Characterization of dialyzer membranes by mass-spectrometric imaging techniques

J. Jankowski, K. Krueger, W. Zidek, V. Jankowski

Institute for Molecular Cardiovascular Research, RWTH Aachen

Charité, Med. Clinic IV, Berlin

BACKGROUND

For physical and chemical characterisation of polymers a wide range of analytical methods is available. Techniques like NMR and x-ray are often combined for a detailed characterisation of polymers used in medical applications. Over the last few years, MALDI mass-spectrometry has been developed as a powerful tool for space-resolved analysis, not least because of its mass accuracy and high sensitivity. MALDI imaging techniques combine the potential of mass-spectrometric analysis with imaging as additional spatial information. MALDI imaging enables the visualisation of localisation and distribution of biomolecules, chemical compounds and other molecules on different surfaces.

METHODS

In this study, surfaces of polymeric dialyzer membranes, consisting of polysulfone (PS) and polyvinylpyrrolidone (PVP) were investigated, regarding to chemical structure and compound's distribution. Flat membranes as well as hollow fibre membranes were analysed by MALDI imaging. In accordance with polymer's characteristics analysis parameters like laser intensity and laser raster step size were established firstly to optimize signal intensity and spatial resolution. Best signal quantity and quality and spatial resolution were achieved with settings of 60 µJ laser intensity and 50 µm laser raster step size. The mass spectrometric investigation of both polymers showed clear differences in ionisation behaviour.

RESULTS

The luminal and abluminal surfaces of dialysis membranes differ in pore structure, pore distribution and in polymer composition. Thus, both membrane surfaces were separately analysed by MALDI-FT-Orbitrap mass-spectrometric imaging. Figures 1A and B present scanning electron microscope (SEM) images of flat membrane surfaces (above) and hollow fibre membrane surfaces (below). The left images show the abluminal membrane side with evenly distributed wide pores. The right images display the luminal surface of the membrane oriented to the lumen. The pore diameters of the luminal surface are smaller compared to pores of the abluminal surface. SEM analysis shows pore diameters of 20 to 60 nm for luminal surface and 600 nm to 3 µm for abluminal surface pores.

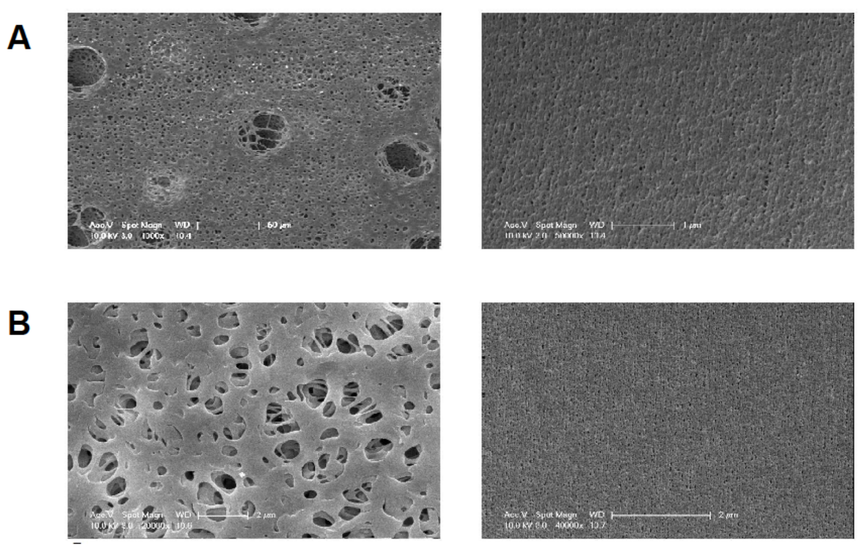

Figure 1: (**A**) Images of abluminal membrane side (left image) and luminal flat membrane surface (right image), (**B**) REM images of abluminal membrane side (left image) and luminal hollow fibre membrane surface (right image) .

The mass spectrum of the abluminal membrane surface utilizing low laser power showed less intensive signals at m/z 907.241, m/z 1349.368 and m/z 1792.496 (Figure 2A). The two-dimensional MALDI image given in Figure 2B shows the localisation and distribution of the polysulfone oligomer (m/z 907.241), accumulated by a laser energy of 60 µJ with a raster step size of 100 µm.

Although mass-signal intensities for polyvinylpyrrolidone were low, we were able to visualize the distribution of the polyvinylpyrrolidone mass m/z 507.295 (Figure 2C). Figure 2D presents the overlay of both images of PS and PVP, showing the different localization of PS and PVP on the identical membrane surface. Whereas polysulfone was evenly distributed the PVP was condensed in clusters.

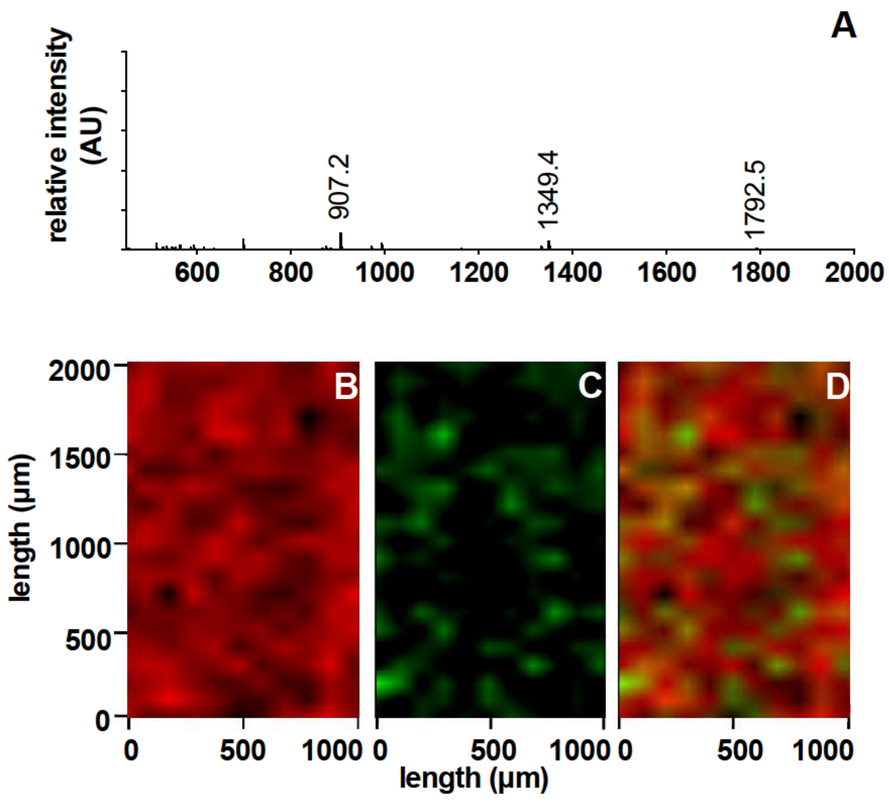

Figure 2: MALDI mass spectra of abluminal membrane surface (A). MALDI images of distribution of (B) PS (red colour) and (C) PVP (green colour) on the same abluminal membrane, obtained with settings of 100 µm grid distance and 60 µJ laser intensity. (D) displays its overlay.

Figures 3A demonstrates the distribution of polysulfone (m/z 907.241), using a laser intensity of 60 µJ and laser grid of 100 µm. Figure 3B shows the MALDI image of PVP mass signal-distribution (m/z 507.295) on the membrane surface. The overlay of these images demonstrates the different distribution of PVP and PS (Figure 3C). PVP is obviously more uniformly distributed than the PS as indicated by the mass signals (Figure 3C).

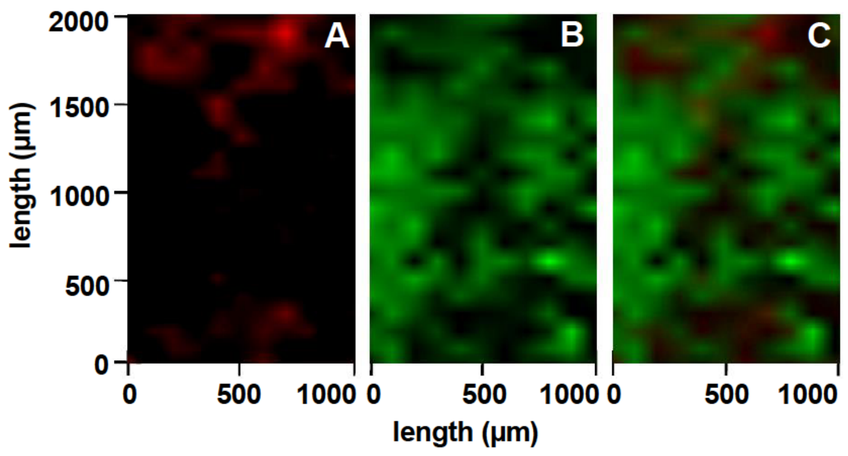

Figure 3: MALDI images of distribution of (A) PS (red colour) and (B) PVP (green colour) on the same luminal membrane, obtained with a grid distance of 100 µm and laser intensity of 60 µJ. (C) represents the overlay of both.

Mass-spectrometry is a powerful tool for investigation of polymers, concerning their molecular masses and mass distribution. The present study focused on MALDI-FT-Orbitrap-imaging as a new method for the characterisation of polymers. MALDI imaging allowed for the first time the simultaneous analysis of composition and distribution of polymers on membrane surfaces. Choosing an optimal matrix and sample preparation, as well as optimizing application parameters is important to obtain high quality mass spectra. It was shown that PS and PVP indicated differences in its ionisation mechanisms. While PS is identified with low laser intensities, a characterisation of PVP is preferentially achievable at high laser power.

CONCLUSIONS

In conclusion, MALDI imaging is a powerful technique for polymer membrane analysis, regarding not only detection and identification of polymers but also localisation and distribution in membrane surfaces.

Contact: jjankowski@ukaachen.de

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Joachim Jankowski

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