exothermic reaction temperature – 40''

From the maximum exothermic reaction temperature to 100° C in the descending line – 2492''

Time between 100° C ascending and descending – 2532'' (42.2')

We noticed that, in a glass, the resin reaches very high temperatures and, in the case of epoxy resin, it exceeds the 200° C.

Nevertheless, and being the volume of pure resin pretty high when we have done the experience in a glass, we deduced that, eventually, the temperature effectively reached by the composite could be less, as the amount of resin by composite volume is inferior.

We made then the measurement of the polymerization temperature when it is used together with the reinforcement, in a plate.

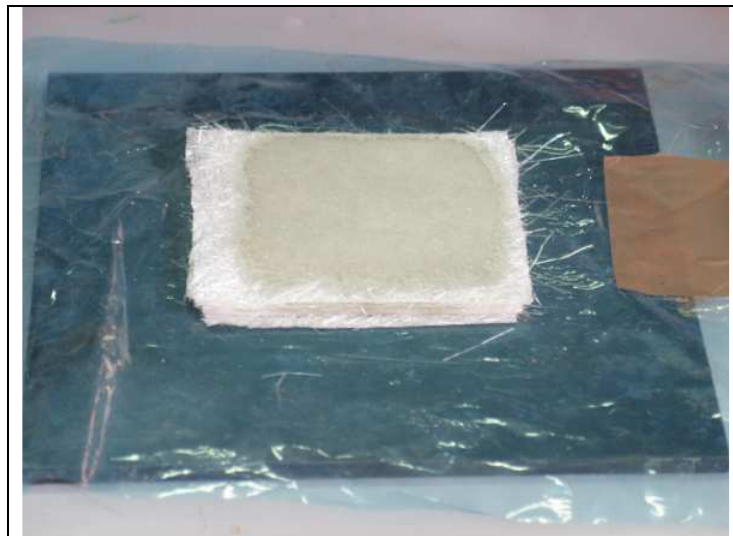


Fig. 12 - Composite plate equipped with a thermocouple

In this experience, we noticed that, effectively, the temperature in the composite didn't reach the 100° C, as the graphic below shows us .

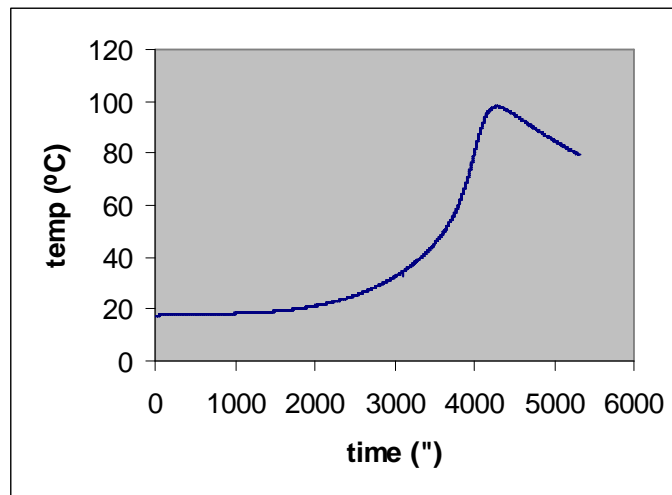


Fig 13 - Composite temperature curing graphic

In the tests sequence, we made an experience with the aim of analysing if the temperature effectively damaged the fibres. For that, we subjected a wool yarn to a temperature of 200° C for 5 minutes.

Comparing to other yarns without heating, we notice a slight contraction.

The initial yarn mass was of 0.0569g and the final one was of 0.0139g, which means a mass loss of 0.0430g. That means 75.57% of the initial mass.

5. CONCLUSIONS

A poor interface fibre-matrix adhesion could led to the appearance of “holes” in the composite, that weakened it and influence an inferior performance in relation with the matrix.

The fibres absorb lots of humidity and have the possibility of the presence of natural fat. These are also two potential weakening agents.

A combination between fibre and resin still requires some study, namely at the level interface adhesion.

A Possible fibre degradation in the composite could be originated by the chemical attack of the matrix, hence it is necessary to eliminate some variables already pointed out in a way we could achieve some conclusion.

The temperature, even above the exothermic pique of a resin, doesn't appear to be damaging the fibres, so this may not be the motive for their degradation in the composite.

6. ACKNOWLEDGEMENTS

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References

1. **C. Romão**, “Study of the Mechanical Behaviour of Composite Polymeric Materials Reinforced with Natural Fibres”, Dissertation presented for obtaining a Master's degree in Mechanical Engineering, from the Engineering Faculty in the University of Porto, 2003.
2. Project SAPIENS POCTI/40201/EME/2001, “Mechanical behaviour of Composites of Polymeric Matrix Reinforced with Natural Fibres”, Report of Annual Progress, 1^o year of activity, 2002.
3. **P. Vieira, C. Romão, A.T. Marques, J.L. Esteves**, “Mechanical Characterisation of Natural Fibre Reinforced Plastics”, MATERIAIS 2003, II International Materials Symposium, April 14-16, 2003.
4. **C. Romão, P. Vieira, F. Peito, A. T. Marques, J. L. Esteves**, “Single Filament Mechanical Characterisation of Hemp Fibres goes Reinforcing of Composite Materials”, The Seventh International Conference on Frontiers of Polymers and Advanced Materials–ICFPAM VII, Bucarest, Romania, 10-15/6/2003,
5. **A.S. Blicblau, R.P. Coutts, A. Sims**, “Strength of raw wool polyester composites”, Swinburn University of Thecnology + CSIRO Division of Forrest Products – Melbourne - Australia