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## Experimental investigation on a membrane distillation based micro-separator

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#### ABSTRACT

In this work a novel micro-separator combining the sweep gas membrane distillation principle with micro-fluidic channels was designed and tested for the separation of a mixture of methanol and water with a low to high methanol concentration. The performance of the new separation device was studied with different liquid-vapor/gas membrane contactors with respect to the separation factor and the distillate flux rate by varying the relevant operating parameters of the process like the methanol concentration in the feed (5-70 wt.%), the feed temperature  $(40-65 \,^\circ\text{C})$ , the feed flow rate (up to 30 ml/min), and the flow rate of the inert carrier gas nitrogen (up to 600 ml/min at standard conditions). For all performed experiments, the feasibility of the separation has been proved and the possibility to separate mixtures with high methanol concentration by using a membrane distillation based micro-separator has been for the first time reported. The inert gas flow rate was identified as the crucial operating parameter influencing the separation performance of the micro-separator. In addition, the selection of an appropriate membrane liquid-vapor/gas contactor was found to be an important design parameter for the reduction of temperature polarization effects.

Finally, the novel micro-separator offers the possibility for screening different polymeric hydrophobic/oleophobic micro-porous membranes, showing their potential and limitations as liquid-vapor/gas contactors for the separation of an aqueous mixture with a low to high methanol concentration.

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### 1. Introduction

Over the last two decades important efforts have been made for the development of new micro-separation processes based on distillation [1–6], stripping [7,8], absorption [9], and extraction [10,11]. In all these investigations the feasibility of a separation at the micro-scale has been established and the potential of such systems for process intensification has been shown.

The intensification of distillation processes by using the principle of a heat pipe, better known as zero-gravity-distillation, was first introduced by Seok et al. in 1985 [1]. To improve the mass and heat transfer in the process, these authors proposed to substitute the gravitational forces by the capillary forces which results in increasing the specific interfacial area of mass transfer and hence in the improvement of the separation performance of the process. They demonstrated that a binary mixture of any composition can be separated without using gravity forces and that the calculated height of transfer units corresponds to the ninth of that obtained with a packed column. More recently, this concept was implemented within the micro-channel technology [2,12]. Such systems present, however, important drawbacks. Indeed they are very slow as they do not allow feed flow rates higher than 1 ml/min [1,2]. Furthermore, the stabilization of the liquid–vapor interface for such systems is strongly dependent on the wettability of the mass transfer contactor for the liquid flow.

In this paper an alternative approach to separation by microdistillation by combining micro-fluidic channels with the so-called sweep gas membrane distillation is presented. At normal-scale, this process offers in comparison to conventional distillation and gas stripping processes, significant advantages such as high system compactness and the possibility to operate with low pressure and low temperature heat sources including waste or solar heat. As compared to pervaporation processes it provides higher permeate fluxes [13]. Compared to other membrane distillation configurations, the sweep gas membrane distillation process combines the low conductive heat loss of an air gap membrane distillation process with the reduced mass transfer resistance of the direct membrane distillation [14] allowing therefore, higher permeate fluxes than those obtained in both other cited configurations. Nevertheless, similarly to all other membrane distillation configurations this process is characterized by important temperature polarization effects leading to low evaporation efficiency [14]. Moreover, is the sweep gas membrane distillation process among

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the other membrane distillation configurations the least studied since it requires an external condenser for the recovery of a tiny volume of condensed permeate from a large volume of carrier gas resulting thereby in a low process efficiency [14].

So far, this process has been investigated for water desalination [15,16], concentration of acids [17], purification of dilute wastewater containing volatile organic compounds [18], and the separation of dilute isopropanol–water mixtures [19]. To the best of the authors' knowledge, no application of such process for the separation of liquid mixtures with a high concentration of volatile organic components has been reported.

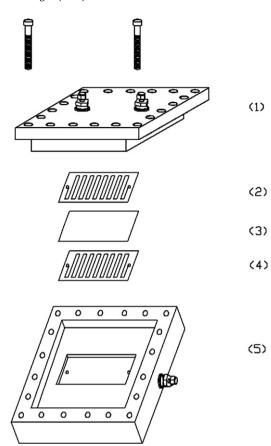
Miniaturization of the sweep gas membrane distillation appears to be a promising route for process intensification. Indeed it may lead to the reduction of the temperature and concentration polarization inside the separation device. Furthermore the use of a small working volume of the inert sweep gas may contribute to the improvement of the distillate (permeate) recovery by using an appropriate micro-fluidic system connected downstream from the permeate channel (micro-separator) for distillate condensation and separation from the inert gas by means for example of capillary forces [20]. Consequently, the external condensation effort required for normal-scale sweep gas membrane distillation process and which constitutes the main drawback of the latter can be greatly reduced.

The aim of this work was to establish the feasibility of a novel micro-separator combining the sweep gas membrane distillation principle with micro-fluidic channels and to test it for the separation of a mixture of methanol and water with a low to high methanol concentration. For the investigated microseparator design the main operating parameters affecting the separation performance have been identified. In addition, several liquid–vapor/gas membrane contactors have been tested with respect to their influence on the separation performance of the micro-separator.

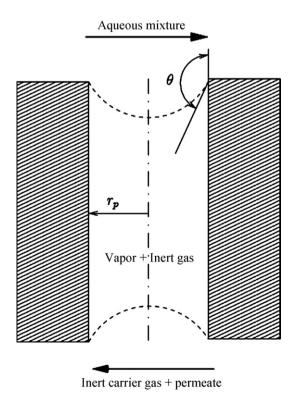
#### 2. Micro-separator

The novel micro-separator consists of two horizontal polycarbonate plates (length: 60 mm, width: 30 mm, thickness: 1 mm) which are joined together holding a flat micro-porous polymeric oleophobic membrane (active area: 171 mm<sup>2</sup>) in between (Fig. 1).

In each plate a meandering rectangular channel is milled (channel hydraulic diameter: 0.677 mm, channel length: 342 mm). Each separation plate is imbedded within a slit machined into the respective cover polycarbonate plate (external dimension:  $110 \text{ mm} \times 110 \text{ mm} \times 30 \text{ mm}$ , internal dimensions:  $80 \text{ mm} \times 80 \text{ mm} \times 20 \text{ mm}$ ) used for mechanical stability, thermal insulation and for visual inspection. Both cover plates are screwed together. The heated feed (subcooled liquid) is circulated through one of the micro-channels and the cold carrier inert gas (dry nitrogen) is circulated through the other one. The two streams can be arranged in co-current or counter-current mode, tangentially to the membrane surface. The membrane serves as a liquid-vapor/gas contactor using interfacial tension to stabilize the liquid-gas interface between the process fluids repelling the liquid phase and creating a liquid-vapor interface at the entrance of the pores. The volatile components (methanol and water) evaporate and diffuse and/or convect through the membrane pores. The inert gas sweeps the permeate distillate through the membrane pores carrying it outside of the device. The membrane does not offer any selectivity for a particular species with respect to another but simply act as a physical separation between the liquid and the gas phase. Fig. 2 shows a cross sectional view of a hydrophobic/oleophobic membrane with straight cylindrical pores in contact with an aqueous mixture (volatile organic compound-water) to illustrate



**Fig. 1.** Exploded view of the micro-separator, (1) cover plate for the permeate channel, (2) permeate channel, (3) membrane contactor, (4) feed channel, (5) cover plate for the feed channel.



**Fig. 2.** Liquid–vapor/gas membrane contactor.

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