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Submitted on 15 Nov 2013

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Eprints ID: 10102

**To link to this article**: doi: 10.1021/la4006086
URL: [http://dx.doi.org/10.1021/la4006086](http://dx.doi.org/10.1021/la4006086)

**To cite this version**: Ledesma-Alonso, René and Legendre, Dominique and Tordjeman, Philippe. AFM tip effect on a thin liquid film. (2013) Langmuir, vol. 29 (n° 25). pp. 7749-7757. ISSN 0743-7463

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On the air permeability of Populus pit

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Keywords: pit; air flow; Populus; AFM; Young’s modulus

1. Introduction
Sap hydrodynamics in vascular cells of trees seems to be controlled by small membranes called pits. Understanding how the pit junctions regulate the sap flow and stop embolism by cavitation is today a challenging issue. The hypothesis that the pit porosity adjusts the flow under negative pressure and stops the air bubble diffusion need to be validated. In this talk, we will present the experimental results on Populus trees that support the idea that pits operate ‘passively’ in a biological point of view. This work is based on atomic force microscope (AFM) experiments, which have been realised to measure quantitatively the mechanical properties of pits at the nanoscale.

2. Methods
Populus branches were obtained from the PIAF laboratory (INRA) at Clermont-Ferrand.

In the first step, we developed an experimental method (Figure 1) to measure the critical pressure that allows the air flow in the vascular cells, which is an intrinsic property of pits. Indeed, the latter is the lowest air flow pressure that corresponds to the capillary pressure of the pit pores at the interface between air and water phases (Choat et al. 2003): 

\[ P_c = \frac{2 \sigma \cos \theta}{R}, \]

where \( \sigma \) is surface tension of water; \( \theta \) is the contact angle at the air–water interface and \( R \) is the pore radius of pits. In this experiment, the Populus branches fixed by special clamps were connected to a bottle of air under pressure. A pressure transducer controls the air pressure injected in the branches at one extremity, the other being submerged in a water recipient.

In the second step, we characterised the pit microstructure by AFM in tapping mode. The samples were prepared by cutting the branches with a microtome. An Agilent 5500 Scanning Probe Microscope of FERMAT was used with scanning tips of Nanotools® model ACT (single-crystal silicon) with a radius of 10 nm. We developed new AFM experiments to measure Young’s modulus \( E \) of the cell wall, the pit wall (PW) and the pit membrane (PM) and the viscoelastic behaviour of pits swelled by water. The first method is an indentation method that needs calibration using a silicon wafer and a model material (PMMA). Indentation experiments were realised in contact mode by measuring the force tension \( V_{A-B} \) between the tip and the sample in function of the piezo-displacement. Assuming that the contact is modelled by the Hertz theory, \( E \) is obtained from the following relation:

\[ V(A - B) = \frac{4R^{1/2}E}{3 \alpha k} \delta^{3/2}, \]

where \( \delta \) is the indentation, \( R \) is the tip radius, \( \alpha \) is the force calibration factor and \( k \) is the probe stiffness, respectively.

In order to measure the \( E \) modulus of thin PMs, we developed a second method based on the membrane flexion by probe contact. In these experiments, \( E \) is determined from the curve of the force tension versus the tip displacement \( z \), knowing the membrane thickness \( d \) (Williams 1980):

\[ z = \frac{V(A - B) R^2}{E d^3} \left[ \frac{(2 - \nu)(1 - \nu^2)}{\pi} \right], \]

where \( \nu \) is the Poisson’s ratio of the membrane. Finally, specific AFM experiments were realised to characterise the viscoelastic behaviour of pits in presence of sap (water) by measuring the force–displacement of samples placed in an AFM liquid cell. The data were analysed in the frame of the

Figure 1. Diagram of air flow experiments.
linear viscoelasticity by comparing the raw data to viscoelastic models. To evaluate the accuracy of the measurements, all the experiments were repeated 10 times.

3. Results and discussion

We have found for Populus branches a critical pressure of $18.0 \pm 0.2$ bar. In intensity, this over-pressure is identical with the critical de-pressure measured from the ‘vulnerability’ curves. These latter were established in the PIAF laboratory with the Cavitron (Cochard 2002) and characterised the air bubble propagation in the vascular cells. This result shows that the mechanism of air flow through PMs is passive and is mainly controlled by the porous structure of the PM. From the critical pressure, we found a mean pore diameter $D$ close to 160 nm.

Furthermore, it was possible to measure the permeability $K$ of the Populus branches for $P \geq P_c$; we obtained $K = 3.5 \times 10^{-14}$ and $5.5 \times 10^{-14} \pm 0.18 \times 10^{-14}$ m$^2$ when $P$ changes from 18 to 24 bar.

From the AFM images (Figure 2(a)), we characterised the pit microstructure: the diameter is around 7 $\mu$m, the membrane diameter around 1.5 $\mu$m, the PW thickness close to 1.60 $\mu$m, and the vessel diameter in the branches around 11.2 $\mu$m. On the other hand, pit observations by TEM (Transmission Electron Microscopy) at the PIAF laboratory displayed that the thickness of the PM is around 200 nm.

For dry branches, Young’s modulus of the cell wall was measured by AFM indentation experiments. We found $E = 7.89 \pm 0.39$ GPa. This value corresponds to the radial modulus of the Populus and is in agreement with the values measured at the macroscopic scale (Bjurhager et al. 2008). It was not possible to measure the mechanical properties of the dry PM due to its flexibility. Then, only flexion AFM experiments allow determining the $E$ value. We obtained $E = 3.62 \times 10^2 \pm 3.80$ MPa. Swelled by water, we studied the viscoelastic behaviour of the PM and of the PWs, close and far to the PM (called PW1 and PW2, respectively). We showed that all the force–displacement curves measured by AFM (Figure 2(b)) were correctly fitted by a Zener viscoelastic model with a relaxation time $\tau$ and two Young’s moduli, $E_1$ and $E_2$. The values of Zener elements are summarised in Table 1.

These results show clearly that the swelled pit behaves as a solid viscoelastic. The close values of the Zener elements indicate that composition of the PM and of the PW are similar. Furthermore, the PW1 close to the PM seems to be more rigid (high moduli). Finally, the comparison of Young’s modulus of the PM, dry and swelled by water, shows that the mechanical properties are not very affected by the sorption of water. Taking into account the mechanical properties of the PM and the value of the critical pressure, the pore diameter of the unstrained PM can be calculated: $D = 96$ nm.

4. Conclusion

We have developed specific experiments at macro and nanoscale to study the air flow mechanisms of the Populus pits. The results established that the porous microstructure of the PM governs the air diffusion and, then, the resistance to the embolism.

Table 1. Zener elements of the swelled PM and PWs (PW1 and PW2) determined by contact force AFM experiments.

<table>
<thead>
<tr>
<th></th>
<th>PM</th>
<th>PW1</th>
<th>PW2</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_1$ (GPa)</td>
<td>0.41</td>
<td>1.66</td>
<td>0.43</td>
</tr>
<tr>
<td>$E_1 + E_2$ (GPa)</td>
<td>0.95</td>
<td>3.35</td>
<td>0.84</td>
</tr>
<tr>
<td>$\tau$ (s)</td>
<td>0.99</td>
<td>63.3</td>
<td>6.2</td>
</tr>
</tbody>
</table>

Note: The standard deviations of the fitted values are around 15%.

References


