

# **(160c) Real-Time Monitoring of Complex Multiphase Behavior in a High Pressure High Temperature Microfluidic Chip**



Microfluidic and Nanoscale Flows: Multiphase and Fields

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## **Real-Time Monitoring of Complex Multiphase Behavior in a High Pressure High Temperature Microfluidic Chip**

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Here, we determined the phase diagrams of aqueous solutions of model biomass compounds in a novel high-pressure-high-temperature microfluidic set-up. We find that the experimental boiling temperature is significantly higher than the theoretical value due to local pressure effects, the energy barrier for bubble nucleation and superheating effects. Furthermore, this set-up provides exquisite control on the point of nucleation by accurate control of temperature.

Good control on the multiphase behavior of aqueous biomass mixtures is of extremely high importance for the biomass conversion industry. Previously, we developed a theoretical model of the saturated vapor pressure of water/biomass model substrate solutions as a function of the temperature and the substrate mole fraction using the AspenPlus software with a Redlich-Kwong-Soave Boston-Mathias (RKS-BM) model. [1] Here, we experimentally validate the calculated vapor/liquid equilibria (VLE) for temperatures between 25 and 200 °C and for pressures between 1 and 10 bar. For this purpose, a microfluidic platform is considered ideal,

as it provides excellent heat transfer properties so that thermodynamic equilibrium is reached fast.

A glass-silicon microfluidic device with a meandering channel (length: 0.4 or 0.2 m; depth: 250  $\mu\text{m}$  and width 250 or 500  $\mu\text{m}$ ) was fabricated using standard microfabrication techniques. A dedicated chip-holder was designed to include the device and heater on the silicon side. Three K-type thermocouples allowed controlling the temperature: two between the heater and the silicon, and one in a 300  $\mu\text{m}$ -wide and 450- $\mu\text{m}$  deep groove on the glass side to determine the heat loss over the device. The pressure was controlled with an adjustable back pressure regulator and pressure meter. Aqueous biomass solutions (10 wt% of ethylene glycol, glycerol, xylose or xylitol) were perfused at a flow rate of 10  $\mu\text{L}/\text{min}$  using a syringe pump. The temperature was increased until gas bubbles were formed as a result of the phase transition.

A significant difference was observed between the theoretical and experimental saturation temperatures; typically experimental values were degrees higher; sometimes saturation could not be achieved. A local pressure increase due to both the thermal expansion of the liquid and the Laplace pressure, superheating effects and possible fouling of the channel wall by hydrocarbon deposits are considered possible explanations for these findings.

The observed multiphase flow pattern indicated that good control of the point of nucleation could be achieved by taking advantage of the pressure drop across the channel and accurate control of the temperature; a higher temperature resulted in nucleation further upstream in the channel. As the void fraction increases downstream, the flow regime changes from a bubbly flow to Taylor flow to annular flow. All three flow regimes were observed simultaneously in a single microfluidic device.

This high-pressure-high-temperature set-up will be further developed to study the mass and heat transfer and kinetics of biomass conversion for sustainable hydrogen production.

## References and acknowledgements

1. 'Influence of the water phase state on the thermodynamics of aqueous phase reforming for hydrogen production' R.M. Ripken, J. Meuldijk, J.G.E. Gardeniers and S. Le Gac, ChemSusChem, under revision

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