Autonomous Phase Separator

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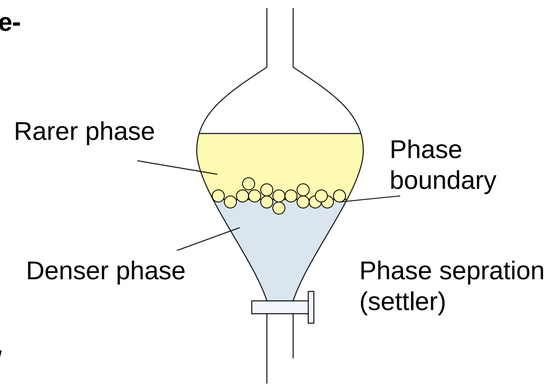
Introduction

Gas- liquid or liquid multi-phase chemistry in micro flow lacks from the unavailability of suitable phase separation equipment, commonly named as settler, mostly in combination as mixer-settler devices used for extraction processes. Until today, phase separators with different configurations are described in literature. The phases are separated, e.g. by membranes, surface modification, capillary forces or free coalescence, and collected by an uncontrolled free effluence[1-3]. These devices can often only be operated in a narrow flow rate window, which is further restricted by an imposed fixed flow rate ratio of the two phases. Additionally, separators that utilize wetting behavior to separate fluid streams are often limited in regard to solvent properties, such as surface tension. One suitable solution is to control the phase-boundary by photoelectric devices and to steer valves in each outlet tubes electronically. Therefore, four optocouplers, consisting of UV-LEDs and phototransistors, are connected to an embedded microcontroller, e.g. an Arduino Uno[4,5]. Such a smart microcontroller provides enough computing power for triggering the optical components and actuating the valves as well as allowing an optionally wireless remote control of the system. A common software, in this case written in Processing, allows a self-regulating operation of the settler, which opens or closes the valves of the two outlets depending on the position of the phase boundary inside the settler. This leads to the most important feature, namely the independence from the feed volume flow, as well as the respective flow rates of the fluid streams, which allows to incorporate the settler into an existing continuous operating synthesis procedure. For example, the continuous extraction of succinic acid in a water/n-butanol mixture is performed by connecting a static micromixer to the settler. Previous experimental results showed that the phase partition almost reaches the thermodynamic equilibrium but it depends on the mixing performance of the used micro mixer, i.e. on the flow rates of the liquids[6]. With an electronically controlled and continuous operating settler an optimization of the extraction process becomes possible.

Common lab-scale phase separation

The typical lab-scale phase separator is an one-outlet separating funnel:

- Open system
- Hand-operated
- Only an outlet for denser phase
- Often no clean separation near interphase

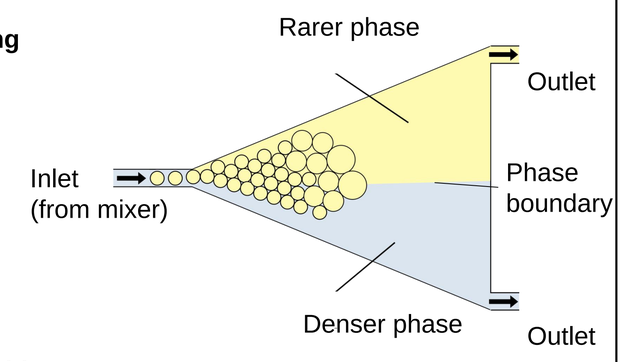


no in-system usage in continuous flow

Common free flow phase separation

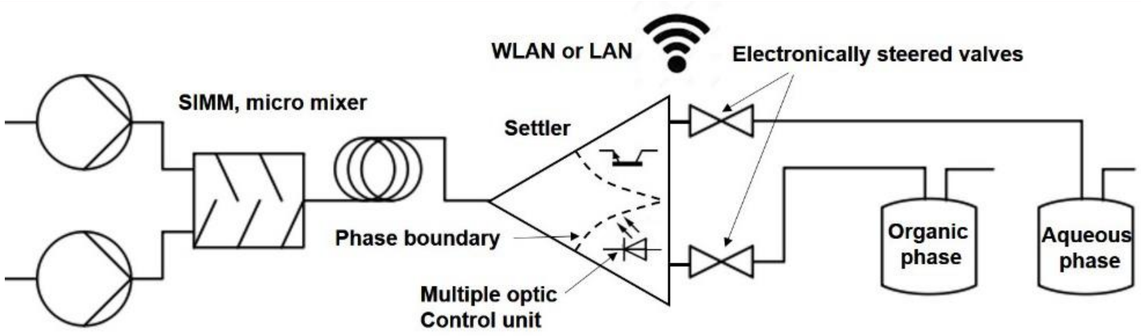
Passive free flow phase separator comes along with a lot of disadvantages:

- Fixed usage for two predefined substances e. g. aqueous and organic phase
- Influence of gravitation on output ratio
- Fixed compound ratio half/half

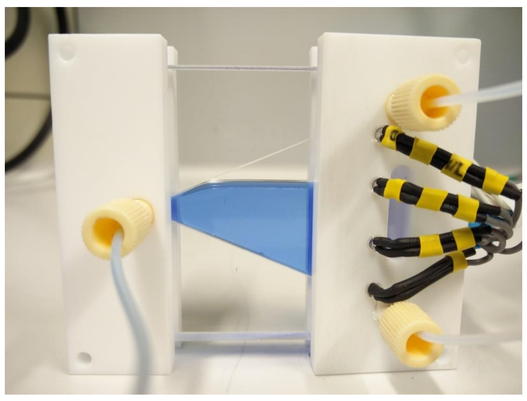


unmanageable, limited, not practicable

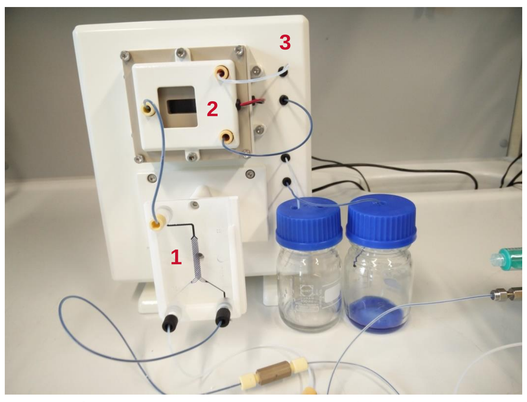
The New autonomous phase separator



- Glass frame triangular phase separator manufactured by Little Things Factory GmbH, Germany
- Four UV-light barriers measuring the transmitted UV-light from four UV-LEDs on the rear
- Two electronically steered valves, connected to each upper and under output
- Light barriers and valves connected to an Arduino UNO[5] which calculates the interphase position and accordingly steer the valves



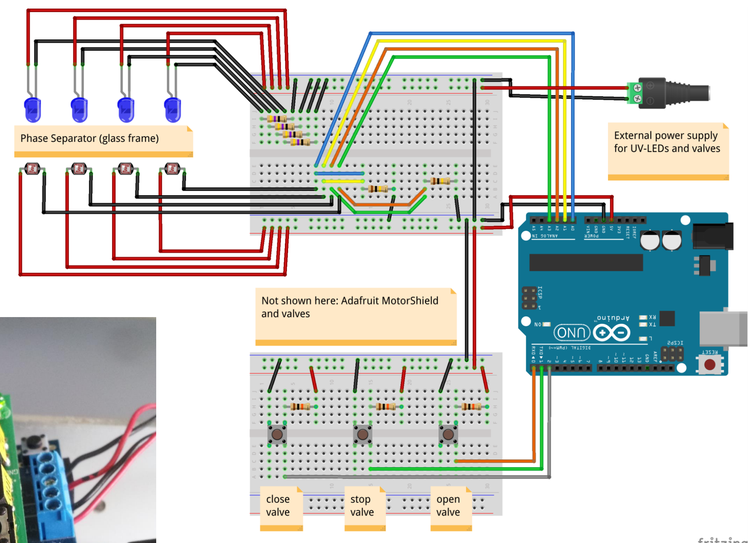
Phase separation of a toluene – water (stained with blue ink) system in the triangular glass frame with 4 UV-light barriers



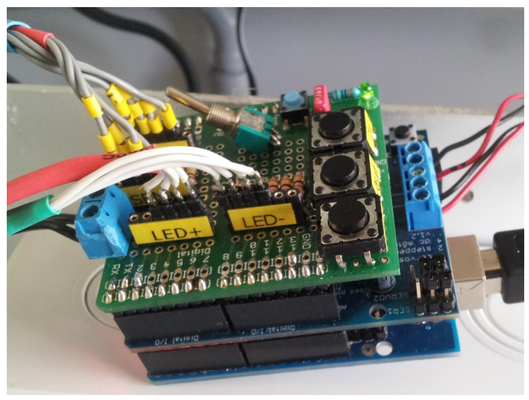
Complete extraction system with mixer (1), phase separator (2) and valve-system (3), mixing and separating toluene – water (blue ink)

Electronics & programming

- Sophisticated Arduino microcontroller platform
- Use of Cheap electronical components
- Easy expandability (more light barriers, LAN/WLAN)

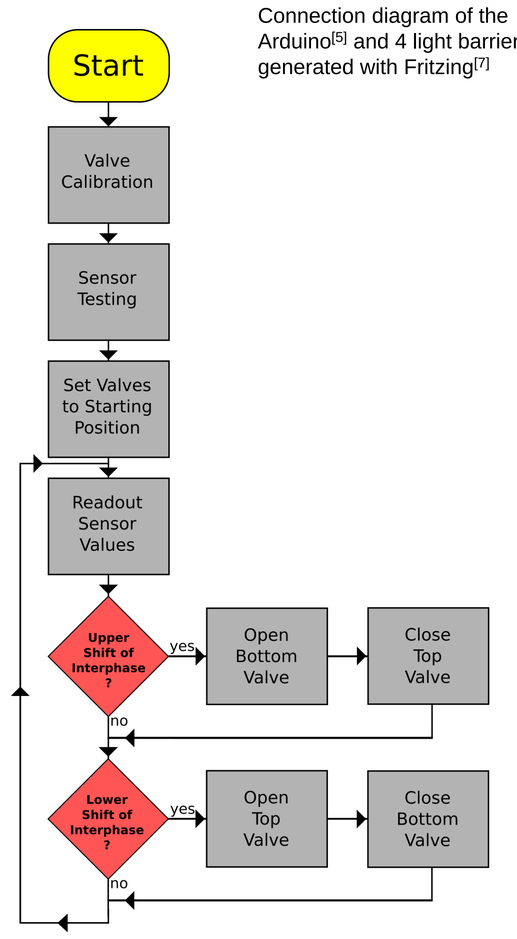


Connection diagram of the Arduino[5] and 4 light barriers generated with Fritzing[7]



The soldered circuit board with its push-buttons, external power supply, sensor- and LED-connectors

- Software written in Processing, a C-like programming language
- Test of valve motors and light barriers during the software initialisation
- A closed infinite loop reading sensor values, comparing with given standard values or the average of previous readings
- A balance of closing the valve next to the interphase and opening the value away from the interphase
- When the interphase reaches the uppermost or undermost light barrier, the valve next to this light barrier closes to avoid contamination of this output with the wrong phase
- Thus even a single-phase only takes predefined output – e. g. while purging an extraction system



Examples for separable liquids

Denser Phase	Rarer Phase	Separable in Phase Separator
Aqueous System	Toluene	yes
Aqueous System	n-Butanol	yes
Aqueous System	n-Octanol	yes
Fluorinated System	Toluene	yes
Fluorinated System	Aqueous System	yes
Acetonitrile	Cyclohexane	no[1]

[1] Due to the humble denstiy difference between Acetonitrile (0.786 g/mL) and Cyclohexane (0.778 g/mL) and its surface tension, the glass frame used is not suitable to separate both liquids

Summary

- An **unattended** in-system phase separation for microfluidic systems, useful for **continuous extraction** systems even in a cascade
- Phase separation even with non-equal compound rates, passed an 8h long-time evaluation with different flow rates
- Novel process making use of **microcontroller technology** for interphase detection and valve steering
- Not restricted to aqueous systems and systems with water as the denser phase. Also a separation of gas/liquid is possible
- Phase separation for reaction/workup/reaction/workup sequencing of **multistep synthesis in closed systems**

References

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