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# Microscale Distillation

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**Abstract**—Vapor−liquid contacting is a key phenomenon encountered in various separation processes, the most commercially important of which is distillation. Although the microprocess technology has been applied on the micro scale for some time, efficient microscale devices applicable for separating components have not been available until past few years. The present review describes the microscale equipment for separation with distillation, with the focus on two operation principles: concurrent and counter-current.

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## INTRODUCTION

Vapor–liquid contacting is a key phenomenon encountered in distillation. Distillation is defined as a method for separating components from a mixture, based on the differences in the volatility of the components. In its simplest form, it is called batch distillation which consists of heating of a liquid mixture to create vapor and withdrawal and condensation of the latter. The continuous form of it is called flash distillation which consists of heating of a liquid stream to create vapor and separation of the two phases. When the two phases flow in a concurrent direction, the separation efficiency is limited to one ideal stage. If a higher separation efficiency is required, the liquid and vapor are brought into counter-current contact. This method is called fractionating distillation, and it is the most commonly used separation method in the chemical process industry.

For the purpose of developing new process concepts it is considered a good engineering practice to test the configuration in on a smaller scale prior the construction of a commercial scale plant. When designing the pilot plant, a separation process utilizing the same separation method should be employed as that planned for the commercial scale process to avoid erroneous scale-up decisions. As research in the field of micro-distillation is rather limited, this requirement may limit the minimization of a pilot plant. Combination of microreactors and microscale separation processes with similar recycles as in the real plant is essential in the piloting phase. In many cases, traces of by-products or impurities in the feed may be present in the process. During initial process development, these may be unrecognized, probably, on account of the fact that their amounts are below the detection limit in once-through reactors. However, in practical processes these impurities may accumulate into the recycle streams with serious consequences to the process. Recognizing these pitfalls is one of the main reasons for the entire piloting phase of process developments.

The distillation methods reviewed here are categorized based on the separation efficiency of a single unit. Although the separation efficiency of single-stage distillation column can be increased by daisy-chaining a number of such units or by recycling the product into the feed flow of the column, the micro-scale in-line pumps required for that are rarely used. Moreover, such a system is difficult to control.

### Concurrent (One-Stage) Microdistillation

Boyd et al. [1] demonstrated separation on the nanoscale. The experimental arrangement consisted of a 30-μm-wide and 5-μm-high microchannel cast in PDMS and sealed with a glass substrate. The glass substrate was coated with gold nanoparticles. The channel was heated with a laser emitting light at wavelenght of 532 nm which exited the nanoparticles in a circular area with a diameter of 10 μm. Liquid at the point of heating was vaporized and condensed 10–20 μm away thus trapping a gas bubble within the liquid. The gas bubble operated as a barrier for

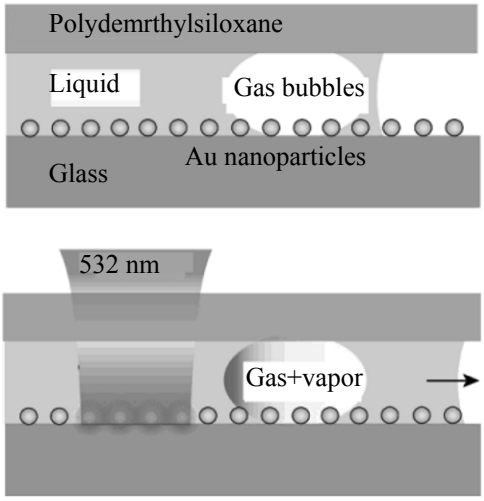


**Fig. 1.** Structure of the microchannel and the principle of separation by bubble-assisted interphase mass transfer [1].

diffusion while allowing for vaporization and condensation across. The process is shown in Fig. 1. The device was tested with a mixture consisting of deionized water, ethanol, and dye mixture. The dye was composed of Coumarin 4 buffered with HCl and tris(hydroxymethyl)aminomethane. During operation only water and ethanol moved across the barrier leaving dye mixture behind, although concentrations were not measured. The rate of vaporization was 0.75 pg/s. The method can be utilized for the preconcentration of temperature-sensitive components, such as biomolecules.

Cypes et al. [2] patented a single-stage flash distillation method consisting of a heated micro-fabricated channel to partly vaporize the feed mixture with subsequent microscopic capillaries to separate the saturated vapor and liquid. The product flow rates were determined by the temperature of the device. The device was manufactured by etching on a silicon plate. The main channel was 500×280 μm$^2$. The length of the channel was at minimum 5 cm, although it could be up to 100 cm to achieve equilibrium. The capillary channels were 10×70 μm$^2$. The temperature of the unit was controlled with an electrical heater. A scheme of the unit is shown in Fig. 2. The device could be operated either as a single unit or as a multitude of modular units, with the product of the first unit being the feed to a subsequent unit. Operation of the device required that the differential pressure over the capillaries was kept below 7 kPa. This was accomplished by controlling the product flow rates with a low-dead volume solenoid valve controlled by a computer with two pressure differential transducers. The device was tested with a binary mixture of pentane and octane. The separation efficiency with a feed flow rate of 0.5 cm$^3$/min was essentially one ideal step. The device was also tested using crude oil as the feed. Results indicated that by operating the device at various temperatures a true boiling point curve could be constructed.

Hartman et al. [3] developed micro-channel distillation column based on the creation of segmented flow with carrier gas. The device was manufactured with etching on a silicon substrate. The microchannel was rectangular with a cross-sectional area of 0.4×0.4 mm$^2$.

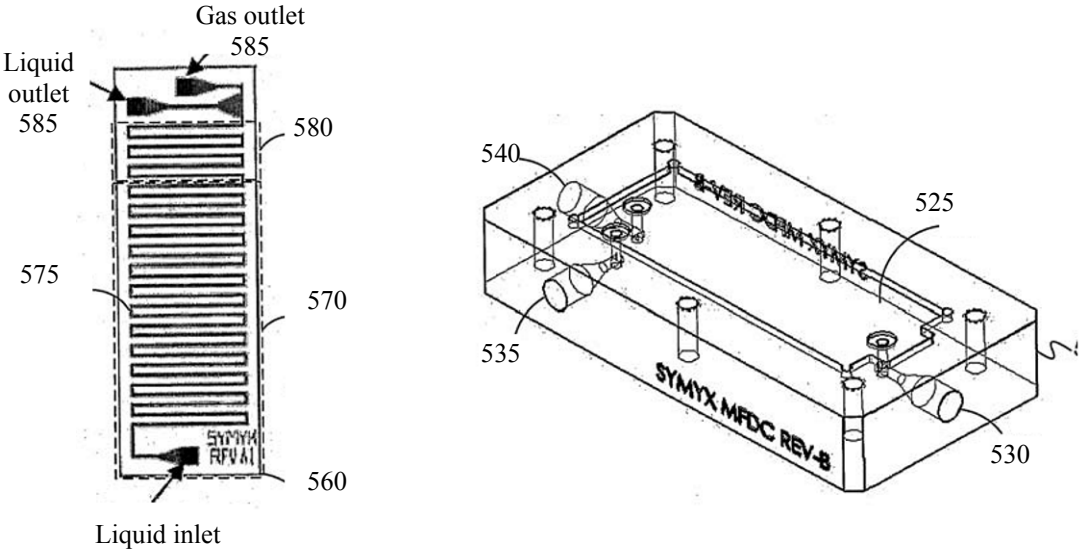


**Fig. 2.** Single-stage distillation device plate and lower half of the supporting framework [2].

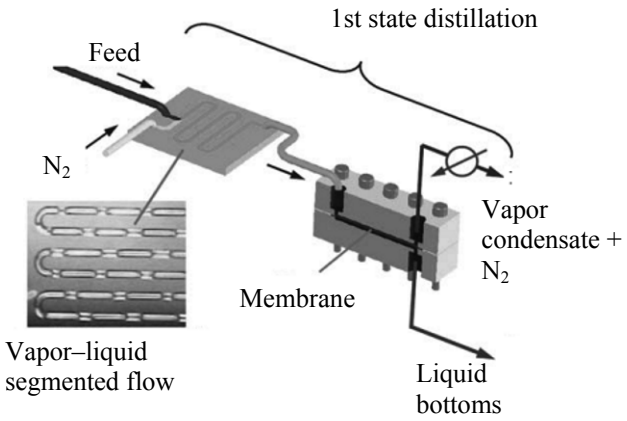


**Fig. 3.** Schematics of the vapor–liquid segmented-flow separation method [3].

The length of the channel was 0.875 m. At the beginning of the channel the feed mixture was fed into a nitrogen stream which operated as carrier gas. The two phases formed segmented flow with liquid slugs flowing between gas bubbles. The flow was heated in the channel with an electrical heater partly flashing the liquid and then separated with the use of a membrane. Schematics of the separation are shown in Fig. 3. Experiments were conducted using binary mixtures of methanol-toluene and toluene-dichloromethane as the feed. Temperature of the device during each experiment was controller based on the feed concentration to obtain desired product flow rates. The separation efficiency was essentially one ideal stage. The operation of the device was further demonstrated as a part of a multistep chemical synthesis [4].

Hibara et al. [5] realized vapor–liquid separation at the nanoscale with the use of nanopillar structures. In the nanopillars, the saturated vapor pressure was less than in microchannel, which caused the evaporated liquid to condense inside the pillar structure. The cross-sectional area of a single pillar was 0.9×0.9 μm$^2$, and the height was 0.25 μm. Capillary channels between the pillars were 0.3 μm wide. The feed was an aqueous ethanol solution with a concentration of 9 wt %. After 2 h the pillar structure was filled with condensate. The nanopillar structure at various times during distillation is shown in Fig. 4. The concentration of the distillate was 19 wt %. With a feed flow rate of 2 μl/min, a separation efficiency of 0.3 ideal steps was observed. The ratio of distillate was 3.7% of the feed flow.

Sotowa and Kusakabe [6,7] developed a microfabricated single-stage contacting device. The channels

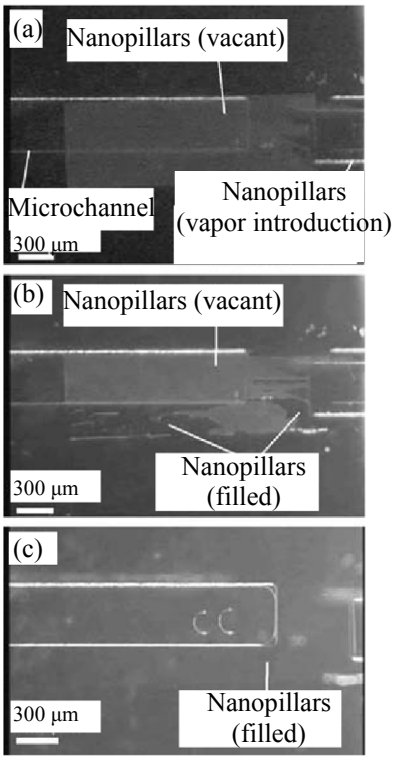


**Fig. 4**. Nanopillar structure with tilted illumination during distillation of the ethanol–water mixture [5].

were machined on a silicon plate which was anodically bonded with glass. The device consisted of mixture inlet followed by a meandering channel for partial vaporization. Vapor and liquid were separated in a flash chamber. The design was similar to that of Cypes et al. [2], except that the separation was based on gravity instead of capillary forces. The vapor phase was created by heating the device in a water bath. Various channel cross-sections between 137×137 μm$^2$ and 500×500 μm$^2$ were manufactured and tested. The channel length was 70 mm. Experiments were conducted with an aqueous methanol solution of 70 mol % at a water bath temperature of 72°C. At feed flow rates of 0.02 and 0.1 cm$^3$/min, the separation efficiencies were 0.8 and 0.65 ideal stages, respectively.

Wootton and deMello [8] presented a single-stage micro-fabricated distillation device using laminar liquid flow and helium as a carrier gas. The channels were etched on a glass substrate and covered with a glass plate by thermal bonding. All channels were 50 μm deep and 100–500 μm wide. The liquid feed mixture entering the device was divided into laminar flow channels that fed the mixture into a carrier gas

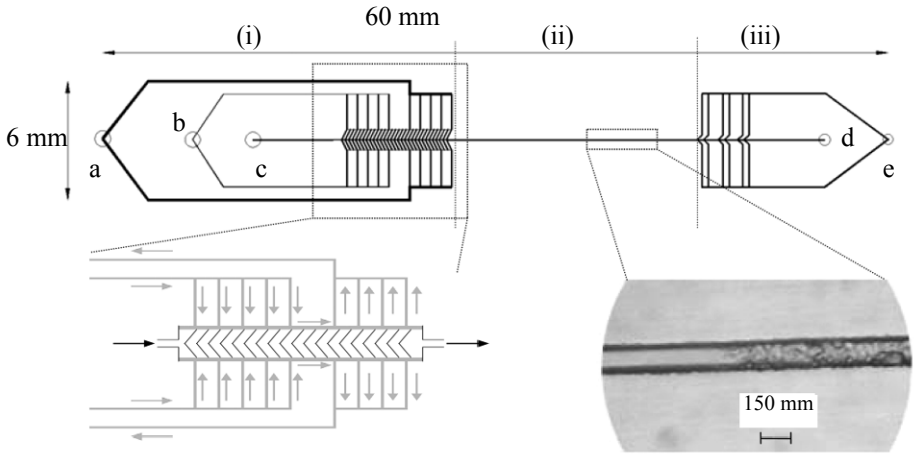


**Fig. 5.** Laminar single-stage separation device [8].

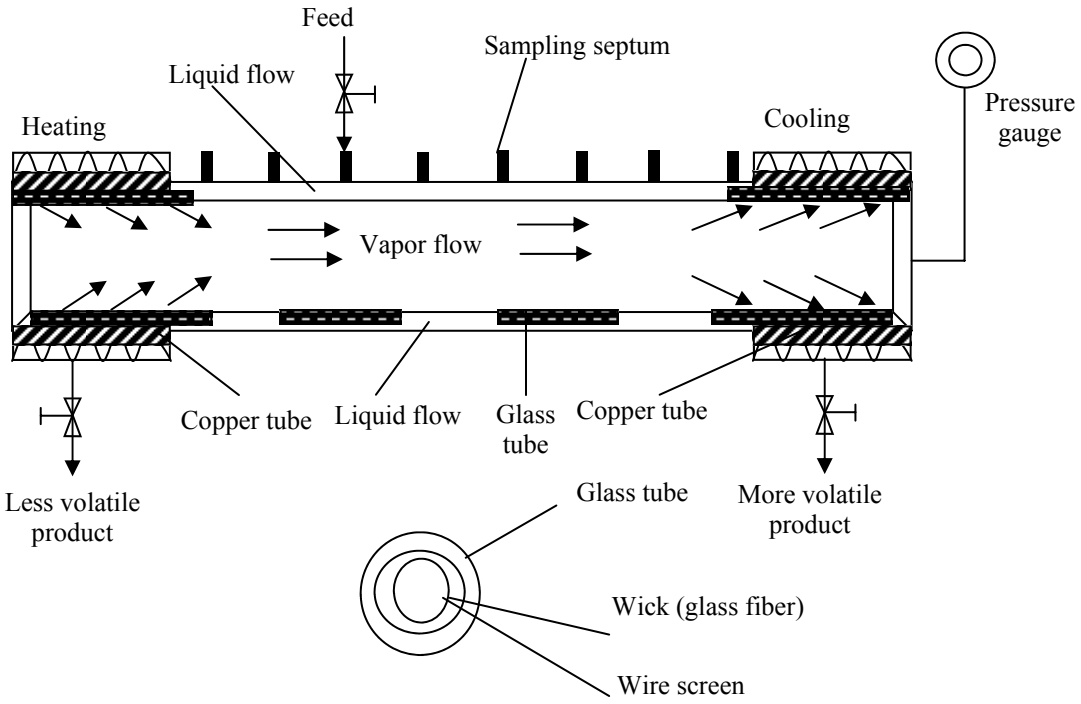


**Fig. 6**. Schematics of a heat-pipe type microdistillation column [9].

stream. After a gas–liquid contact of 10 mm, the liquid was removed from the stream. The temperature of the contact zone was kept at an elevated temperature (+60°C) with an electrical heater. The vaporized liquid and the carrier gas flowed to the condensation section, where condensed liquid was separated from gas. Schematics of the device are shown in Fig. 5. Equimolar mixture of acetonitrile and DMF was used as the feed at feed flow rates of up to 0.15 $cm^3$/min. The separation efficiency was up to 0.7 ideal stages.

Nanoscale vapor–liquid separation is still far from being of practical use, as demonstrated both the laser-assisted [1] and nanopillar-structured separation [5]. Both have unresolved issues concerning removal of one of the products, which means that they are closer to batch distillation than continuous-flow separation.

All microscale distillation techniques employ a similar approach: Vapor is generated in the heating section, optionally with the help of a carrier gas,

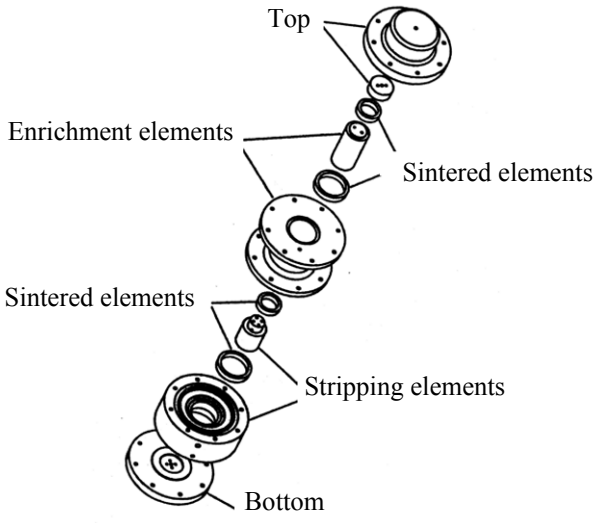


**Fig. 7**. Design of a collimator-type distillation column [12].

followed in succession by the vapor–liquid-contact section to saturate the gas phase and the phase separation section. Also, they can achieve a separation efficiency equivalent to a height equivalent to a theoretical plate (HETP) of 0.1 m or better. However, only two of them achieve one complete ideal stage: the flash separation method by Cypes et al. [2] and the segmented-flow method by Hartman et al. [3]. Both have solved successfully the question of how to create the vapor phase in a steady manner and, what is more important, how to separate the phases. However, both methods [2, 3] require accurate control of differential pressures over the unit, which may cause difficulties when the separation efficiency above one ideal stage is pursued by the daisy-chaining technique. As shown in Table 1, the methods employing gravitation [6, 7] or channel patterning [8] for phase separation suffer from decreasing separation efficiency as a function of capacity.

It is worth noting that the HETP of a concurrent-flow device may not represent the minimum length required for an ideal step but rather the total length of the equilibrium chamber, as is the case with the columns of Cypes et al.. [2] and Hartman et al.. [3]. Also, the capacity describes the feed flow per one channel or a single unit, and it can be increased by operating a number of channels in parallel, aka the numbering up method.

### Countercurrent Microdistillation

Millimeter-scale continuous distillation was carried out by Seok and Hwang [9]. The column consisted of a

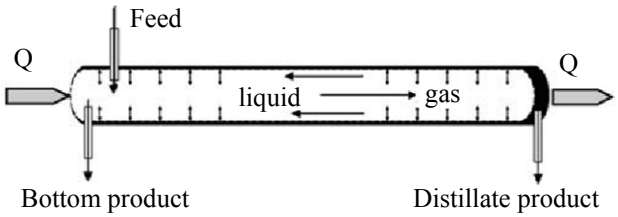


**Fig. 8.** Schematics of planar microdistillation in a horizontal-orientation column [15].

340-mm-long adiabatic section manufactured from glass with an inner diameter of 10.5 mm and two 100-mm-long sections manufactured from copper for heat exchange. One end of the column was electrically heated and the other end was cooled with water circulation. The inside of the tube was lined with a 1.2-mm-thick layer of rolled fiberglass. The purpose of the fiberglass wick was to facilitate liquid flow back to reboiler by capillary forces instead of gravitational forces and to increase the mass transfer and heat transfer area. The wick structure enabled the column to be operated in a horizontal position. Schematics of the column are shown in Fig. 6. Direction of vapor flow was countercurrent to liquid. The device was tested using water–methanol and water-ethanol mixtures as feed. The feed flow rate was from 0.8 mol/h up to 6 mol/h. Separation at total reflux yielded essentially pure water and 88 wt % of ethanol. With continuous operation, the reflux ratio was controlled to between 2 and 10 by controlling the heater coil voltage. The separation efficiency in terms of a height of a transfer unit was between 6 and 16 cm. Ramirez-Gonzales [10] explained the improvement of mass transfer by film mixing caused by the porous media. Tschernjaew et al. [11] modeled the separation in the microdistillation

**Table 1**. Summary of concurrent-flow separation methods[a]

| N/HETP, m | Capacity,[b] mol $s^{-1}$ | τ, s | Type | Reference |
|---|---|---|---|---|
| NA | $4.2\times10^{-11}$ | NA | Batch | [1] |
| 1/0.05– 1 | $6.0\times10^{-5}$ | 9 | Flash | [2] |
| 1/0.88 | $4.5\times10^{-6}$ | NA | Flash | [3] |
| 0.3/NA | $1.5\times10^{-6}$ | NA | Batch | [5] |
| 0.65–0.8/0.108–0.088 | $1.0–5.1\times10^{-5}$ | 11–53 | Flash | [6, 7] |
| 0.7/0.057 | $3.8\times10^{-5}$ | 0.06 | Flash | [8] |

[a] (N) Number of ideal stages, (HETP) height to a theoretical plate, and (τ) residence time. [b] Capacity is the feed flow rate.

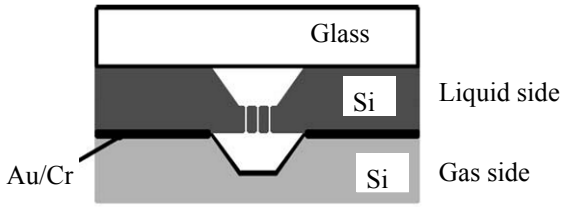


**Fig. 9.** Cross-section of a micro-fabricated stripping column [17].

column for multicomponent system, using the Stefan–Maxwell approach with good accuracy.

Fink and Hampe [12] presented a simulation and design study of a distillation column as part of a microplant design study. The distillation column consisted of sintered elements for distributing liquid on the surfaces of an annulus. The inner and outer diameters of the annulus were 16 and 19 mm, respectively. The total height of the device was 40 mm and liquid holdup was 3 $cm^3$. The design of the column is shown in Fig. 7. The column was heated via steel plate at the bottom of the unit. The column was tested at a feed flow rate of 20 $cm^3/h$. The separation efficiency of 2.5 ideal stages was observed. However, concentrations of the product flows were reported to be highly sensitive to changes in the column temperature [13].

TeGrotenhuis and Stenkamp [14] patented a method for separating fluid components, using distillation inside a planar channel. The channel would comprise of a wick for the liquid flow and an open area for gas flow. The purpose of the wick would be to facilitate liquid flow towards the hot end by capillary force. In addition the thickness of the liquid film could be decreased compared to that of a falling film thus reducing the mass transfer resistance. The vapor would be created at least partly within one end of the channel and condensed at least partly in the other end of the channel, although additional external devices could also be used. The results of a 100-h continuous distillation run were first presented by Zheng et al. [15] and later published by Huang et al. [16]. Schematics of a single channel of the microdistillation column are shown in Fig. 8. In a device with 15 parallel microchannels and a feed flow rate of 2.5 g/min of JP-8 fuel using a reflux ratio of 4.3 the estimated HETP was 18 mm. The operation of the device was in a horizontal orientation.

Cypes et al. [17] demonstrated operation of a micro-fabricated stripping column using nitrogen as

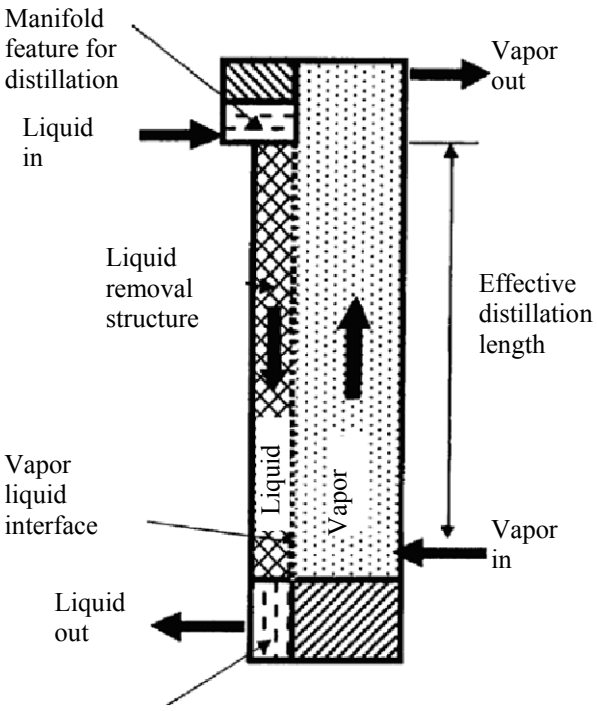


**Fig. 10.** Microscale distillation channel by Tonkovich et al. [18].

stripping gas. The column was fabricated from silicon wafer with etching. The silicon plates were joined with Au–Si eutectic bonding. The gas and liquid channels were identical, 33.5 mm long with a cross-sectional area of 0.92×0.33 $mm^2$ at the top and 0.45×0.33 $mm^2$ at the bottom. The upper channel was perforated on the bottom to create a contacting area between the phases. Design of the unit is shown in Fig. 9. Feed was divided between four channels. Gas flow was countercurrent to liquid. The device was tested with a toluene–water mixture. Separation efficiency compared to that of a traditional packed column indicated 10 times higher mass transfer coefficients for the micro-fabricated column.

Tonkovich et al. [18] described a distillation device with multiple separation sections. The device consisted of a wick structure to minimize the thickness of the liquid film and liquid capture structure to prevent entrainment. The vapor and liquid flow was in countercurrent direction. A variation of the column included a laminated structure with heat exchange channels between the distillation channels. Schematics of a single channel are shown in Fig. 10. Fanelli et al. [19] modeled the operation of the unit with a mixture of hexane and cyclohexane. Liquid flowed down as a falling film stabilized inside a 178 μm thick steel

mesh. Vapor flowed upwards in a 1.3 mm thick open channel. With a feed flow rate of 1 $cm^3$/min of a hexane–cyclohexane mixture, the HETP was 21 mm. Fanelli et al. [20] published experimental results for ethane–ethylene separation at 18 bar pressure. With a flooding factor of 2 the number of ideal stages was 2 within 114 mm long contact zone of the micro-distillation unit. This equals a HETP of 57 mm. As a comparison, traditional C2-splitters have typically a HETP value of approximately 500 mm.

Sundberg et al. [21–24] presented distillation columns consisting of thick metal walls and a planar chamber using metal foam as packing. The first design employed brass as the column wall material to achieve a high heat transfer rate from the heater towards the cold end. The hot end was heated with electrical heater and the cold end was air cooled. The chamber was 140-mm-long and 3.5-mm-high with 2-mm-thick metal foam on the bottom. The separation efficiency with total reflux using a mixture of hexane and cyclohexane was 11 ideal stages, which equals a HETP of 13 mm. On a later design the length was increased to 290 mm with the chamber height of 5 mm and packing thickness of 3 mm. Temperature control was via a heated aluminum plate attached to above and below of the column. The separation efficiency at total reflux was 11 ideal stages, which equals a HETP of 25 mm. After eliminating the empty space in the chamber with a metal plate, the separation efficiency was 18±2 ideal stages, which equals a HETP of 16 mm**.** Schematics of the column are shown in Fig. 11**.** With a feed flow rate of 1 mol/h the reflux ratio was equal to 2.2 and the separation efficiency was equal to a HETP of 80 mm**.** Also, design with a cross-sectional area of 5×5 $mm^2$ was manufactured and tested [25, 26].

Ziogas et al. [27] developed a plate column. The column consisted of an insert with several plates that could be removed and changed. The unit was operated in a vertical orientation. Schematics of the device are shown in Fig. 12. The separation efficiency was 12 ideal steps for a close-boiling mixture of iso-octane and n-octane. The separation efficiency was equal to a HETP of 8 mm.

A novel device using a spinning spiral micro-channel to produce multistage was published by Mac-Innes et al. [28]. The microchannel was etched in the shape of a spiral on a glass disc then thermally bonded onto a second one. The channel depth was 95 μm, width 250 μm and length of contacting channel 35.3 mm.

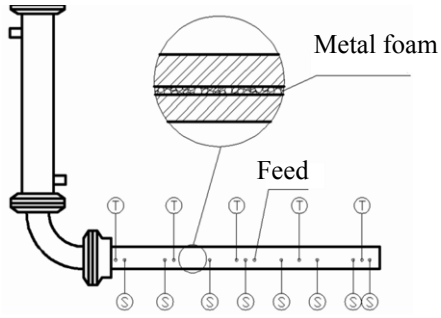


**Fig. 11.** Schematics of a metal-foam filled microdistillation column [23].

Feed was introduced from a reservoir to the middle of the disc via tubing with inside diameter of 150 μm. The disc was rotated during operation at a speed of 5000 rpm. The products were withdrawn from the edge of the disc into two reservoirs via 50 μm diameter tubes. Schematics of the device are shown in Fig. 13. The device was tested with an equimolar feed mixture consisting of 2-methylbut-2-ene and 2,2-dimethyl-butane. The device was operated as a stripping column with the distillate-to-bottoms-ratio varying from 1.4 to 7.7 and the feed flow rate of 0.17 $cm^3$/s. The HETP of the contacting channel was 5.3 mm. MacInnes noted that to use the column with continuous feed the

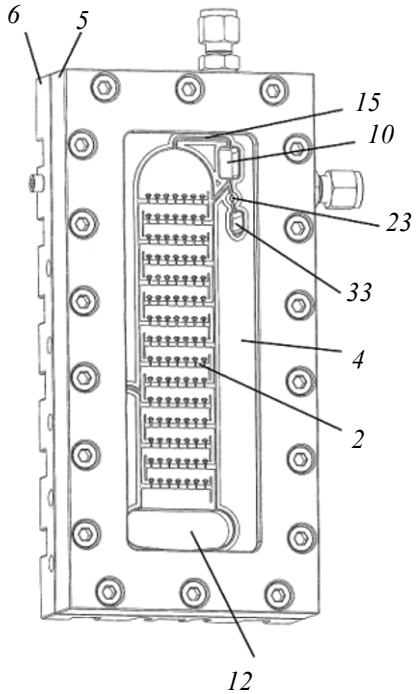


**Fig. 12.** Microrectification column by Ziogas et al. [27].

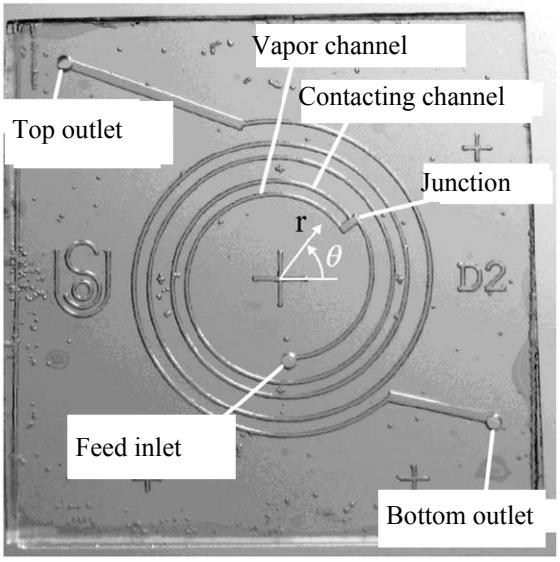


**Fig. 13.** Spinning spiral microdistillation column [28].

development of slip rings suitable for transporting fluids into the rotating column is required.

The countercurrent designs presented are summarized in Table 2. As some of the designs are mainly published as patents, the information is scarcely available. Also, as the columns can be operated in varying internal mass flux or flooding factor ranges, HETPs are slightly difficult to compare.

Of the countercurrent microdistillation columns presented, the designs of Seok and Hwang [8], Fink and Hampe [11], and MacInnes et al. [28] will be most difficult to adopt into commercial use. The round shape [8] can be difficult to scale without affecting the diffusion-based mass transfer rate. The collimator-type column [11] had instability problems related to temperature control of the column. A spinning spiral column with more than a total of one fluid inlet or outlet will be difficult to link to external tubing [28].

**Table 2.** Summary of countercurrent-flow separation methods

| N/HETP, m | Capacity, mol $s^{-1}$ | τ, s | Type | Reference |
|---|---|---|---|---|
| NA/0.06–0.16 | 0.013–0.1 | NA | Heat pipe | [8] |
| 2.5/0.016 | NA | 540 | Collimator | [11] |
| NA/0.018 | $1.5\times10^{-4}$ | NA | Planar | [14] |
| NA | $0.7–32\times10^{-4}$ | 1–25 | Stripper | [17] |
| NA/0.021 | NA | NA | Falling film | [18] |
| 18/0.016, 5/0.058 | total reflux, $6.3\times10^{-5}$ | 180 | Plate | [21–26] |
| 12/0.008 | NA | NA | Plate | [27] |
| 6.7/0.053 | $2.7\times10^{-5}$ | 0.13 | Stripper | [28] |

The design by Sundberg et al. [21–26] is mainly intended for research purposes, since its capacity may not be sufficient for industrial-scale use. The stripper by Cypes et al. [17] is promising and may have commercial applications. The devices by TeGrotenhuis and Stenkamp [14] and Tonkovich et al. [18] are suitable for industrial use, but, to the authors' knowledge, they have not yet been commercialized.

## CONCLUSIONS

Microdistillation columns offer efficient separation with short residence times. The problems encountered with the devices range from temperature instability induced concentration fluctuations to required differential pressure control over the capillaries. Changing of the temperature profile of the device is often used to control the internal reflux ratio and the product flow rate. Temperature profile control can be inaccurate for the purpose of adjusting the device. An easier reflux ratio control should be developed. A working reflux ratio control is crucial for accurate adjustment of product concentrations and for enlargement of the operating window.

Manufacturing methods and materials used in the single-stage concurrent contacting devices are similar to microreactors. The structural material is commonly glass or silicon and channels are made by various etching techniques. The countercurrent distillation devices are manufactured either by mechanical methods or by etching; the wall material is mainly steel.

The operating principle of all the single-stage concurrent devices is of the same type. Vapor phase is created with heating or with the help of a carrier gas inside a long meandering channel. After the contact channel the phases are separated and vapor is condensed. What makes some methods superior to others depends on how well they succeed in phase separation. The successful designs use either a capillary structure or a membrane for the task.

For the countercurrent, continuous fractionating columns the most working designs all use porous materials to enhance the liquid flow. The gravitation force is too weak inside microchannels, and, therefore, structures employing capillary forces are preferred.

Also, in a similar manner to concurrent flow devices, phase separation plays traditionally equally important part in the countercurrent devices, especially in designs utilizing a multitude of distillation sections.

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