

Lucas F.M. da Silva  
Editor

# Materials Design and Applications

 Springer

*Editor*

Lucas F.M. da Silva  
Department of Mechanical Engineering  
Faculty of Engineering of the University  
of Porto  
Porto  
Portugal

ISSN 1869-8433

Advanced Structured Materials

ISBN 978-3-319-50783-5

DOI 10.1007/978-3-319-50784-2

ISSN 1869-8441 (electronic)

ISBN 978-3-319-50784-2 (eBook)

Library of Congress Control Number: 2016960268

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The registered company is Springer International Publishing AG

The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

# Preface

This volume of *Advanced Structured Materials* contains selected papers presented at the 1st International Conference on Materials Design and Applications 2016 (MDA 2016), held in Porto, Portugal, during June 30–July 1, 2016. The goal of the conference was to provide a unique opportunity to exchange information, present the latest results, as well as discuss issues relevant to materials design and applications. The focus is on fundamental research and application areas in the field of the design and application of engineering materials, predominantly within the context of mechanical engineering applications such as automobile, railway, marine, aerospace, biomedical, pressure vessel technology, and turbine technology. This includes a wide range of materials engineering and technology, including metals, e.g., lightweight metallic materials, polymers, composites, and ceramics. Advanced applications include manufacturing in the new materials, testing methods, and multi-scale experimental and computational aspects (e.g., microscale and nanoscale techniques). Approximately, 150 papers were presented by researchers from nearly 30 countries.

In order to disseminate the work presented in MDA 2016, selected papers were prepared which resulted in the present volume dedicated to ‘Materials Design and Applications.’ A wide range of topics are covered resulting in 31 excellent papers dealing with metals, ceramics, composites, design, power generation, additive manufacturing, machining, and joining. This book is state of the art of materials design and applications and also serves as a reference volume for researchers and graduate students working with advanced materials.

The organizer and editor wish to thank all the authors for their participation and cooperation, which made this volume possible. Finally, I would like to thank the team of Springer-Verlag, especially Dr. Christoph Baumann, for the excellent cooperation during the preparation of this volume.

Porto, Portugal  
September 2016

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# Comparison of Mechanical Properties of Polyester Composites Reinforced with Autochthonous Natural Fibres: Flax and Hemp

J. Rocha, J.E. Ribeiro and L. Queijo

## 1 Introduction

Nowadays, increasing pollution is causing climate change, and therefore the need to reduce carbon footprints has brought the growing use of autochthonous natural materials. On the other hand, there is new demand from the world population that requires the increased use of recyclable materials, and natural fibres are excellent candidates for this role. However, only in the last few years has this subject been studied for technical applications [1]. Natural fibre is a very interesting material, and in association with a matrix it forms a composite material. Natural fibre composites (NFCs) have some advantages compared with synthetic ones, with the most relevant being their lower density and higher specific strength and stiffness. Furthermore, they are a renewable resource whose production requires little energy and involves CO<sub>2</sub> absorption. Nevertheless, NFCs also have disadvantages, such as lower durability when compared to synthetic fibre composites, although this can be improved significantly with specific treatments; a greater variability of properties; and the fact that they suffer from higher moisture absorption, which results in swelling [2].

There are many factors that can influence the performance of natural fibre reinforced composites, of which the most important is the fibre selected [3]. The properties of natural fibre reinforced composites can also be influenced by the fibre dispersion or fibre volume fraction as well as the fibre orientation. In general, a high

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J. Rocha · J.E. Ribeiro (✉) · L. Queijo  
Polytechnic Institute of Bragança, C. Sta. Apolónia, 5301-857 Bragança, Portugal  
e-mail: jribeiro@ipb.pt

J. Rocha  
e-mail: jrocha@ipb.pt

L. Queijo  
e-mail: lqueijo@ipb.pt

fibre volume fraction is essential to achieve high performance of composites [4], while the fibre orientation yields composites with very different properties [5]. It is frequently observed that an increase in the fibre loading leads to the growth of tensile properties [6]. An additional factor that has an important influence on the mechanical properties of NFCs is the composites' interfacial strength, which can be changed using chemical treatments [7]. Hence, appropriate processing techniques and parameters must be cautiously selected in order to yield optimal composite materials [8]. Despite all these factors, matrix selection is also a very important factor that must be analysed to characterize the NFCs' mechanical properties [9]. In this work, the studied factors that affect the NFCs' mechanical performance are the fibre type and the interfacial strength, which are changed by chemical treatments.

Some of the most frequently used fibres in Portugal are hemp and flax. These fibres have their origins in plants that have been grown in the northeast of the country. In the territory that is now Portugal, flax growing and the manufacture of fabrics derived from it date from prehistory. There are traces of linen dated from 2500 BC in the Algarve region. More recently, in the nineteenth century, flax cultivation had great economic and social importance in the north of Portugal, having suffered a decline with the emergence of simpler and cheaper fibres, as was the case of cotton. In several historical periods, attempts were made to relaunch the linen industry [10]. In northeast Portugal, more specifically in the Vilarica valley, hemp was grown for manufacturing ropes in the National Cordage, which equipped caravels and other ships during the period of Portuguese discoveries throughout the fifteenth and sixteenth centuries. In the twentieth century, there were several hemp producers in the Vilarica valley. Industrial hemp growth is complex as it is a variant of cannabis (*Cannabis sativa*): these two variants differ only in terms of the tetrahydrocannabinol (THC) content, and thus the production of this plant is regulated by law [11].

The interfacial adhesion between fibres and matrix plays an important role in the mechanical properties of composites. As the stress is transferred from fibre to fibre across the matrix interface, a good interfacial adhesion is required to achieve a good reinforcement. Nevertheless, if the interface is too strong, fissures are able to propagate, which can reduce the toughness and strength. However, for fibre composites based on plants, the interaction between the fibres, which are usually hydrophilic, and matrices, which are generally hydrophobic, is very limited, leading to a low interfacial adhesion, which affects the mechanical properties. On the other hand, low humidity decreases the resistance of the properties over a long time period. To guarantee good adhesion, the matrix and fibres must be very close and, as is usual in any adhesion phenomenon, the property of wettability is fundamental to ensure the adhesion between the adhesive and adherent. In this particular case, insufficient wetting of the fibres originates interfacial flaws, which can act as stress concentrators [12] and affect the mechanical properties [13]. There are different types of fibre surface treatments, which can be classified as physical and chemical

and can improve the fibres' wettability and therefore improve the interfacial strength [14–16].

Interfacial adhesion may take place by different mechanisms, namely chemical bonding, inter-diffusion bonding, mechanical interlocking, and electrostatic bonding [17]. To improve the interfacial adhesion in NFCs, the authors of this work used the chemical approach. This approach includes many different techniques using chemical products like zirconate, peroxide, benzyl, acryl, titanate, permanganate, acetyl, alkali, and silane, among others [18]. These products can be used singly or in combination [7]. The most commonly used products are alkali, acetyl, and silane [19] and for this reason alkali–silane treatment [20] was selected for implementation in this work. Alkaline treatment consists in immersing the fibres in an alkaline solution, normally NaOH, for a period of time. This treatment removes fibre constituents including lignin, hemicellulose, pectin, and wax, which exposes cellulose and increases the surface roughness per unit area, providing an improved interfacial adhesion [21]. Silane treatment generally involves moistening the fibres in a solution of diluted silane in a water/alcohol mixture that will be broken down into silanol and alcohol in the presence of water. Silanol reacts with the cellulose OH groups in the natural fibres, forming stable covalent bonds in the cell walls [22]. Silane treatment improves the amount of reticulation in the interface region and increases the fibre surface area, causing a stronger adhesion between the matrix and the fibre [23].

To evaluate the influence of fibre type and chemical treatment, it is necessary to develop experimental work in which combinations of different factors are used, and for that reason it is important to organize experiments systematically using design of experiments techniques. These techniques make it possible to conveniently organize the experiments and then process the results statistically. The first published works that used design of experiments were done by Fisher [24], who initially used these techniques in the field of agriculture. His technique was based on factorial design, in which he created an orthogonal array with multi-factors and different levels for each factor.

Factorial design has different approaches depending on the number of experiments chosen by the researcher, and it can be general or fractional. In general factorial design, all possible combinations must be implemented, so this approach is only viable if the number of factors and levels is small. However, if the number of factors and levels is high, it is impractical to implement all the experiments because of economic and time costs. There are some methods that use the fractional approach [25], but the most popular method was developed by Genichi Taguchi (Tokamachi, Japan, 1924–2012) [26]. In the present work, only two factors were used: types of fibres and surface treatments, and considering this small number of factors, it is appropriate to implement a design of experiment model based on the general factorial approach.

## 2 Experimental Procedure

### 2.1 Materials and Properties

NFCs are constituted by the matrix and fibres. In this case, the matrix used was polyester with 1% catalyst to activate the polymerization. The natural fibres used were flax and hemp produced in northeast Portugal. The typical mechanical properties of these materials are listed in Table 1.

For the alkali-silane treatment, different chemical solutions were prepared according to the specificities of each treatment. Thus, solutions of sodium hydroxide (NaOH) with concentrations of 2 and 5% by mass were used for the alkali treatment. On the other hand, the silane treatment was performed using 5% by weight of 3-aminopropyltriethoxysilane, which was diluted in a 50% aqueous solution of methanol.

### 2.2 Plan of Experiments

The experiments were conducted in accordance with a standard orthogonal array, where each column defines the factor control. So, the first column corresponds to the type of fibre (A) and the second one is used for the fibre surface alkali-silane treatment (B), as can be seen in Table 2.

As mentioned before, in this work, two types of natural fibres were used: hemp and flax, some of which were used untreated while others were subjected to alkali-silane treatment of the fibre surface with NaOH concentrations of 2 and 5%.

**Table 1** Mechanical properties of matrix and natural fibres

		Density (g/cm <sup>3</sup> )	Tensile strength (MPa)	Young's modulus (GPa)
Matrix	Polyester [29]	1.2	70–103	2.1–4.4
Natural fibre	Flax [1]	1.5	345–1830	27–80
	Hemp [1]	1.5	550–1100	58–70

**Table 2** Orthogonal array of experiments

Experiment	A	B
1	1	1
2	2	1
3	1	2
4	2	2
5	1	3
6	2	3

**Table 3** Factors and respective levels

		Levels		
		1	2	3
Factors	A—Type of fibre	Hemp	Flax	–
	B—Surface alkali–silane treatment	Untreated	2% NaOH	5% NaOH

The reason for the choice of these concentrations was based on the paper published by Asumani et al. [20]. According to their work, the maximum improvement in tensile strength occurred for the 5% NaOH concentration, while and 2% concentration corresponds to an intermediate value between the untreated case and the case with an NaOH concentration of 5%. Table 3 lists the levels for each factor.

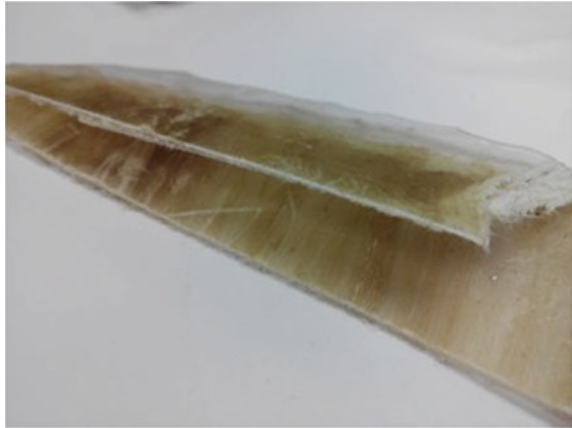
### 2.3 Specimen Manufacture and Tensile Tests

The specimens used in the tensile tests to determine the tensile strength of each combination of factors and levels were cut from a composite plate (200 × 200 1 mm<sup>3</sup>) manufactured previously. The fibres of these composite plates were aligned in one direction corresponding to the direction of tensile load application. Firstly, to prepare the composite plate, the untreated and treated fibres were wrapped around a steel plate with a thickness of 2 mm using a lathe. To perform this operation, the metallic plate was first greased with release agent and then fixed in the chuck. Next, the spindle was set in very slow rotation, wrapping the fibres around the plate. During the process it was ensured that the fibres were very close and aligned in one direction. Subsequently to this operation, the metallic plate with the fibres was immersed in polyester resin (99% resin with 1% catalyst) during approximately 30 s, and while unpolymerized, placed into a two-part mould. This mould was placed on a press table and a pressure of 1 MPa [27] was applied on the composite plate. After 12 h of polymerization in the press, the composite was removed from the mould and the metallic plate was taken away from the middle of the composite. By cutting the composite set in the same plane as the direction of the fibres, it was possible to obtain two composite plates, as seen in Fig. 1.

The specimens were cut from the composite plates using a laser system (X252 from GCC). The dimensions and geometry of specimens were chosen according to the ASTM D 3039 M standard [28]. In Fig. 2, it is possible to observe some examples of the specimens used for the tensile tests. For each composite plate, corresponding to a combination of the different factors and levels, three specimens were cut, to give a total of 18.

The alkali–silane treatment of fibres was done in two steps. The first step was the alkali treatment using sodium hydroxide (NaOH) solutions with concentrations of 2 and 5% by mass. Hemp and flax fibre mats were immersed in the NaOH solution for 24 h at a temperature of 45 °C. After this period, the fibres were washed with tap water and submerged in distilled water that contained 1% acetic acid to neutralize

**Fig. 1** Two composite plates obtained after the manufacturing process



**Fig. 2** Composite specimens for tensile tests

the residual NaOH. These fibres were then dried in an oven at 45 °C during 12 h. The second step was the silane treatment. For this treatment, a solution of 5% 3-aminopropyltriethoxysilane by weight (weight of silane relative to the weight of hemp or flax mat) diluted in a 50% aqueous solution of methanol was used. The pH of the solution was kept between 4 and 5 using acetic acid. The mats were submerged in the solution for 4 h at 28 °C. After that period, they were washed with distilled water and finally dried in an oven at 45 °C for a period of 12 h.

After manufacturing the specimens, the tensile tests were performed using a universal test machine (Instron 4485). The specimens were fixed in the machine grips and the tensile test was performed at a speed of 1 mm/s. For each experiment (Table 1), three tensile tests were executed, corresponding to a total of 18 trials.

### 3 Results and Discussion

In Table 4 and Fig. 3, it is possible to observe the average of the results obtained. We can see that Experiments 1, 5, and 6 give higher values of tensile strength, especially Experiment 6, in which a flax composite was tested using the alkali-silane surface treatment with 5% NaOH concentration.

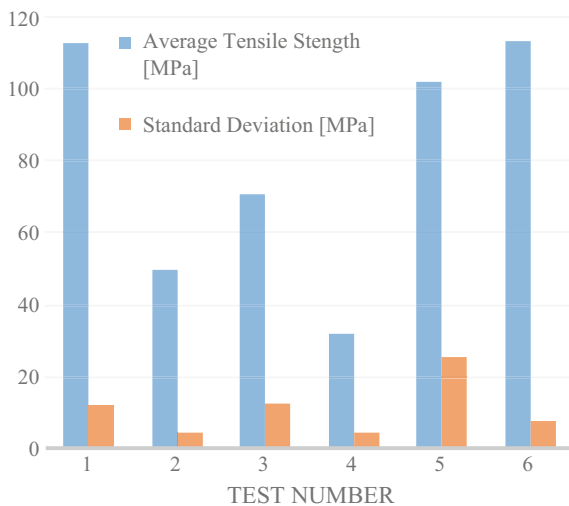
In Table 4, is also possible to verify that Experiment 5 has the highest value of standard deviation (25.4 MPa), which means that the data are widely spread (less reliable), while Experiments 2, 4, and 6 have relatively low values of standard deviation for the NFCs: 4.6, 4.7, and 7.8 MPa, respectively; in other words, the experimental data are closely clustered around the mean (more reliable).

Using the data presented in Table 4, it is possible to obtain an analysis of variance (ANOVA). The ANOVA is a group of statistical models used to analyse the changes among group averages and their associated procedures, developed by Ronald Fisher [24]. Table 5 presents the ANOVA for the tensile tests performed.

**Table 4** Experimental results: average tensile strength and standard deviation

Experiment	A	B	Average tensile strength (MPa)	Standard deviation (MPa)
1	1	1	112.8	11.8
2	2	1	49.7	4.6
3	1	2	70.8	12.7
4	2	2	32.2	4.7
5	1	3	102.0	25.4
6	2	3	113.4	7.8

**Fig. 3** The average tensile strength and standard deviation for each tensile test



**Table 5** ANOVA results

Source	DF	Adj SS	Adj MS	F-Value	Percentage contribution
A—Type of fibre	1	1358	1358.1	1.89	22.8
B—Surface treatment	2	3157	1578.6	2.19	53.0
Residual	2	1441	720.3		24.2
Total	5	5956			

In this table, DF refers to the degrees of freedom, Adj. SS is the sum of squares, and Adj. MS is the mean squares. The F test is a statistical tool to determine which parameters affect the quality more significantly. Observing Table 5, it turns out that the chemical surface treatment of the fibre has the largest influence on the tensile strength, with a contribution of 53.0%.

## 4 Conclusions

The influence of two types of fibres and alkali–silane surface treatment on the tensile strength of NFCs was studied in this work. An array of experiments was implemented using three levels for the surface treatment and two levels for the types of fibres.

To determine the tensile strength of NFCs, a total of 18 tensile tests were performed. From these tests it was verified that the combination that gives the highest value of tensile strength is the flax composite associated with the alkali–silane surface treatment with an NaOH concentration of 5%, and for this case the average tensile strength was 113.4 MPa. Based on the experimental results and using the ANOVA approach, it was determined that the most influential factor for maximizing the tensile strength was the alkali–silane surface treatment, with a contribution of 53.0%.

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