THE ALFA FIBERS IN COMPOSITE MATERIALS

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SUMMARY : This work presents a study of the influence of fibers extraction processes on the behavior of composite materials made of natural fibers as reinforcement and polyester matrix. The obtained results show that when Alfa fibers are extracted using a 3N NaOH aqueous solution at 100°C, in the ambient pressure, during 2hours, then bleached with a 40% NaClO aqueous solution during 1hour they lead to fibers able to produce composite materials with interesting proprieties (tensile modulus : 4GPa, tensile strength : 41MPa, deformation : 1.4%).
This study describes also the influence of the extraction process on the fibers density. We noticed that the density decreases with the soda reaction time and the bleaching period. The Kappa index gave us the amount of lignin kept in the fibers and confirms the obtained density results.

This work is a continuation of a former study that we performed on the field of biocomposites and which concerns the Agave fibers in composites materials[1].

KEYWORDS : Alfa fibers, Esparto fibers, Polyester Alfa composite, Density, Tensile test, Kappa index.

INTRODUCTION

Eighty years ago, nearly all the materials used for the production of commodities and many technical products were derived from natural textiles, ropes and paper made of natural fibers. Some of them are still used today. The first composite materials were applied for the fabrication of big quantities of sheets, tubes and pipes in electronic usage. This composite was made of paper or cotton as reinforcement and phenol resins as matrix [2].

However, because of the low price and steadily rising performances of technical and standard plastics, the application of natural fibers came to a near halt. The current critical discussion about preservation of natural resources and recycling, led to a reflection about natural materials as raw materials.

Today, the renaissance of natural fibers as reinforcement in technical application is taking place mainly in the automobile industry where door panels and car roofs where made of natural fibers fleece (flax) with epoxy resin or PUR composite[3].

At the moment we can number a large range of natural fibers. Some of them are well known in the composites universe such as flax, sisal, jute and coir and many other are still subjects of studies, researches and experiments. From these fibers we can mention the Alfa fiber.

The Alfa is the Arab name of the esparto grass. The esparto is the plant Stippa Tenacissima of the dry regions of North Africa. It belongs to the graminacies family and grows to a height of about 1m, with cylindrical shape. The Alfa is widely cultivated in Tunisia especially in the centre of the country and cover about 3500Km². The annual Tunisian production of Alfa is 60000 tons and it's price is around 40 dollars/ton.

Short, fine and light cellulosic fibers are extracted from the Alfa stem. These fibers are mostly used in the production of high quality papers. The length of Alfa fibers is about 25mm and their diameter closes the 10microns (See figure 1). One of their particularities is the high value of the angle between the microfibers and the principal axis of the fiber which can have an important impact on the relationship fiber-fiber or fiber-matrix. The Alfa stems are composed of about 45% of cellulose,25% of pentozane, 23% of lignin, 5% of wax and 2% of cinders. In practical it's difficult to extract more then 43% of cellulosic fibers.

Figure 1 : The Alfa fibers extracted in the laboratory

The Alfa fibers are generally extracted from the plant by the soda process which is a hydrolysis in a NaOH solution. To study the behavior of the Alfa fibers in polyester matrix, we have begun by the research of the best extraction conditions and the different steps to follow to get the best composite Alfa/Polyester. We will start by presenting the extraction process.

EXTRACTION OF ALFA FIBERS

In the industry, the Alfa fibers are extracted from the stem by principally the soda process which begins by the cooking of the plant in a 2N NaOH solution at 170°C under a steam jet of 7.5bars during 14 minutes. Then the obtained fibers are washed by water to separate the chemicals from the cellulose. The fibers are then filtered from the solution and the obtained product in commonly called 'Alfa Pasta'. We should notice that the soda transforms all the Alfa constituents in aqueous phase except the cellulose.

The obtained fibers are bleached in three steps by chlorination, sodation and hypochlorite oxydation.

In Tunisia, a large amount of Alfa fibers are extracted in this way and used in paper manufacturing.

In the laboratory, Alfa fibers were extracted using a 3N NaOH aqueous solution at 100°C in the ambient pressure and then were bleached in 40% aqueous NaClO solution.

We decided to extract our own fibers to be used as a reinforcement instead to use the commercial fibers mainly for two principal reasons :

-The first one is to be able to control all the extraction steps and to use them as parameters in this work, especially the sodation reaction time and the influence of the NaClO bleaching on the fiber resistance.

-The second reason is to be sure that we are always using the same fibers origin, cut in the same place, the same period and treated in the same way.

We extracted different samples of Alfa fibers using different reaction times in Soda. We cooked the Alfa stems during 1, 2, 3, 4 and 5 hours in the 3N NaOH solution at 100°C under the ambient pressure. The obtained fibers were then bleached in NaClO solution during 1, 2, 3 and 4 hours.

Finally we obtained 16 extracted samples of Alfa fibers. Table 1 gives the labels of these different samples as named in the next paragraphs.

Table 1 : Extraction times and bleaching periods of Alfa fiber and labels of the samples.

Soda extraction time	**NaClO bleaching time**			
	1hour	2hours	3hours	4hours
1hour			1h3hN	1h4hN
2hours	2h1hN	2h2hN	2h3hN	2h4hN
3hours	3h1hN	3h2hN	3h3hN	3h4hN
4hours	4h1hN	4h2hN	4h3hN	4h4hN
5hours	5h1hN	5h2hN		

We calculated the yield of the different reactions and we obtained an overall average of 40.2% of cellulosic Alfa fibers.

We determined the density of all the extracted fibers according to the standard NF T 20-053 [4]. We used for this purpose a pycnometer and a vacuum pump. The liquid used for this experiment is the carbon tetrachloride (CCl_4) with a known density. The principle of this experiment consists to weight a certain quantity of fiber in the pycnometer then to extract all the air that they can contain in their bodies, and finally to fill, under vacuum, the volume of the pycnometer by CCl_4. The weight of the liquid inside the pycnometer give us its volume and consequently the volume of fibers.

The obtained results are shown in table 2.

According to the results presented in table 2, we can notice that the extraction method affects the fiber density. However, more the soda reaction time is high more the fiber density is low, and more bleaching time is high, more the fiber density is low. This is expectable since both NaOH and NaClO solutions contribute to the lignin destruction. We can imagine that the fibers remain almost in their same volume but they lose weight by loosing lignin.

We can also notice that there is not too much difference in density for the fibers bleached during just 1 hour, whatever the soda cooking time beyond 5 hours. Nevertheless as an average, we can notice clearly that the density of the Alfa fibers varies between 1.3 and 1.4 whatever the extraction conditions.

To continue studying the extraction influence on the fiber composition, we measured the amount of lignin covering the fibers by the Kappa index method. The Kappa index indicates the degree of lignin that cellulosic fibers may contain after a soda extraction. We used the method described in the standard ISO 302-1981 (F) [6] which consists to measure the pure cellulose amount in a sample of fibers by oxidizing the cellulose with a solution containing potassium permanganate, sulfuric acid, sodium thiosulfate and potassium iodide.

Table 2 : Density of the different extracted samples of fibers.

Sample			**1h3hN**	**1h4hN**
Density			1.409	1.393
Sample	**2h1hN**	**2h2hN**	**2h3hN**	**2h4hN**
Density	1.391	1.380	1.372	1.297
Sample	**3h1hN**	**3h2hN**	**3h3hN**	**3h4hN**
Density	1.388	1.368	1.366	1.350
Sample	**4h1hN**	**4h2hN**	**4h3hN**	**4h4hN**
Density	1.384	1.357	1.353	1.311
Sample	**5h1hN**	**5h2hN**		
Density	1.352	1.345		

We measured the Kappa index for only the fibers bleached during 1h. The obtained results are shown in table 3.

Table 3 : Kappa indication.

Fiber samples	**2h1hN**	**3h1hN**	**4h1hN**	**5h1hN**
Kappa index, χ	18.03	19.38	19.81	20.60

We can notice from the above tables that the fibers loose lignin with the increase of the soda reaction time. In fact, the Kappa index allows the determination of the conditions which give the best fibers when needed for a specific application. For instance, the Kappa index of Alfa fibers destined to paper manufacturing is not the same as the Kappa index of fibers which will be used as reinforcement in composites. In paper they need pure cellulose in order to get final product with some special characteristics such as the capability to absorb ink, the opacity, and the permeability to the air. This doesn't mean that they will give a good composite material when used as a reinforcement.

COMPOSITE MATERIAL REINFORCED WITH ALFA FIBERS

We worked up with the obtained fibers Polyester/Alfa fibers composite materials. We manufactured test tubes for tensile tests with the Alfa samples according to the instruction of the standard ISO 527-4 [6]. The filling-up of the composite Polyester/Alfa is 20% and the fibers were put in random. We worked up also a Polyester/glass fibers composite with the same filling up to compare the behavior of Alfa fibers to the E-glass fibers, in polyester composites.

Table 4 gives the technical characteristics of the resins and the fibers used to work up the composites mentioned above.

Table4 : Technical characteristics of the polyester resin and glass fiber used

	Viscosity 23°C(MPa.s)	Density	Tensile Modulus(MPa)	Tensile Strength (MPa)	Deformation (%)
Polyester resin	180	1.1	2600	40	2
E-glass fiber		2.6	73000	3400	4.5

We have produced 3 test tubes from every fiber sample using a mold shown in figure 2. With this mold, we realized Alfa/Polyester composite plates from which we cut the desired test tubes.

The test tubes dimensions are 250 x 25 x 3mm. The composite plate dimensions are 250 x 85 x 3mm. Every plate gave us 3 test tubes. At the total we realized 54 test tubes.

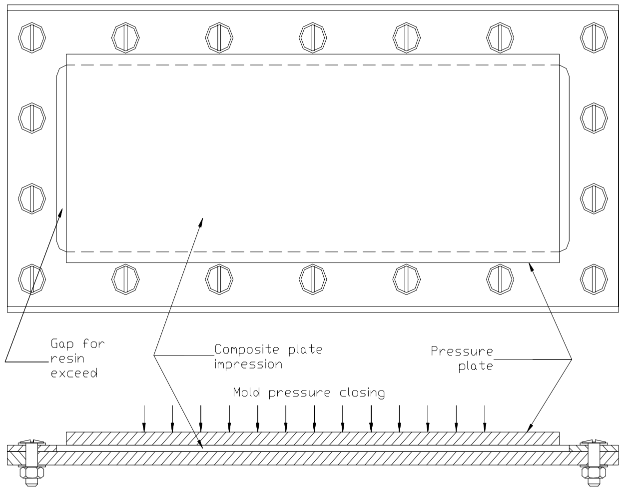

Figure 2 : Design of the used mold to realize the test tubes

We have performed mechanical tensile tests according to the standard ISO 527-1 [7]. The obtained results are presented in table 5.

According to table 5 we notice that the best mechanical characteristics of the composites Alfa/Polyester are obtained with a soda reaction time of 2 hours and a bleaching time in NaClO of 1hour. We notice also that for this kind of samples, the NaClO solution treatment has a big influence on the Alfa composites characteristics (see figure 3). This can be noticed by the decrease of the deformation, the increase of the tensile modulus and the tensile strength according to the decrease of the bleaching time.

Besides, table 5 shows that the soda reaction time has also an influence on the composite behavior for all the bleaching times. Figure 4 confirm this result for the fibers bleached during 1 hour.

Table 5 : Results of the mechanical tensile tests

Sample	Polyester	E-Glass	1h3hN	1h4hN
Tensile strength (MPa)	28.67	49.32	30.72	32.77
Deformation (%)	2.97	2.49	2.4	2.26
Tensile Modulus (MPa)	1572.67	2996.34	3325	3320
Sample	**2h1hN**	**2h2hN**	**2h3hN**	**2h4hN**
Tensile strength (MPa)	40.95	39.54	37.96	37.69
Deformation (%)	1.38	1.46	1.48	2.00
Tensile Modulus (MPa)	3506	3443	3328	3078
Sample	**3h1hN**	**3h2hN**	**3h3hN**	**3h4hN**
Tensile strength (MPa)	38.88	35.68	34.92	32.18
Deformation (%)	1.58	1.76	1.84	2.18

Tensile Modulus (MPa)	3323	3209	3011	2804
Sample	**4h1hN**	**4h2hN**	**4h3hN**	**4h4hN**
Tensile strength (MPa)	36.69	34.82	34.05	31.43
Deformation (%)	1.62	1.80	1.84	1.86
Tensile Modulus (MPa)	3258	3195	2945	2830
Sample	**5h1hN**	**5h2hN**		
Tensile strength (MPa)	31.69	30.86		
Deformation (%)	2.28	2.64		
Tensile Modulus (MPa)	2982	2853		

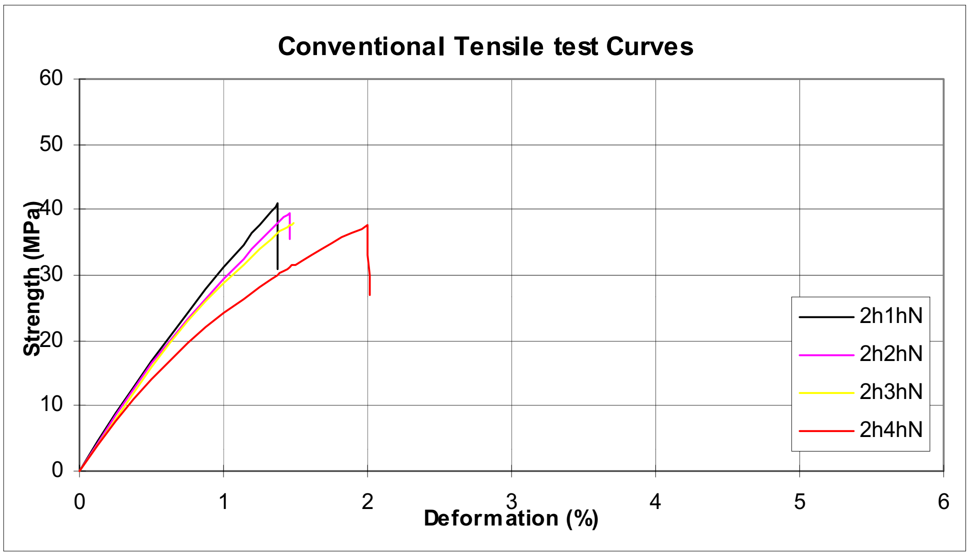

Figure 3 : Conventional tensile test curves of Alfa/Polyester composites treated during 2hours in 3N NaOH solution and bleached for different times.

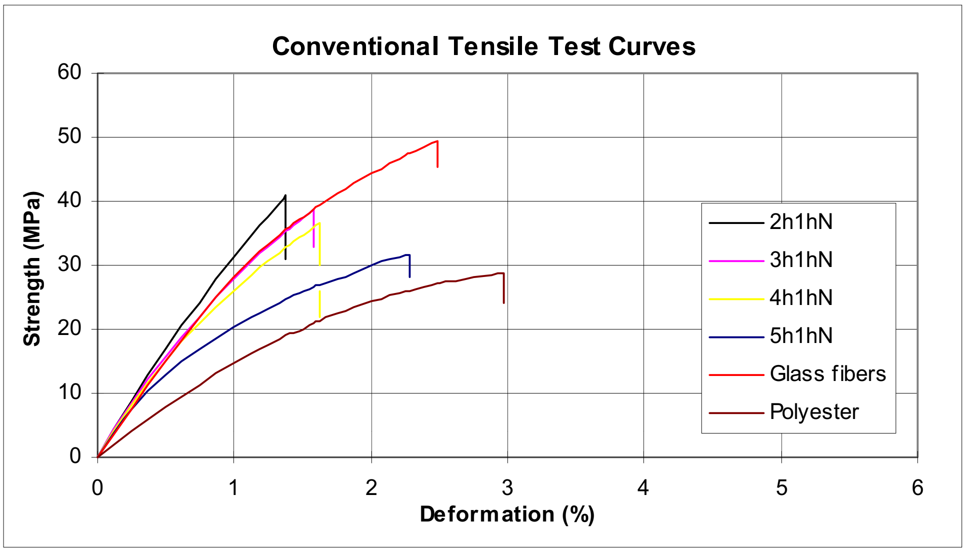

Figure 4 : Conventional tensile test curves of Alfa/Polyester composites treated for different times in NaOH solutions and bleached during 1hour in NaClO solution.

As global results, we can notice the higher tensile modulus of the Alfa/Polyester composites comparing to the E-Glass/Polyester composites. In the other hand, the E-Glass/polyester composites have a larger deformation coefficient and tensile strength comparing to the Alfa fibers reinforced composites.

CONCLUSION

This study give us a global view about the traction mechanical characteristics of Alfa/Polyester composites in which the fibers were put in random. The obtained characteristics are not to far from the E-Glass/Polyester composites and have also a better tensile modulus. This results encourage to continue studying this kind of fibers and especially their influence on the composite when the fibers filling up vary.

We will also try to study the behavior of the composites made of short and long natural fibers used in alternative layers. As long fibers we will use the Agave fibers .

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