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An efficient enzymatic-based process for the extraction of high-mechanical properties alfa fibres

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ABSTRACT

Alfa fibres, which are generally extracted from the leaf of a plant belonging to the Poaceae family (*Stipa tenacissima* L), originating from the center of Tunisia, are mainly used for pulp and paper applications. Their potential use as reinforcement in polymer composites requires the understanding of their microstructure and mechanical properties and a proper control of fibre extraction and transformation processes. This work investigates the morphology of the alfa plant (leaves and fibres) through optical and electron microscopy. The extraction process combining mechanical, chemical and enzymatic stages and the reaction time of the enzymes have been optimised to achieve the highest mechanical properties of fibres. The effect of enzymatic treatments (laccase, pectinases and xylanases) on the morphological, chemical composition and mechanical properties of alfa fibres was investigated and the effectiveness of enzymatic treatments has been evaluated. The chemical compositions of alfa are correlated with its mechanical properties. The result indicates that the tensile properties of isolated fibres were greatly improved when an optimised enzymatic-based process is used to separate the fibres from the leaves. Using pectinase and xylanase activities, results show really high mechanical properties, with an average rigidity and strength up to, respectively, 66 GPa and 1300 MPa, which make alfa fibre promising reinforcements for load-bearing composite materials. This work also showed that enzymes offer an attractive and eco-friendly approach to efficiently extract high-performance plant fibres.

1. Introduction

In Tunisia, *Stipa tenacissima* also known as Arab name alfa is located in the centre of the country, where it covers about 3500 km^2 with an annual production of approximately 60.000 t (Ben Brahim and Ben Cheikh, 2007). Alfa plant farming is relatively eco-friendly since it does not need insecticides and pesticides and only a small amount of water is necessary for its growth. This species plays an important role in social, economic, cultural and industrial development. The alfa leaves have long been used for crafts such as cordage and objects of sparterie (objects made out of rough fibres such as plaiting materials, basketware and wickerwork), alfa germinated seeds may be consumed by humans, the alfa leaves have unsaturated fatty acids, including oleic acid and linoleic acid (Mehdadi et al., 2000), which could be valued in the dietary domain and younger alfa leaves can be grazed by horses, camels but it is too rich in lignin for other herbivores. It also constitutes an essential factor in the pastoral balance and desertification fighting. With its powerful root system, it prevents land degradation and soil erosion caused by wind and water dry land region (Zeriahène, 1987).

Alfa fibres extracted from the leaves are mainly used for pulp and paper applications. The Tunisian company SNCPA (National Company of Cellulose and Alfa Paper) produces about 40.000 t of alfa pulp per year from alfa plant. Thirty percent of this production is exported to Europe, Asia and the United States of America. The alfa fibres are generally extracted from the leaves by the sodium hydroxide process which consists in cooking the leaves in NaOH solution, followed by the bleaching of the obtained fibres in bioxide chloride solution or hydrogen peroxide solution (Bouiri and Amrani, 2010). The extraction process provides access to short,

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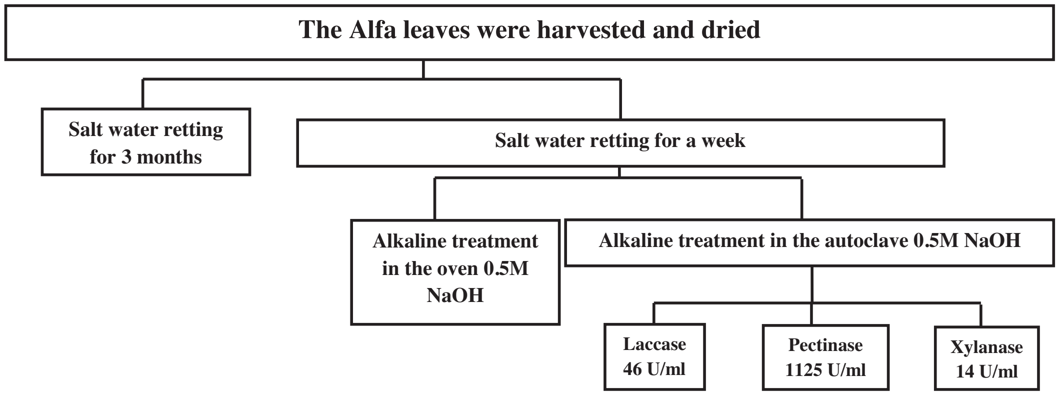

Fig. 1. Representative diagram of the selected and tested strategies for the fibres separation and extraction.

fine and white fibres and then a high quality pulp. This process also leads to a degradation of the cell wall, which is detrimental for the mechanical properties of the isolated fibres. The experimentally determined properties are in the order of magnitude of 5–20 GPa and 50–250 MPa, respectively, for the tensile rigidity and strength according to literature (Ben Brahim et al., 2007; Dallel, 2012). Bessadok et al., 2009 show that high-mechanical properties can be reached (tensile strength greater than 1400 MPa and Young's modulus up to 70 GPa) if the defibration process is optimised.

In pulp and paper application, the mechanical potential appears to be poorly exploited. An expected step for Maghreb countries is to attract industrial interests towards the use of alfa fibres in high added value applications, such for textiles and composite materials. In fact, some studies and applications have dealt with the elaboration of composites. Ben Brahim et al. (2007) have reported that mechanical properties of the alfa/polyester composites are, for the same weight, close to those measured on composite reinforced with synthetic fibres such as glass fibres. In the case of textile applications and for the first time, Dallel, 2012 produced yarns based alfa fibres (up to 90%) by the conventional ring spinning method. These yarns have an average tenacity of 6.5 CN/tex and a relative elongation of 4.3%. Before considering such applications, an extensive mechanical characterisation work as a function of the fibre extraction process has to be performed. Alfa fibre has been far less studied in comparison to other plant fibres and its morphology, biochemical composition, ultra structure and mechanical behaviour are far from being fully understood.

Very few researches have concerned the analysis of alfa fibre quality and, as far as we know, none of them concerned the comparison of different extraction methods and their effects on fibre mechanical properties. The well-known processing methods for flax and hemp could probably be adapted for alfa: they include water or microbiological retting plus mechanical scotching or sometimes enzymatic processing (Akin et al., 2001; Kashyap et al., 2001; Hoondal et al., 2002; Van Sumere, 1992). These controlled methods are more expensive than dew retting but usually produce better quality fibres (Bacci et al., 2010) and a better reproducibility of the fractionation level.

The use of enzyme technology is becoming increasingly substantial for the processing of natural fibres (George et al., 2014; Liu et al., 2012; Alix et al., 2012). A major reason for embracing this technology is the fact that the application of enzymes is environmentally friendly. The reactions catalyzed are also very specific and have a focused performance (Bledzki et al., 2010).

The main purpose of this work is to implement eco-friendly process based on enzymatic treatment on alfa fibres, in order to extract and separate the fibres from the leaves and to preserve their native mechanical properties. In first time, we analyse the fibres morphologies; using electronic and optical microscopy for the observation of the transverse and longitudinal cross-sections of alfa leafs. Then, we propose, in the second time, an eco-friendly process for the fibres extraction.The effectiveness of enzymatic treatments has been evaluated using four main criteria: (i) determining the amount of solubilized sugars after enzyme incubations, (ii) by observing bleachability, (iii) observing the fibre separation level, (iv) determination of the chemical composition of the fibres extracted and (v) measuring the mechanical properties of isolated fibres.

Tensile tests were performed for each extraction step on isolated alfa fibres in order to determine their mechanical performances and evaluate the severity and efficiency of the process.

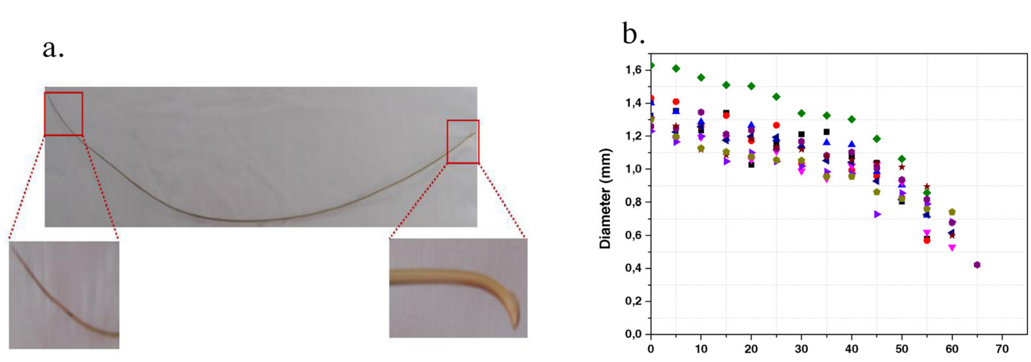

Fig. 2. Variation of the diameter along the length of the leaf. (a) Optical picture. (b) Measurement performed on 7 leaves every 5 cm along the length.