

Liquid-Liquid Extractive De-aromatization of Toluene from n-hexane by Using Three Deep Eutectic Solvents (DES) in Two Different T- junction Geometries

Vikas Kumar Choudhary^{*,**}, Bushra Khatoon^{*}, Sadhana Sachan^{*} and M Siraj Alam^{**†}

^{*}Department of Chemical Engineering, MNNIT Allahabad, Prayagraj-211004, India

^{**}Department of Chemical Engineering, Chandigarh University, Mohali, Punjab140313, India

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Abstract – In the present study, the extractive de-aromatization of hydrocarbons (i.e., toluene in n-hexane) was investigated using different deep eutectic solvents (DESs) in a multidimensional T-junction microchannel with diameters of 0.5 mm and 1 mm, respectively. Three different DESs were synthesized using ethylene glycol, triethylene glycol, and propionic acid as hydrogen bond donors (HBD) in combination with choline chloride, methyl triphenyl phosphonium bromide (MTPB), and tetrabutylammonium bromide (TBAB) as hydrogen bond acceptors (HBA). The effects of flow rate and channel diameter variation on slug volume were studied. The percentage removal of aromatics, slug volume, specific interfacial area, and volumetric mass transfer coefficient were investigated for the three different synthesized DESs. For the slug flow, the maximum slug length was in the range of 0.96-1.53 mm in the 1 mm T-junction and 0.48-1.02 mm in the 0.5 mm T-junction microchannel, obtained for a corresponding flow rate of 3.0-12.0 mL/min. The maximum interfacial area was in the range of 7315-7898 m²/m³ for the 0.5 mm T-junction and 2714-3053 m²/m³ for the 1 mm T-junction channel. The extraction efficiencies in slug flow ranged from 30.82% to 74.32%. Similarly, the volumetric mass transfer coefficients ($k_{l,a}$) were estimated to be in the range of 0.018 to 0.113 s⁻¹. Comparing the various results obtained during the experimentation, it was found that better performance in both T-junction microchannels was achieved by the MTPB and TEG synthesized DES with a molar ratio of 1:6.

Key words: Microchannel, Dearomatization, Deep eutectic solvent, Slug flow, Extraction efficiency

1. Introduction

In order to adhere to requirements for sulfate-free fuel, aromatic hydrocarbons in ppm and ppb are extracted from solutions through refinement. Aromatic hydrocarbons are more versatile and stable, and they can be used in a variety of ways [1]. The petrochemical industry places a high priority on the separation of aromatic as well as cyclic aliphatic hydrocarbon, particularly the benzene or cyclohexane mixture, which is also one of the most challenging processes [2]. One of the most popular methods, suitable for materials with an aromatic content of 20 to 65%, is liquid extraction [3]. It is used to separate aromatics from petroleum mixes like naphtha reformat and lubricating oils in contactors like mixer-settler, sieve plate columns, etc.

Using standard organic solvents such as sulfolane, N-methylpyrrolidone, N-formylmorpholine, or glycols along with liquid extraction, naphtha's aromatic hydrocarbons have been most frequently removed. So, discovering substitute solvents is essential because these substances are volatile, toxic and combustible even though they have good yields. These new solvents ought to enable an efficient separation and

harm the environment less, in accordance with the principles of green chemistry [4]. One of the most promising remedies to resolve these drawbacks of such solvents has been suggested in the literature over the past few decades [5] substituting ionic liquids for organic solvents. Recently, the Environmental Protection Agency [EPA] lowered the amount of benzene in petrol to an average volume of 0.62% (with a maximum of 1.3%) [6].

From a long time, ionic liquids [ILs], which are essentially organic ionic molecules with low melting points that enable them to persist in a liquid state at decreased temperatures, had appeared to offer a convincing solution. These ILs have desirable properties such as low volatility, strong chemical and thermal stability, ionic conductivity, and a powerful ability to dissolve a wide range of substances [7]. Additionally, by choosing the proper cations or anions, this dissolution capability might be precisely altered, enhancing their allure [8,9]. In the industrial sector, the recovery of aromatic chemicals is typically performed by a liquid-liquid extraction process. These alkanes make up a sizable portion of the extracted aromatic compounds during the aromatic's extraction process. The results of the simulation may be inaccurate if important Liquid-Liquid Extraction [LLE] data for these alkanes are not included. It's also important to recognise that different aromatic configurations can have a variety of effects on how effectively aromatics are extracted [10,11].

The increased popularity of microfluidic systems in recent times can be attributed to its multifunctional features, which include

[†]To whom correspondence should be addressed.

E-mail: msalam@mnnit.ac.in

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enhanced process intensification [12]. Microchannels have a higher interfacial area, which is a common technique for increasing mass-transfer efficiency [13] and have applications in a variety of fields, including aeronautics, transportation, refrigeration, superconductors, microelectronics, and others [14-17]. The fast intrinsic kinetics, high heat, and mass transfer of such microchannels make them preferable to traditional reactors in terms of performance. Nowadays, microdomains are used in a larger range of fields, including biomedicine and extraction. They are also successfully used in sectors such as pharmaceuticals, petrochemicals, and biofuels.

In microdevices, liquid-liquid two-phase flow is extensively utilized for extraction [18,19], emulsion/demulsification [20,21], liquid-liquid reaction [22,23], and synthesis of nanomaterials [24]. A great deal of interest in microdevices of liquid-liquid two-phase flow is attributed to the high surface-to-volume ratio, which may greatly enhance the heat and mass transfer process [25]. Additionally, a microdevice can save reagent costs and increase the safety of dangerous substances and intense exothermic reactions. Therefore, it is believed that liquid-liquid two-phase flow in microdevices is a potential concept that has recently garnered a lot of interest.

Liquid-liquid two-phase flow study is based on flow pattern because these factors significantly affect mass transfer [26,27], pressure drop [28,29] and equipment corrosion [30]. With liquid-liquid two-phase flow, interfacial area and surface renewal rate can vary depending on the flow patterns, which are connected to mass transfer. According to Matsuoka, A. and Mae, K. [31] due to convective mass transfer by recirculating the flow throughout each phase, the volumetric mass transfer coefficient in the slug flow was higher than that in the annular flow, although the annular flow's specific interfacial area was bigger. When looked into the pressure decrease of a two-phase, helical micro-shaped liquid-liquid flow, Verma, R. K. and Ghosh, S. [32] discovered that the continuous phase's higher viscosity was what caused the higher pressure drop. In studying liquid-liquid two-phase flow, the first goal is to determine the microdevice's flow pattern.

Slug flow, droplet flow, parallel flow, irregular flow, and combinations thereof are the most prevalent flow patterns in microdevices [18,33]. The flow pattern is heavily influenced by the flow circumstances, including flow rate, velocity, and phase ratio. Slug flow patterns were seen in a Y-junction microchannel, according to Darekar et al. [34], while parallel flow patterns were seen at higher flow rates of both the aqueous and organic phases. Furthermore, the flow pattern is correlated with the physical properties of two phases, including density, viscosity, and interfacial tension. When Zhao et al. [35] looked into the effect of the microdevice's wettability on flow characteristics; it was only observed parallel flow in the microchannel following surface alteration. The implications of microdevice structures (straight up or helical form, either circular or rectangular cross section, inlet of Y-junction or T-junction) on flow patterns were the subject of numerous researches [36-38]. There are a number of innovative studies on the double emulsion droplet hydrodynamics under shear, and studies like these help us understand how droplet flow is deformed and broken up [39,40].

A class of green solvents known as deep eutectic solvents is synthesized when a hydrogen bond donor and hydrogen bond acceptor are mixed together in a fixed proportion, which results in a significant drop in their melting point. They are quite good substitutes for conventional organic solvents because of their low toxicity, biodegradability, and adjustable qualities. Since DESs may efficiently solubilize target solutes while reducing their negative effects on the environment, they hold promise for the extraction of a variety of aromatic chemicals [45]. DESs, for example, have been effectively used to extract Polycyclic Aromatic Hydrocarbons (PAHs) from complex mixes, such as food and fuel samples. The distinct solvation mechanisms of DESs that improve interaction with aromatic molecules are responsible for recent research' remarkable extraction efficiency, which frequently surpass those of traditional solvents [44].

Recent studies showed that the efficiency of several DES formulations in liquid liquid extractions of aromatics in microchannels has been investigated. For instance, using a ternary system of propylene carbonate, toluene, and n-heptane in microchannels showed extraction efficiencies that were nearly identical to equilibrium separations [43]. So, the latest research goals have been studies pertaining to performance parameters for mass transfer, uniform flow distribution, and flow distributor design in Parallel and Multiple Microchannels (PMCs) [41]. Furthermore, an extensive amount of research has been done on how operational factors like channel diameter and flow velocity affected extraction performance. It was discovered that while larger diameters occasionally resulted in worse extraction efficiency because of reduced mass transfer, increasing the flow velocity generally improved it [43].

2. Material and Methods

2-1. Materials used

T-junction glass microchannels with internal diameters of 1 mm and 0.5 mm were used for the experiments. The capillary lengths ranged from 30 to 50 mm. Two liquids were pumped through the microchannels using two syringe pumps. In this experiment, the chemicals used were n-hexane, toluene supplied by Loba Chemicals Pvt. Ltd. India along with chemical used to synthesize deep eutectic solvents namely choline chloride, ethylene glycol, methyl triphenyl phosphonium bromide, triethylene glycol, tetra butyl ammonium bromide, propionic acid (supplied by Sisco Research Laboratories (SRL) Pvt. Ltd. India with the purity of $\geq 99\%$). These chemicals were employed without additional purification. The experiments were conducted at room temperature.

2-2. Physical properties of chemicals

2-2-1. n-Hexane

The chemical compound n-hexane has the formula C_6H_{14} . It has six carbon atoms and is a straight-chain alkane. Owing to its non-polar solvent qualities, this volatile, colourless liquid finds extensive application in a wide range of industrial applications.

Table 1. Physical properties of n-Heptane and Toluene

Chemical	n-hexane	Toluene
Molecular formula	C ₆ H ₁₄	C ₇ H ₈
Molar mass	86.18 g/mol	92.14 g/mol
Appearance	Colorless-liquid	Colourless-liquid
Density	0.660 g/mL	0.87 g/mL at 293 K
Odour	Odourless	Sweet, pungent
Viscosity	0.30 cP at 293K	0.590 cP at 293 K
Melting Point	177.7 K	178K
Boiling Point	341.7 K	384 K

2-2-2. Toluene

Toluene is a significant organic molecule with the chemical formula C₇H₈, and it belongs to the hydrocarbon class because it has hydrogen and carbon atoms in it. It is a colourless, extremely flammable liquid with a pleasant aroma. It is extensively utilized as a solvent and an industrial feedstock.

2-3. Advantages of using DES solvent

Eutectic systems with a very large depression of the melting point are known to be deep eutectic solvents (DES's). Deep eutectic solvents are the new class of solvents that has received much attention in recent years for aromatic separation. Ionic liquids are solvents that have been utilized as a part of the past time. They are environmental friendly compared to the organic solvents used in the operations of liquid-liquid extraction and separation processes. The issue with ionic liquids in large scale applications is their complex reaction mechanism, synthesis processes and expensive raw materials. An alternative had been recognised to ionic liquids, named deep eutectic solvents (DES). The Main advantages of DES's are their cost effectiveness, availability, non-toxicity, recyclable and generally are non-reactive.

2-4. Synthesis of DES solvent

The correct quaternary ammonium salt was combined with various hydrogen bond donors in a screw-capped bottle according to the intended molar ratio of organic salt to hydrogen bond donor to create the deep eutectic solvents. After that, the bottle was shaken at the appropriate temperature and rotational speed until a clear, homogenous shape formed [43]. The following three DESs were selected as the solvent for our work in order to extract aromatic compounds from hydrocarbons:

- DES1: 1 mol choline chloride + 4 mol ethylene glycol.
- DES2: 1 mol Methyl triphenyl phosphonium bromide (MTPB) + 6 mol triethylene glycol
- DES3: 1 mol Tetrabutyl ammonium bromide (TBAB) + 3 mol Propionic acid

Every DES was created by mixing in a molar ratio, and the combination was then mixed for a certain period of time, at an appropriate temperature, and at a specified rotating speed until a homogenous, transparent, colourless liquid was produced. Precautionary

Table 2. Physical properties of the three DES's

DES	DES1	DES2	DES3
Molar Mass	96.38	134.23	102.54
Density	1.19 g/mL at 293 K	1.24 g/mL at 293 K	1.21 g/mL at 293 K
Freezing Point	207 K	223 K	233 K

Table 3. Temperature and rotational speed required for proper mixing of DES's

S. No	DES	Mixing time	Temperature (K)	Rotational speed (rpm)
1	DES1	3 hrs.	333	200
2	DES2	3 hrs.	353	400
3	DES3	2 hrs.	323	300

steps were made to ensure that the manufactured deep eutectic solvents were kept airtight and moisture-tight in sealed laboratory vials.

2-4-1. Physical Properties of DES's

The physical properties of three DES's were given in Table 2. The temperature and rotational speed maintained for the proper mixing of the DES's were tabulated in Table 3.

2-5. Experimental setup and procedure

Fig. 1 showed the experimental set-up, which included a light source, a camera that was connected to a computer, two glass syringes, two syringe pumps, a T-junction micro-channel and PVC tubes for connections. Syringe pumps were used to inject the two liquids into the T-junction microchannel each with an accuracy of $\pm 1\%$. The syringe pumps have a volumetric flow rate that ranges from 1 to

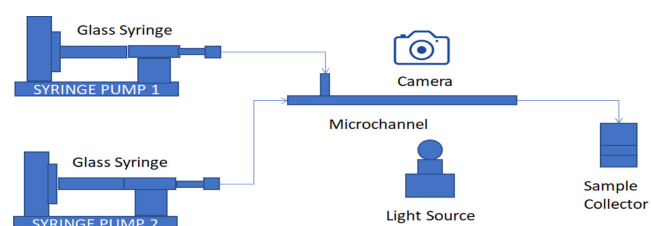
**Fig. 1. Schematic diagram of experimental setup.****Fig. 2. 1 mm T-junction Microchannel.**

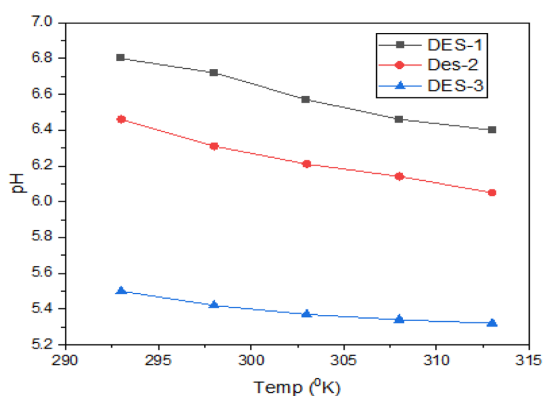


Fig. 3. 0.5 mm T-junction microchannel.

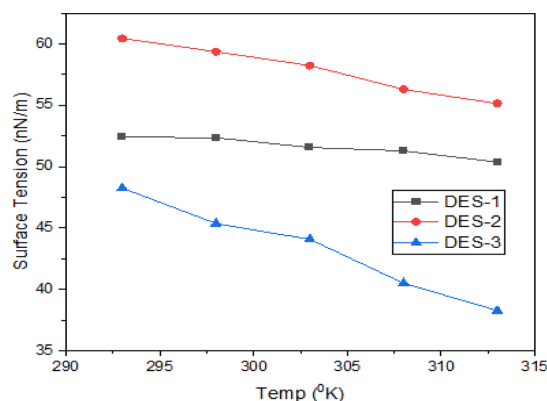
50 mL/min. Two distinct circular microchannel of diameters 1 mm and 0.5 mm and length 50 mm featuring a T-shaped geometry were utilised for the liquid-liquid extraction of aromatic compound experiment. Both the liquids met each other at the junction of the microchannel and observed the formation of slugs. After steady state was established liquids were collected in the separating vessel, where they formed two different layers and they were separated by a funnel and reused. The samples from both raffinate and extract phases from the separating funnel were analysed by refractometer to measure the composition by measuring refractive indexes of raffinate and extract phase. For this measurement ATRVx Refractometer was used which was supplied by Lab India Analytical Instruments Limited.

Table 4. Properties such as surface tension, density and pH of DES-1, DES-2 and DES-3 at varying temperature

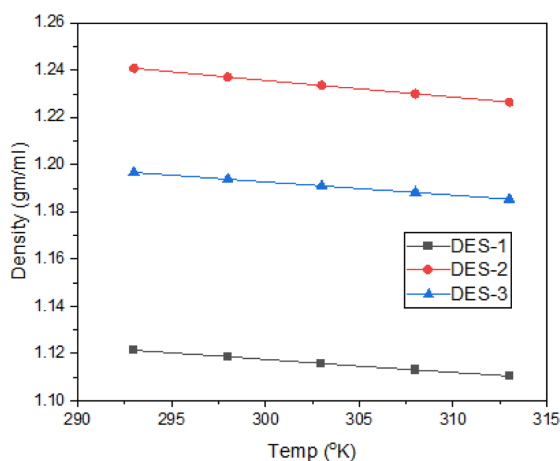
Temperature	DES-1	DES-2	DES-3	DES-1	DES-2	DES-3	DES-1	DES-2	DES-3
K	Surface Tension (mN/m)			Density(g/ml)			pH		
293	52.41	60.45	48.26	1.1967	1.2408	1.1214	6.46	6.8	5.51
298	52.36	59.36	45.36	1.1938	1.2371	1.1187	6.31	6.72	5.42
303	51.57	58.24	44.08	1.1910	1.2335	1.1158	6.21	6.57	5.37
308	51.29	56.30	40.51	1.1882	1.2304	1.1131	6.14	6.46	5.34
313	50.38	55.14	38.27	1.1854	1.2265	1.1105	6.05	6.40	5.32



(a)



(b)



(c)

Fig. 4. Study of properties at varying temperature for DES-1, DES-2, DES-3, (a) pH, (b) Surface Tension, (c) Density.

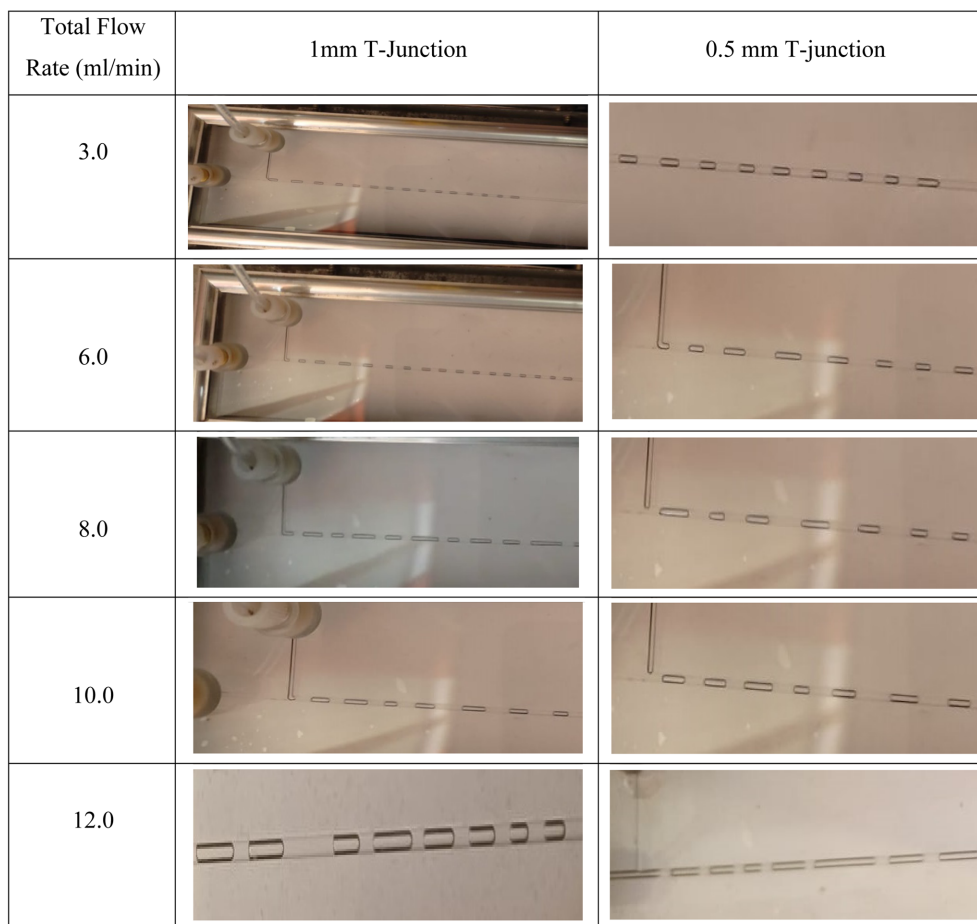


Fig. 5. Variation of slug flow pattern with respect to flow rates.

3. Results and Discussions

3-1. Variation of physical properties with temperature

The physical properties of the DES, such as pH, density, and surface tension, were measured using a pH meter, a hydrometer, and a tensiometer, respectively. Although we conducted our experiment at room temperature, it was necessary to check the dependency of DES solvents on temperature due to weather conditions varying from 293 to 313 degrees Kelvin. This variation could affect the mass transfer performance. However, for our experiment, it was observed that the physical properties did not change significantly within the mentioned temperature range. Table 4 provided the values of the physical properties, and Graph 4 showed the variation with changes in temperature.

3-2. Hydrodynamics study of slug flow

In this study, the slug flow patterns of the two liquids in the T-junction microchannel, initially saturated with DES, were presented in Fig. 5. The mass transfer performance between the two liquid phases in the microchannel was closely related to the hydrodynamic behaviour. Experiments were conducted to observe and generate slug flow patterns by varying the total flow rate from 2 mL/min to 12 mL/min in the T-junction microchannel. In the slug flow pattern, the feed

liquid (n-hexane) flowed as the continuous phase, while the DES solvent flowed as the dispersed phase. Literature indicated that the slug flow pattern, with its larger interfacial area, was required for better mass transfer and extraction efficiency.

3-3. Variation of interfacial surface area with flow rate for different DES

In Figs. 6(a) and (b), the variation of interfacial surface area with the flow rates for 1 mm and 0.5 mm T-junctions was shown for DES-1, DES-2, and DES-3. Table 5 provided the related values. It was observed that with increasing flow rates, the interfacial area also increased. However, this increase was more prominent for the DES-2 solvents in both the 1 mm and 0.5 mm T-junctions. This was observed because maximum mass transfer occurred due to a larger slug flow volume. It was highest for DES-2 due to its high surface tension. As a result, the maximum removal of aromatics occurred for DES-2 at the 0.5 mm T-junction, with an extraction efficiency of 74%. For the other DES solvents, the efficiency remained lower.

3-4. Variation of slug length with different flow rates

The variation of slug length was shown in Figs. 7(a) and (b) for the 1 mm and 0.5 mm T-junctions for DES-1, DES-2, and DES-3. Table 6 provided the related numerical data. It can be observed that slug

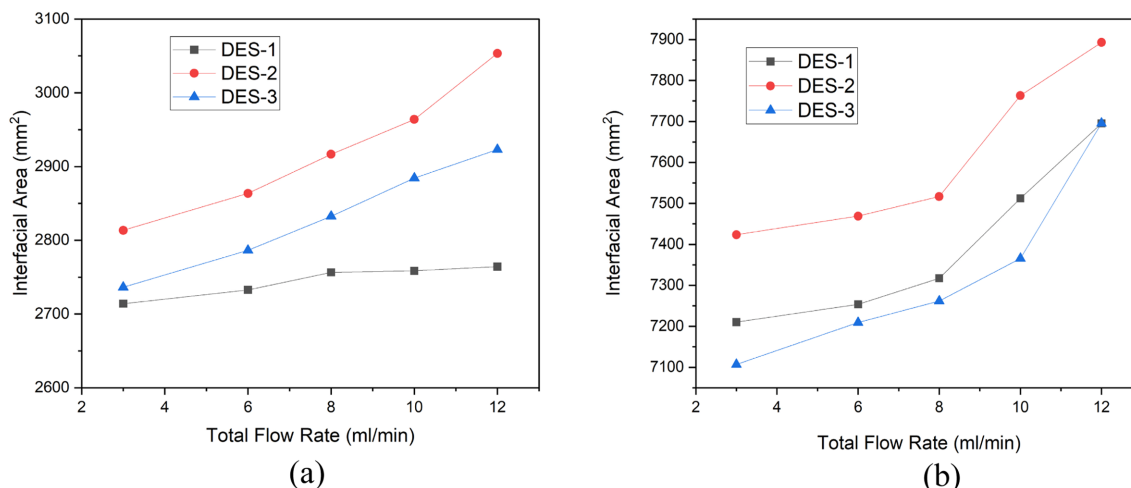


Fig. 6. (a) Effect of interfacial area on the flow rate at 1 mm T-junction, (b) Effect of interfacial area on the flow rate at 0.5 mm T-junction.

Table 6. Variation of slug length for different flow rates for 1 mm T-junction, and 0.5 mm T-junction

Total Flow rate (For 1 mm T-junction)	DES-1	DES-2	DES-3	Total Flow rate (For 0.5 mm T-junction)	DES1	DES2	DES3
3	0.96	1.12	1.03	3	0.48	0.63	0.39
6	1.08	1.21	1.13	6	0.63	0.74	0.58
8	1.2	1.32	1.28	8	0.77	0.88	0.63
10	1.31	1.46	1.36	10	0.89	0.95	0.85
12	1.42	1.53	1.48	12	0.99	1.02	0.96

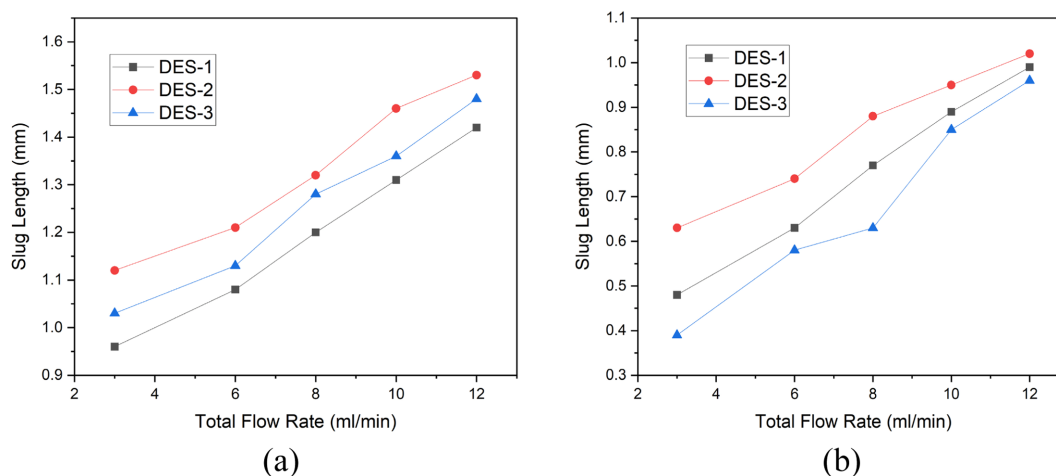


Fig. 7. Flow rate with the variation in slug length: (a) 1 mm T-junction (b) 0.5 mm T-junction.

length increased with increasing flow rates, reaching a maximum for DES-2 at a 12 mL/min flow rate in the 1 mm T-junction. At lower flow rates, the differences in slug lengths became more pronounced than at higher flow rates, likely due to more dominant inertial forces compared to viscous forces. The extraction efficiency for this slug length was approximately 44%, higher than at other flow rates.

3-5. Variation of mass transfer coefficients and extraction efficiency with flow rates

Mass transfer coefficients ($k_{l,a}$) values were determined by the correlation given by Kashid et al. (2011) [37].

$$k_{l,a} = \frac{1}{\tau} \ln \left(\frac{C_{2,sat} - C_{2,in}}{C_{2,sat} - C_{2,out}} \right)$$

Where C_{2in} , C_{2out} and C_{2sat} were the inlet, outlet, and equilibrium concentrations of the solute (i.e., toluene), and τ was the residence time. Fig. 8 provided the mass transfer coefficients in relation to different flow rates for the 1 mm and 0.5 mm T-junctions for DES-1, DES-2, and DES-3. Table 7 gave the numerical values for the mass transfer coefficients in relation to flow rates. It can be seen that the maximum mass transfer coefficient occurred at the highest flow rate of 12 mL/min for the 0.5 mm T-junction with DES-2, which was due to the higher residence time and better internal circulation at higher flow rates.

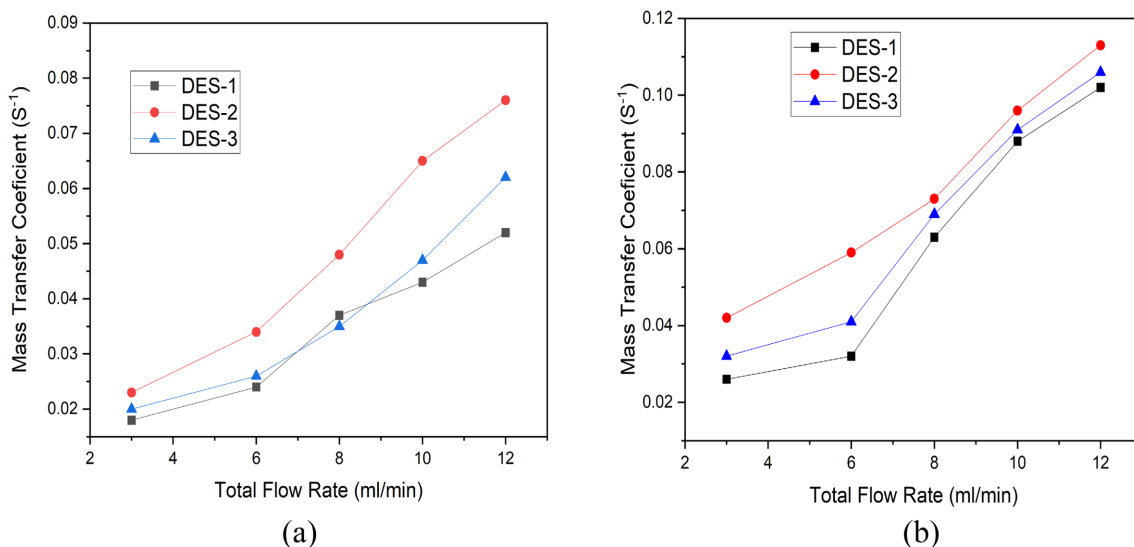


Fig. 8. Mass transfer coefficient with variation in flow rates. (a) 1 mm T-junction, (b) 0.5 mm T-junction.

Table 7. Variation of mass transfer coefficient with different flow rates at 1mm T-junction and 0.5 mm T-junction

Total flow rate (For 1mm T-junction)	DES-1	DES-2	DES-3	Total flow rate (For 0.5 mm T-junction)	DES-1	DES-2	DES-3
3	0.018	0.023	0.02	3	0.026	0.042	0.032
6	0.024	0.034	0.026	6	0.032	0.059	0.041
8	0.037	0.048	0.035	8	0.063	0.073	0.069
10	0.043	0.065	0.047	10	0.088	0.096	0.091
12	0.052	0.076	0.062	12	0.102	0.113	0.106

Table 8. Variation of extraction efficiency with total flow rate for 1 mm T-junction and 0.5 mm T-junction

Total Flow rate (For 1mm T-junction)	DES-1	DES-2	DES-3	Total Flow rate (For 0.5 mm T-junction)	DES-1	DES-2	DES-3
3	30.82	42.08	34.26	3	42.3	48.33	43.91
6	37.08	54.28	38.27	6	50.69	57.25	53.27
8	46.53	59.36	44.08	8	57.23	65.24	58.05
10	57.62	64.28	48.26	10	60.41	71.68	62.83
12	52.41	61.81	50.43	12	68.36	74.32	66.21

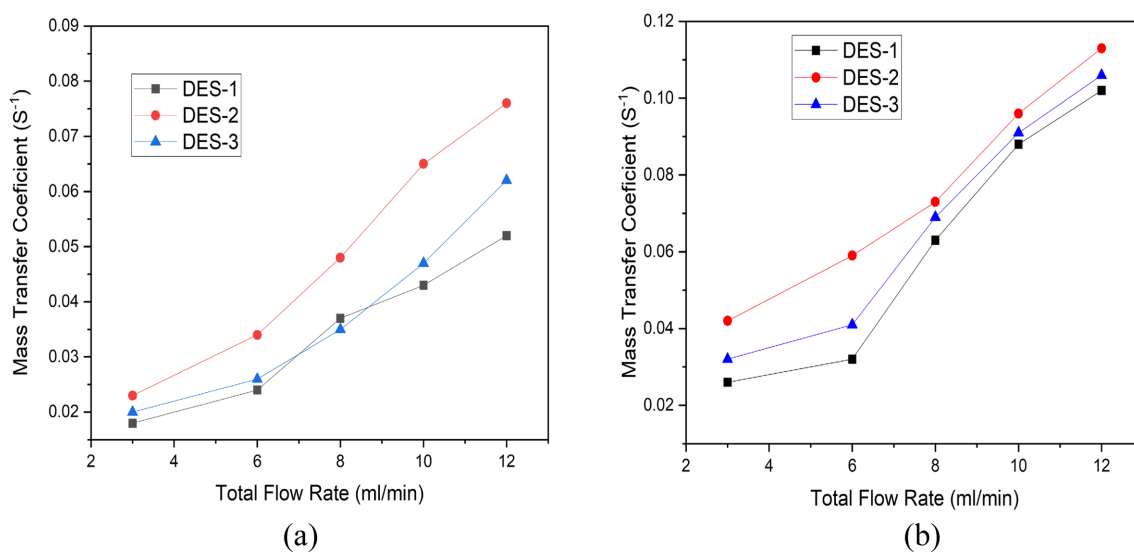


Fig. 9. Variation of mass transfer efficiency with total flow rate. (a) 1 mm T-junction, (b) 0.5 mm T-junction.

The extraction efficiency directly related to the residence time and the internal circulation, slug length, slug interfacial area, as well as slug volumetric flow rates. The extraction efficiencies were calculated by the equation proposed by Zhao et al. (2007) [42].

$$\%E = \frac{(C_{initial} - C_{final})}{(C_{initial})} \times 100$$

where $C_{initial}$ and C_{final} were the initial and final concentration of the aromatic compound. As we increased the flow rate, the length of the slug increased which led to increase the interfacial area whereas the residence time decreased which in turn increased the internal circulation that was directly related to extraction efficiency. For a total volumetric flow rate of 12 mL/min the maximum extraction efficiency was achieved 74% for DES-2 in 0.5 mm T-junction microchannel as shown in Fig. 9.

4. Conclusion

In this study, the liquid-liquid extraction of the aromatic compound toluene from n-hexane using three different deep eutectic solvents (DES) was carried out for slug flow in a circular T-junction microchannel with diameters of 1 mm and 0.5 mm. For this experiment, three DESs were synthesized using various hydrogen bond donors (HBD) and hydrogen bond acceptors (HBA). The variation of slug flow hydrodynamics with respect to flow rates was studied for both microchannels. It was observed that with an increase in flow rates, the length and interfacial area of slugs increased, with the optimum flow rate ranging between 6 to 10 mL/min. At higher flow rates, the size of the slugs increased, causing them to lose their shape, which affected the mass transfer performance. Through this experiment, it was observed the dependency of slug length and interfacial area on flow rate. As we increased the total flow rates, the size of slugs increased, which was required for better mass transfer between the two phases. However, very high flow rates generally led to a decrease in residence time and, thus, a decrease in mass transfer efficiency. Therefore, it was suggested to use an optimum flow rate of 6 to 10 mL/min for this experiment for better mass transfer and extraction efficiency.

The physical properties of the DES solvents could also affect mass transfer performance. For our experiment, temperature changes were related to weather conditions, as we started our experiments when the outside temperature was low and continued when it was high. We studied the temperature variation with the physical properties of the DES solvents and observed that they did not change significantly until the temperature reached a much higher value. Results showed extraction efficiency and volumetric mass transfer for two phases using these three different DES's were best achieved by DES 2 in T-junction microchannel and it has been found that physical properties such as density surface tension and viscosity were changed with increase in temperature and it affected the extraction efficiency. DES-2 was quite stable within the increased temperature range and

gave higher efficiency in both the microchannel.

The highest mass transfer coefficients and extraction efficiency were obtained at 0.113 s^{-1} and 74.32%, respectively, in a 0.5 mm T-junction microchannel for DES-2, which was an MTPB and TEG synthesized DES with a molar ratio of 1:6.

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Authors

Vikas Kumar Choudhary: Ph.D. Research Scholar, Department of Chemical Engineering, MNNIT Allahabad, Prayagraj-211004, UP, India; vikas.chaudhary59@gmail.com

Assistant Professor, Department of Chemical Engineering, Chandigarh University, Mohali-140313, Punjab, India; vikas.e13477@cumail.in

Bushra Khatoon: Ph.D. Research Scholar, Department of Chemical

Engineering, MNNIT Allahabad, Prayagraj-211004, India; bushra.khatoon193@gmail.com

Sadhana Sachan: Professor, Department of Chemical Engineering, MNNIT Allahabad, Prayagraj-211004, India; sadhanas@mnnit.ac.in

M Siraj Alam: Professor, Department of Chemical Engineering, MNNIT Allahabad, Prayagraj-211004, India; msalam@mnnit.ac.in