

## THE STRUCTURE AND MECHANICAL PROPERTIES OF SPINES FROM THE CACTUS *OPUNTIA FICUS-INDICA*

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The mechanical properties and structure of cactus *Opuntia ficus-indica* spines were characterised in bending and by means of x-ray diffraction. Using spruce wood cell walls for reference, the modulus of elasticity of *Opuntia* cactus spines was high in absolute terms, but comparable when specific values were considered, which can be explained by similarities in the cell wall structure of both materials. Differently from the modulus of elasticity, the bending strength of cactus spines was unexpectedly high both in absolute and in specific terms. The unique cellulose-arabinoxylan composite structure of cactus spines, together with high cellulose crystallinity, may explain this finding.

*Keywords:* Cactus spines; Cellulose; Stiffness; Strength

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### INTRODUCTION

Bio-based materials with unusual or extreme properties are of interest to materials scientists who look for possibilities to transfer construction principles found in nature to technical applications. The outstanding strength and stiffness of cactus spines, for example, are obvious and well known – often through painful experience (Lindsey and Lindsey 1988; Doctoroff et al. 2000, Jansen et al. 2005). However, quantitative information on these properties and their relation to structure is scarce. Schlegel (2008) conducted three-point bending tests with spines of 55 cactus species and reported values for the modulus of elasticity from less than 1 GPa up to 12.7 GPa. Tensile tests with dry *Opuntia ficus-indica* spines yielded an average modulus of 6 GPa and a strength of 84 MPa (Malainine et al. 2003). As these rather modest values do not correlate with the physical impression incited by touching cactus spines with ones own hands, additional mechanical tests with *Opuntia ficus-indica* spines were performed in the present study and related to selected structural features.

### EXPERIMENTAL

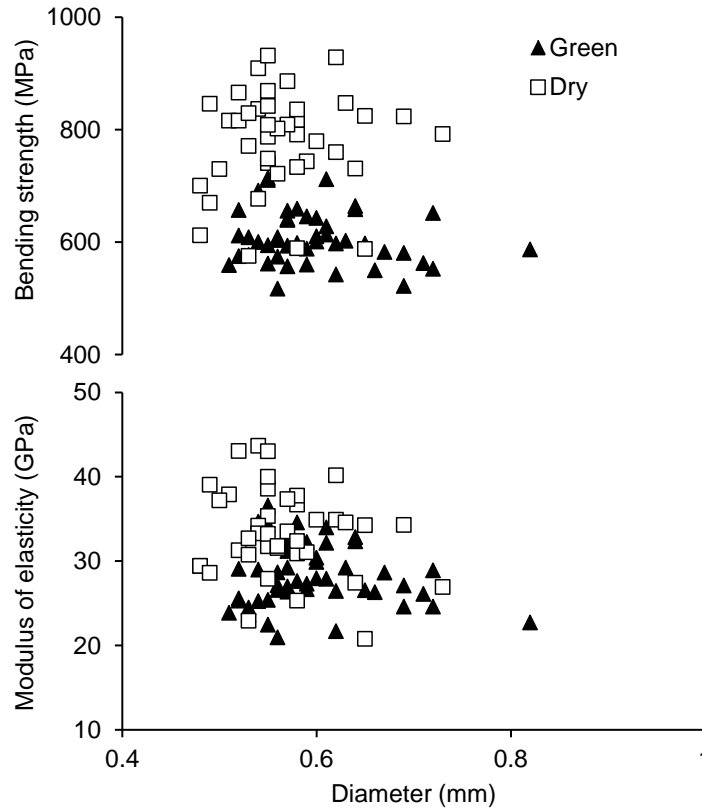
Fully developed spines were collected from an approx. 80 cm tall living *Opuntia ficus-indica* plant. While a few specimens were set aside for structural characterisation, 50 specimens (“green” fully developed specimens with native moisture content) were tested in three-point bending on the same day. Another 50 specimens were dried in an

oven at 103°C for two days and tested with the same set-up (dry specimens). A gravimetric determination of moisture content resulted in 24% for green specimens and 0% for dry specimens. Mechanical tests were performed on a Zwick Roell universal testing machine equipped with a 50 N load cell. The tested specimen length, i.e. the distance between the supports, was 12 mm, and the cross-head speed was 1 mm min<sup>-1</sup>. The bending tests were evaluated assuming an elliptical cross section for spines, in a way that the smaller diameter of the ellipse was assumed to be oriented vertically, i.e. parallel to the load direction. On average, the smaller diameter of the tested spines was  $0.58 \pm 0.061$  mm and the larger diameter was  $0.82 \pm 0.099$  mm. For light microscopic characterisation, small pieces of cactus spines were embedded in epoxy resin and sectioned with a Leica ultracut R microtome equipped with a diamond knife. Microscopy was done with a Zeiss Axioplan research microscope in incident light mode. Wide angle x-ray diffraction was performed with a Bruker Nanostar system. 2D detector images were acquired, and evaluated using Fit2D software ([www.esrf.eu/computing/scientific/FIT2D/](http://www.esrf.eu/computing/scientific/FIT2D/)).

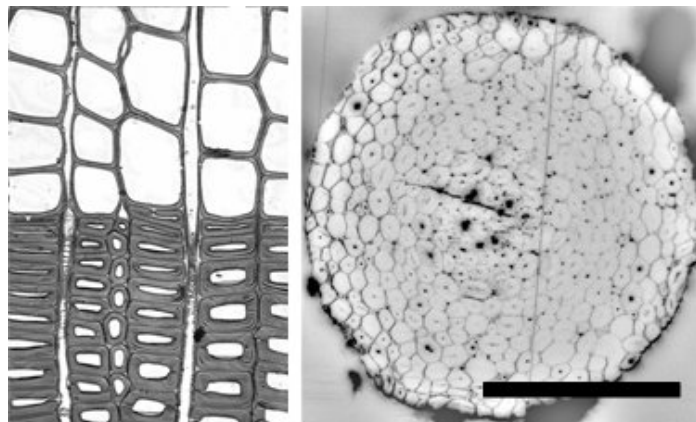
## RESULTS AND DISCUSSION

The results of three-point bending tests for green and dry *Opuntia* spines are shown in Fig. 1. With an average modulus of elasticity of  $28.0 \pm 3.66$  GPa and bending strength of  $609 \pm 48.1$  MPa (green), and  $33.5 \pm 5.15$  GPa and  $779 \pm 87.7$  MPa (dry), respectively, very high values were measured, compared to earlier studies (Malainine et al. 2003; Schlegel 2008). However, an analysis of the structure of cactus spines compared to spruce wood, a well studied reference material, shows that the magnitude of our values is very reasonable. Taking the example of spruce wood shown in Fig. 2, it is obvious that the average density in an *Opuntia* cactus spine is much higher than that of wood. At a diameter of 5 to 10 µm, only a very small lumen is left in cactus spine cells, whereas the diameter of wood cells ranges between 10 to 40 µm, with a cell wall thickness of 1 to 5 µm. Wood shows high variability in density within and between different species, which is largely determined by the thickness of the cell wall relative to the cell diameter, and in concurrent mechanical properties. Since most mechanical properties of wood, including bending strength and modulus of elasticity, rise in a linear fashion with increasing density (Panshin and de Zeeuw 1980), it is possible to normalise mechanical properties to a specific, density-independent value for comparison. By dividing the neat wood cell wall density ( $1.5 \text{ g cm}^{-3}$ ) by the density of spruce wood ( $0.43 \text{ g cm}^{-3}$ ), a scaling factor of 3.5 is obtained. By multiplying values of 11 GPa for the modulus of elasticity and 71 MPa for bending strength of average spruce wood (values for wood conditioned to 12 % moisture (Sell 1989)) with the scaling factor, a specific cell wall modulus of 38.5 GPa and a specific bending strength of 248 MPa are obtained. By determining the mass and volume of small pieces of cactus spine, again assuming an elliptical cross section, a density of  $1.3 \text{ g cm}^{-3}$  was calculated. Based on the fact that the cell wall polymers lignin, hemicellulose, and cellulose show a similar density of approx.  $1.5 \text{ g cm}^{-3}$  (Fengel and Wegener 1989) it may be assumed that the cell wall density of cactus spines, which consist primarily of cellulose and a particular hemicellulose (Vignon et al. 2004) is in the same order as wood. Thus a scaling factor of 1.15 is obtained, resulting in a specific modulus of 32 GPa

(green) and 38.5 GPa (dry), and a specific bending strength of 700 MPa (green) and 896 MPa (dry), respectively. These values correspond well with spruce wood with regard to the specific modulus of elasticity, but the calculated specific bending strength of cactus spines is more than twice as high as the corresponding value for wood. A study of the structure of both spruce wood and cactus spines by means of x-ray diffraction helps to explain this discrepancy.

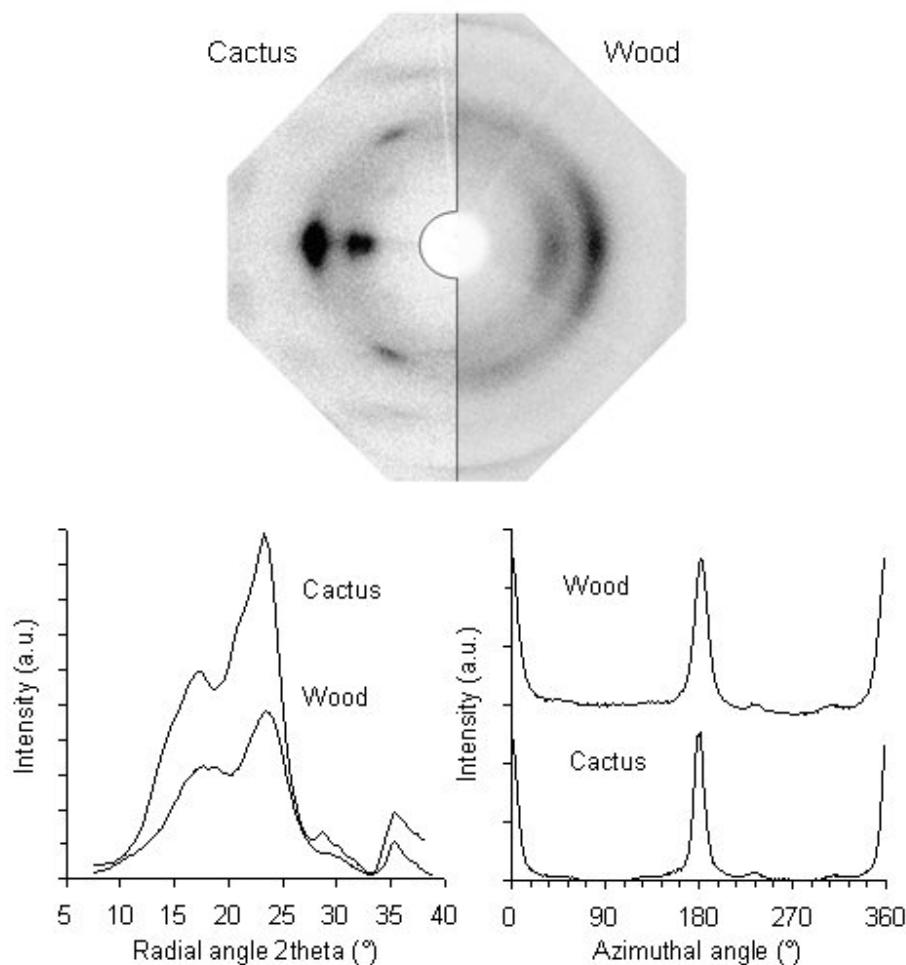


**Fig. 1.** Bending strength and modulus of elasticity for green and oven-dry *Opuntia* cactus spines plotted versus diameter. In the context of this figure the term green stands for fully developed green spines with a moisture content as received immediately after harvesting from the living plant.



**Fig. 2.** Light micrographs of spruce wood (left) and an *Opuntia* spine (right) in cross section (bar valid for both images = 100  $\mu\text{m}$ )

The results of wide angle x-ray diffraction with a *Opuntia* cactus spine and spruce wood are shown in Fig. 3. The orientation of cellulose in the cell wall of the specimens was evaluated from the azimuthal scattering intensity distribution of the cellulose 200 reflection, which is the most intense reflection in cellulose, and is oriented normal to the direction of the cellulose chain. As seen in Fig. 3 (bottom left), the azimuthal intensity distribution curves were very similar for cactus spine and spruce wood, showing sharp peaks, which indicate a high degree of cellulose orientation.



**Fig. 3.** Top: 2D detector images from wide angle x-ray diffraction with a cactus spine (left image half) and a piece of spruce wood (right image half). Bottom left: Azimuthal integrated scattering intensity distribution along the cellulose 200 reflection, which is the most intense reflection of cellulose, for a cactus spine and wood. Bottom right: Radial integrated scattering intensity distribution for a cactus spine and wood.

The degree of preferred orientation was quantified by means of Herman's orientation factor  $f$ , which ranges from 0 for random orientation to 1 for perfect uniaxial orientation (Gindl et al. 2006). In the case of spruce wood and cactus spine, similar orientation factors of 0.57 and 0.58, respectively, were obtained. It is a well established fact that the modulus of elasticity of polymeric fibres depends on the modulus of the constituent polymeric chains and on their degree of orientation with respect to the fibre

axis (Northolt et al. 2005). Since the cellulose content of both spruce wood and *Opuntia* cactus spines is in an order of 50 % (Malainine et al. 2003; Fengel and Wegener 1989) and the degree of orientation is almost identical, the similarity of the determined specific bending moduli for both materials, i.e. 35.8 GPa for conditioned spruce wood and 32 GPa (green) to 38.5 GPa (dry) for cactus *Opuntia* spines is very reasonable.

With regard to specific strength, outstanding values of 700 MPa (green) to 896 MPa (dry) were found for cactus spine compared to spruce wood (248.5 MPa). The very high values measured for cactus spine indicate that additional factors apart from cellulose content and cellulose orientation, which suffice to explain similar moduli, have to be considered in an analysis of strength. In a detailed study of the chemical composition and molecular structure of *Opuntia* spines, Vignon et al. (2004) revealed that the chemical composition of cell walls from *Opuntia* cactus spines is very different from wood, which roughly consists of 50% cellulose, 25% lignin, and 25 % non-structural polysaccharides. By contrast, the cell walls of *Opuntia* spines are formed by a unique composite of 50% cellulose and 50% arabinan, which interact strongly with each other at the molecular level (Vignon et al. 2004). In addition to this significant difference in the composite structure of cactus spine and spruce wood cell walls, x-ray diffraction performed in the present study revealed an additional structural feature that possibly contributes to the high strength of cactus spine. Radial scattering intensity distribution curves of spruce wood and cactus spine (Fig. 3, bottom right) provide clear evidence of higher crystallinity in cactus cellulose compared to wood, inferred from significantly higher scattering intensity for cactus. A crystallinity index was calculated for both materials from the ratio of highest scattering intensity corresponding to the cellulose 200 reflection, to the scattering intensity at an angle of 33°, where negligible crystalline scattering is assumed. This crystallinity index is 57 for cactus, compared to 34 for spruce wood.

## CONCLUSIONS

1. Cactus *Opuntia ficus-indica* spines show a modulus of elasticity comparable to wood, but their bending strength is unusually high. This observation correlates with high cellulose orientation and high crystallinity in the cactus spine fibre cells observed by means of x-ray diffraction.

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