

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

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**Abstract :** Controlling the multiphase behavior of aqueous biomass mixtures is essential when working in the biomass conversion industry. Here, the vapor/liquid equilibria (VLE) of ethylene glycol, glycerol, and xylitol were studied for temperatures between 25 and 200 °C and pressures of 1 to 10 bar. These experiments were performed in a microfluidic platform, which exhibits excellent heat transfer properties so that equilibrium is reached fast. Firstly, the saturated vapor pressure as a function of the temperature and the substrate mole fraction of the substrate was calculated using AspenPlus with a Redlich-Kwong-Soave Boston-Mathias (RKS-BM) model. Secondly, we developed a high-pressure and high-temperature microfluidic set-up for experimental validation. Furthermore, we have studied the multiphase flow pattern that occurs after the saturation temperature was achieved. A glass-silicon microfluidic device containing a 0.4 or 0.2 m long meandering channel with a depth of 250  $\mu\text{m}$  and a width of 250 or 500  $\mu\text{m}$  was fabricated using standard microfabrication techniques. This device was placed in a dedicated chip-holder, which includes a ceramic heater on the silicon side. The temperature was controlled and monitored by three K-type thermocouples: two were located between the heater and the silicon substrate, one to set the temperature and one to measure it, and the third one was placed 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 silicon. An adjustable back pressure regulator and a pressure meter were added to control and evaluate the pressure during the experiment. Aqueous biomass solutions (10 wt%) were pumped at a flow rate of 10  $\mu\text{L}/\text{min}$  using a syringe pump, and the temperature was slowly increased until the theoretical saturation temperature for the pre-set pressure was reached. First and surprisingly, a significant difference was observed between our theoretical saturation temperature and the experimental results. The experimental values were 10's of degrees higher than the calculated ones and, in some cases, saturation could not be achieved. This discrepancy can be explained in different ways. Firstly, the pressure in the microchannel is locally higher due to both the thermal expansion of the liquid and the Laplace pressure that has to be overcome before a gas bubble can be formed. Secondly, superheating effects are likely to be present. Next, once saturation was reached, the flow pattern of the gas/liquid multiphase system was recorded. In our device, the point of nucleation can be controlled by taking advantage of the pressure drop across the channel and the accurate control of the temperature. Specifically, a higher temperature resulted in nucleation further upstream in the channel. As the void fraction increases downstream, the flow regime changes along the channel from bubbly flow to Taylor flow and later to annular flow. All three flow regimes were observed simultaneously. The findings of this study are key for the development and optimization of a microreactor for hydrogen production from biomass.

**Keywords :** biomass conversion, high pressure and high temperature microfluidics, multiphase, phase diagrams, superheating

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