A Comparative Study for Separation of Binary-mixture in Batch and Microchannel based Distillation Module

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Abstract

The distillation process is a very useful mass transfer operation in different industries like food, chemical, and petrochemical. This research work is focused on the experimental comparison between the performance of the batch distillation module to the microchannels based distillation module based on separation and product purity. A stainless-steel, Y-junction microchannel, of hydraulic diameter 0.85 mm having dimension 90mm X 1mm X 0.75mm (LX W X D) has been fabricated through mechanical engraving technique. A binary mixture of 50% w/w of acetic acid + n-hexane was used as a test sample in the batch and micro-distillation module. The effect of resistance time, system temperature on distillation efficiency, and mole fraction in both the systems have been compared. The maximum separation and purity have been observed in the microchannel distillation module for the same residence time. Experimentally, the mole fraction of n-hexane in distillate product has been observed as 0.9652 for distillate and 0.18 for bottoms in microchannels-based distillation while in batch distillation it was observed in 0.81 to 0.36 of n-hexane mole fraction in distillate and bottom product, respectively.

Keywords: - *Microchannel; Separation; Y-junction; Distillation; Residence time*

INTRODUCTION

Distillation is an important unit operation for the chemical, petrochemical, beverages, and pharmaceutical industries. It is used for the separation of multicomponent, single components and also used for obtained high purity products. Nowadays many process industries such as fermentation, petrochemical, and food industries using different separation equipment such as packed towers, batch distillation, Sieve tray column, etc, [1], this convention equipment having several disadvantages such as the provides less surface to volume ratio less[2] heat and mass transfer and consumed higher energy, hence a process intensification is required to develop a new technology to overcome the disadvantages of conventional equipment,[3] In recent years, microfluidic devices have been used in a variety of applications including molecular biology, small-molecule organic synthesis, immunoassays and

cell manipulations [4] and separation [5-7] From the literature is was observed that many reports have used the solvent extraction [8,9] method for separating the valuable product and no study was found on the recovery of the solvent and solute after the solvent extraction process distillation was used for the separation.

Author Sundberg et al.,[10] in their review, mentioned the works of Sotowa and Kusakabe [11] where they used a silicon-glass microchannel to separate aqueous methanol (90% methanol) solution and achieved an efficiency equivalent to 0.65 ideal stages. Wooton and deMello [12] separated the acetonitrile + DMF binary mixture by using helium as the carrier gas in a glass microchannel. Separation efficiency equivalent to 70% of an ideal stage was reported. Similarly, Lam et.al., 2011 [6] investigated the distillation of 50 mol% acetone-ethanol mixture into 81 mol% acetone in the distillate and 13% in the bottom at 0.15 mL/h feed flow rate.

It is seen in the literature that no report that most of the reports on distillation in microchannels have not compared the results with a batch distillation, hence this works focuses on conducting the distillation in batch &microchannels and, estimating the separation of acetic acid. effect of flow rate on the extraction efficiency as well as the volumetric mass transfer coefficient was estimated.

OBJECTIVES AND SCOPE OF THE STUDY

The objective of this study is to demonstrate and the compression of the separation process distillation in the batch system and microchannel based distillation columns. it also includes the comparison based on the performance and the power requirement for both of the systems. for this type of comparison, a stainless steel microchannel device was designed and fabricated and a simple distillation setup is used.

MATERIAL AND METHODS

Materials

Chemicals used in this experimental work are analytic grade. All the chemicals used in this study had ≥ 0.97 purity (based on mass fraction). aceticacid 0.99, and n-hexane 0.97. All the chemicals were used as it is without further purification. Their physical properties are given below in the table.

Table 1 list of the chemical used in experimental studies

Chemical used	Purity in mass fraction	Density (g/cm^3)	Refractive index	Viscosity (cP)
n-Hexane	>0.97	0.672	1.3749	0.297
Acetic Acid	>0.99	1.043	1.3716	1.1393

Fabrication of experimental setup

A rectangular microchannelwas fabricated on a stainless steel plate, having hydraulic diameter D_H = 0.83 mm with a junction angle of 45°. Initially, A channel was designed by using coral draw suit software and this design was fabricated on the stainless-steel plate by using a pantograph engraving machine, and the engraving was done with the help of a carbide cutting tool.

Before the engraving process, some basic operations for surface finishing were also performed such as buffing, smoothing, and polishing. Engraving has been performed on the plate only and another same size plate was fixed with the help of bolting.

Discerption of the experimental setup

The schematic diagram of a microchannel based distillation module was shown in Figure 1. The distillation module consists of a nitrogen gas cylinder, Temperature control hot water bath for heating the gas, a Microchannels distillation module, Condenser bottle, bottom product collection flask, PID temperature controller, Surface heaters, Pressure sensors, peristaltic pump, power meter, and two input and output streams.

An input of the microchannels was connected to the nitrogen gas and the second input was connected to the pump through connecting tubes. A temperature-controlled water bath was used for heating the carrier gas. A gas mass flow controller was used for maintaining a constant gas flow rate throughout the experiments. And the gas flow rate was controlled with the accuracy of +/- 0.01ml/min and calibrate with a bubble flow meter.

Output streams of the micro-distillation module were connected to the rubber crock fitted glass bottle with the leak-proof arrangement. The temperature of the top and bottom product was measured using a thermocouple. The distillate and raffinate streams were connected with reagent bottles as product collection units. The micro distillation module was heated using ceramic and steel surface heaters (Anupam heater Pvt Ltd. Thane,Maharashtra,India) of dimensions (25mm*55mm*8mm) having power rating 0.25kWh and 0.4kWh. A 9- channel temperature indicator was used for observing the zone temperature of the micro-distillation module at a different location from the heating source with an accuracy of 0.1^0C.

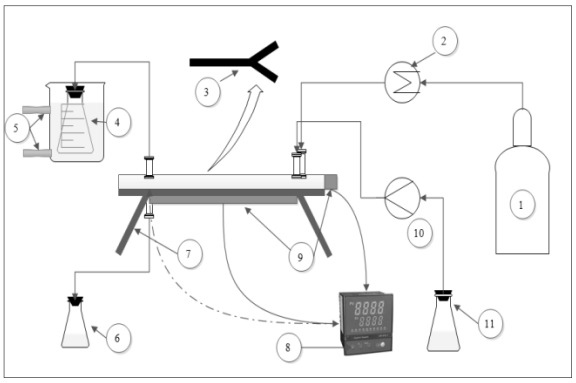

Figure 1(a). Schematic diagram of the experimental setup, 1- Nitrogen gas cylinder, 2- Water bath for heating the gas, 3- Microchannel, 4- Distillate product, 5- Chilled water I/O, 6- Bottoms Product, 7- Stand,8- PID controller, 9- Surface heaters, 10- Parenthetic pump, 11- Feed sample

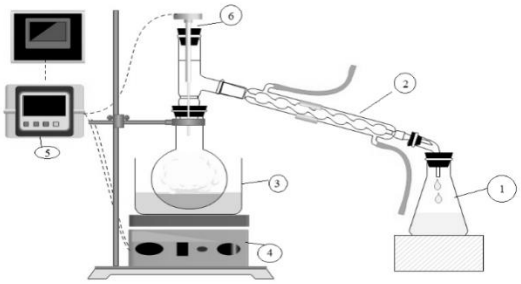

Figure 1(b). Schematic diagram of experimental setup batch system, 1- collection flask, 2- condenser, 3- Oil bath, 4- Magnetic starrier, 5- PID controller, and temperature indicator, 6- Thermocouple.

Experimental procedure

To investigate the performance of the batch and microchannel distillation process. A mixture of two components was prepared on a weight basis. A test sample of the 50 % w/w solution of acetic acid and n-hexane was prepared. A peristaltic pump was used for feeding the feel mixture into the microchannels distillation module with a flow rate of 0.02 ml/min to 0.2 ml/min. with an accuracy of 0.01 ml/min. the gas flow rate was maintained 7 ml/min with 90 ℃, which was obtained from the trial and error method. The temperature of the distillation module was kept 70

℃. and the chiller water temperature was kept constant at 7 ℃.it was also investigated through the trial and error method.

RESULTS AND DISCUSSION

A comparative study of the separation of the binary mixture was conducted in a batch distillation & micro distillation. The batch system has a 2-neck round bottom flask attached with a condenser and collecting flask the temperature was measured with the help of a thermometer and maintained through a magnetic starrier. The microchannels distillation module comprised of a microchannel, pump, hot water bath for gas heating, surface heater, nitrogen gas cylinder, PID thermocouple sample collection bottles (Distillate and bottom products), and chiller. Micro-distillation experiments were conducted precisely under a controlled temperature profile and feed flow rates. For the study, the operation parameters such as temperature, feed flow rate, and feed concentration were maintained from 70 ℃, 0.02 to 0.2ml/& 50 % w/w, respectively. Mole fraction (purity) of distillate & bottom products was estimated from the output concentration of the n-hexane and acetic acid in distillate and bottoms. The power required for separation was also estimated. The results are described below.

Experiment procedure for batch distillation-

For the batch distillation,the same feed composition was used and about 150 ml of feed sample was loaded into the round bottom flask which is placed in the oil batch, and the magnetic starrier is used for heating and mixing the feed. The PID controller is used for maintaining the temperature of the oil bath with the accuracy of +/- 0.5 ℃. Feed mixture heated at the desired temperature then after some time. The vaporis generated, when thisvapor passes through the condenser, they get condense and collected in a collection flask.

Effect of flow rate on percentage separation in microchannels

To check the effect of flow rate on separation the feed flow rate was maintained 0.02 ml/min to 0.2 ml/min, and the temperature of the separation point temperature was maintained at 70 ℃. thechiller temperature was maintained at 7 ℃, and the gas flow rate was maintained at 10 ml/min. with a temperature of 70 ℃. Figure 2(A) shows that as the flow rate of the feed mixture increases the % separation of the solvent decreases. (10) It was due to the less residence time causes less heat and insufficient heat have been transfers to the mixture, resulting in less vapor of the more volatile components that were developed inside the microchannels to carry towards the condenser.

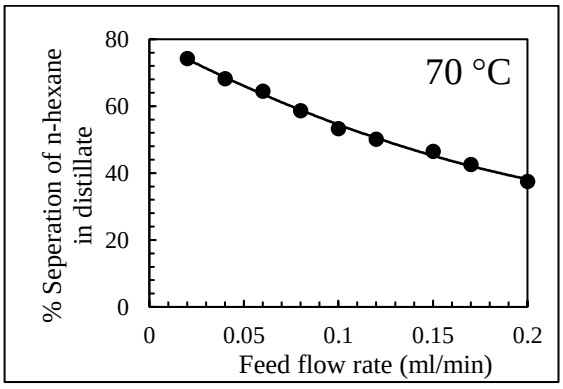

Figure 2 (A) Effect of feed flow rate on % separation of n-Hexane in the distillate, at temperature 70 ℃, feed concentration 50 % w/w, Condenser temperature 7 ℃, with Carrier gas flow rate 6 ml/min with temperature 90 ℃,

Effect of flow rate on the mole fraction

To investigate the effect of the flow rate on mole fraction (purity) of the solvent and solute, a similar operating condition was maintained for the separation such as the gas flow rate was

maintained at 10 ml/min. with a temperature of 70 °C., the Feed flow rate was maintained at 0.02 ml/min to 0.2 ml/min, and the temperature of the separation point temperature was maintained at 70 °C. thechiller temperature was maintained at 7 ℃, Feed concentration 50 % w/w. Figure 2 (B) shows the effect of the feed flowrate on the mole fraction of the n-Hexane in distillate and PA in bottoms, Maximum purity of the n-hexane in the distillate, and PA in bottoms was obtained at a feed flow rate of 0.02 ml/min. In this condition, the rate of vaporization is equal to the feed flow rate causes a higher degree of heat transfer to the mixture. (6), n-hexane mole fraction was observed 0.9562 in distillate and PA was observed 0.8144 in bottoms.

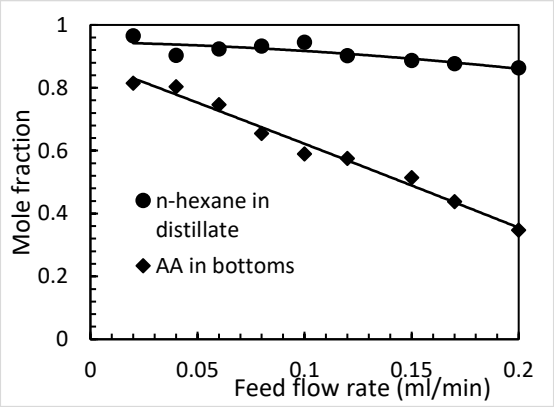

Figure 2(B) Effect of feed flow rate on the mole fraction of n-hexane in distillate & PA in bottoms at temperature 70 °C, feed concentration 50 % w/w, Condenser temperature 7 °C, with Carrier gas flow rate 7 ml/min with temperature 90 °C,

Comparison with a batch system

For the comparison study of distillation, a batch distillation was performed in a simple distillation experimental setup as shown in Figure 4.1, The batch system contains an oil bath, Magnetic starrier, Round bottom flask, Condenser, chiller water I/O, collection pot, and power meter was used for the estimation pf power required for separation. For this 100 ml of the feed, the mixture was filled in the round bottom, condenser magnetic starrier collection pot was arranged accordingly. The thermometer was used for measuring the temperature, the desired temperature was maintained and the samples were taken at a constant interval to check the effect of the residence time on % separation and mole fraction and power required for separation.

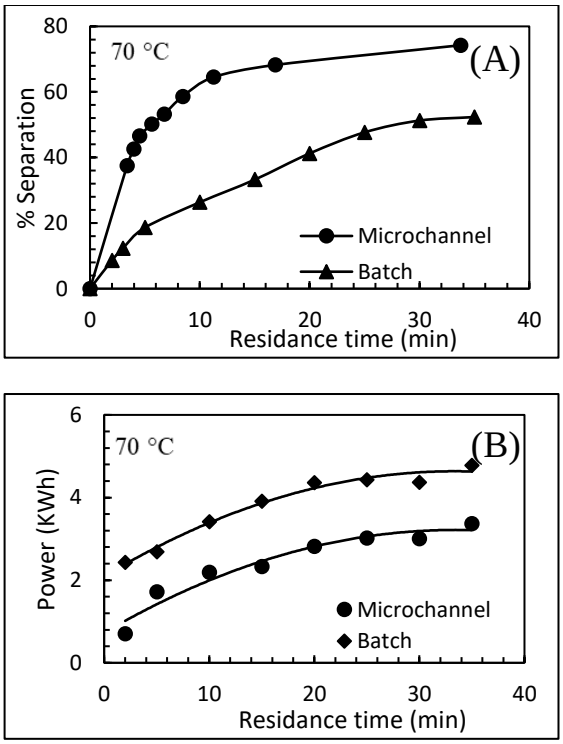

Figure. 3 Comparison of distillation in batch and microchannels module, (A) Effect of residence time on % separation, (B) Effect of Residence time on power requirement for separation of one mole of solvent to the distillate.

CONCLUSION

Separation of acetic acid from n-hexane was conducted in a Y-junction microchannel of DH = 0.85 mm. The effect of flow rate and residence time and temperature were studied to evaluate the percentage separation, mole fraction, and power requirement. The percentage separation of the solvent and solute are found to decrease with an increase in flow rate. The lowest separation was

found 37.5% while the highest was achieved 74.2 % for microchannel at flow rate 0.2 ml/min and 0.02 ml/min respectively, it was observed that at a lower temperature less purity of the solvent has been observed low in both of the systems as the temperature increases the purity increases as start decreasing further increment in temperature. A comparison of microchannel extraction and batch extraction was made. It was found that the microchannel gave the highest % separation about 74.25% than the batch distillation 52.3%. and also consumed less power for separation of per mole of the solvent in the distillate. Batch distillation consumed 4.78 kWh (17208KJ/mol) power while the microchannel distillation module consumed 3.37 kWh (12132 KJ/mol) power for separation of per mole of solvent. Thus, microchannels appear to be a better choice for the separation of a binary mixture in the distillation process.

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