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Conceptual Process Design for Aromatic/Aliphatic Separation with Ionic Liquids

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Abstract

The investment and annual costs for a separation of aromatic hydrocarbons from a naphtha feed (~10 % aromatics) can be 35 to 70 % lower for an extraction with ionic liquids compared to an extraction with sulfolane. Aromatic hydrocarbons (benzene, toluene, ethyl benzene and xylenes) and C_4 - C_{10} aliphatic hydrocarbons are normally separated by liquid extraction, extractive distillation or azeotropic distillation. The application of ionic liquids for separation processes is promising because of their non-volatile nature. This facilitates solvent recovery using techniques as simple as flash distillation or stripping. Ionic liquids are organic salts that are liquid at low temperature ($T < 100$ °C) and they consist of organic cations based on methylimidazolium, *N*-butylpyridinium and others, and organic or inorganic anions.
The extraction of toluene from a mixed toluene/heptane stream with the ionic liquid 4-methyl-*N*-butyl-pyridinium tetrafluoroborate ([mebupy]BF_4) was evaluated in a pilot plant Rotating Disc Contactor (RDC). With the NRTL interaction parameters determined for this separation, a process model could be developed using ASPEN Plus 12.1. The data measured in the pilot plant and the data calculated with the model are in reasonable agreement with each other. With the results of process simulations, an economic evaluation of the process was made and compared to the extraction process with sulfolane.
The total investment costs in the sulfolane extraction were estimated by UOP, the supplier of this process, to be about M€ 86 for a naphtha feed of 300 t/h containing about 10% aromatic hydrocarbons. The investment costs for an extraction process with the ionic liquid [mebupy]BF_4 was estimated to be M€ 56, including an ionic liquid inventory of M€ 20. The lower investment in the ionic liquid process is mainly due to the fact that the regeneration of the ionic liquid is much simpler than that of sulfolane. Since also the energy costs are lower, the total annual costs with the ionic liquid process are estimated to be M€ 27.4, compared to M€ 58.4 for sulfolane. The main conclusion of the process evaluation is that ionic liquids which show a high aromatic distribution coefficient, $D_{arom} = 0.6$ m/m, with a reasonable aromatic/ aliphatic selectivity, $S_{arom/alif} = 40$, could reduce the investment costs of the aromatic/ aliphatic separation to about M€ 25 to 30 and the annual costs to M€ 16 to 17.

Keywords: ionic liquids, extraction, Rotating Disc Contactor, naphtha cracker, cost evaluation.

1. Introduction

Most ethylene cracker feeds contain 10 to 25% of aromatic components, depending on the source of the feed (naphtha or gas condensate). A typical naphtha feed composition is given in Table 1. The aromatic compounds present are not converted into olefins and even small amounts formed during the cracking process in the cracker furnaces [1]. Therefore, they occupy a large part of the capacity of the furnaces and they put an extra load on the separation section of the C_5^+-aliphatic compounds. Moreover, the presence of aromatic compounds in the feed to the cracker also has a negative influence on the thermal efficiency. Aromatic compounds present in the feed tend to foul the radiation sections (coking of the coils) and the Transfer Line Exchangers.

Table 1. Typical composition of a naphtha feed containing 10 wt% aromatics [DSM]

Component	wt%	Component	wt%	Component	wt%
N-Butane	1.5	Cyclohexane	2.8	2,6-Dimethyl-heptane	1.9
I-Pentane	4.2	2-Methyl-hexane	2.8	Ethyl benzene	2.0
N-pentane	10.3	3-Methyl-hexane	3.8	P-xylene	1.9
Cyclopentane	1.5	N-heptane	4.4	3-Methyl-octane	2.7
2,3-Dimethyl-butane	0.8	Methyl-cyclohexane	4.8	O-xylene	1.0
2-Methyl-pentane	6.0	Toluene	3.0	N-nonane	2.6
3-Methyl-pentane	4.0	2-Methyl-heptane	2.4	N-decane	3.0
N-hexane	8.6	1,3-Dimecyclohexane	7.0	I-decanes	4.0
Me-cyclopentane	4.1	N-octane	5.4		
Benzene	1.8	Ethyl-cyclohexane	2.0	Total	100.3

The separation of aromatic hydrocarbons (benzene, toluene, ethyl benzene and xylenes) from C_4 - C_{10} aliphatic hydrocarbon mixtures is challenging since these hydrocarbons have boiling points in a close range and several combinations form azeotropes. The conventional processes for the separation of these aromatic and aliphatic hydrocarbon mixtures are liquid extraction, suitable for the range of 20-65 wt% aromatic content, extractive distillation for the range of 65-90 wt% aromatics and azeotropic distillation for high aromatic content, >90 wt% [2]. Typical solvents used are polar components such as sulfolane [3-7], N-methyl pyrrolidone (NMP) [6], N-formyl morpholine (NFM), ethylene glycols [7-9] or propylene carbonate [10]. This implicates additional distillation steps to separate the extraction solvent from both the extract and raffinate phases and to purify the solvent, with consequently, additional investments and energy consumption. Overviews of the use of extraction and extractive distillation for the separation of aromatic hydrocarbons from aliphatic hydrocarbons can be found elsewhere [11-14].

According to Weissermel and Arpe [2], no feasible processes are available for the separation of aromatic and aliphatic hydrocarbons in the range below 20% aromatics in the feed mixture. The feed stream of naphtha crackers may contain up to 25% aromatic hydrocarbons, which must be removed. Preliminary calculations, with confidential information from UOP, showed that extraction with conventional solvents is not an option since additional separation steps are required to purify the raffinate, extract and solvent streams, which would induce high investment and energy costs. The costs of regeneration of sulfolane are high, since the sulfolane, which has a boiling point of 287.3 °C, is in the current process taken overhead from the regenerator and returned to the bottom of the aromatics stripper as a vapour [15].

2. Extraction with ionic liquids

The application of ionic liquids for extraction processes is promising because of their non-volatile nature [16]. This facilitates solvent recovery using techniques as simple as flash distillation or stripping. The extraction of toluene from mixtures of toluene and heptane is used as a model for the aromatic/aliphatic separation. A large number of ionic liquids with different cations and anions were tested with a mixture of 10 (v/v)% toluene in heptane as a reference for the selection of ionic liquids for the aromatic/aliphatic separation [17-19]. The solvent sulfolane is used as a benchmark for this separation ($S_{tol/hept} = 30.9$, $D_{tol} = 0.31$ mole/mole at 40 °C), because it is one of the most common solvents for extraction of aromatic hydrocarbons from mixtures of aromatic and aliphatic hydrocarbons used in industry. Therefore, suitable ionic liquids for this separation must show $S_{tol/hep} \geq 30$ and/or $D_{toluene} \geq 0.3$ at 40 °C.
Liquid-liquid equilibrium data were collected for mixtures of 10 (v/v)% toluene in heptane at 40 °C with the selected ionic liquids [20-21]. The distribution coefficient, D_i, is directly calculated from the ratio of the mole fractions in the extract and raffinate phases at equilibrium. The distribution coefficients of toluene and heptane are defined by the ratio of the mole fractions of the solute in the extract (IL) phase and in the raffinate (organic) phase, according to:

$$D_{tol} = C^{IL}_{tol}/C^{org}_{tol} \text{ and } D_{hept} = C^{IL}_{hept}/C^{org}_{hept} \qquad (1)$$

The selectivity, $S_{tol/hept}$, of toluene/heptane is defined as the ratio of the distribution coefficients of toluene and heptane:

$$S_{tol/hept} = D_{tol}/D_{hept} = (C^{IL}_{tol}/C^{org}_{tol})/(C^{IL}_{hept}/C^{org}_{hept}) \qquad (2)$$

The IL [mebupy]BF_4 exhibited the best combination of a high toluene distribution coefficient ($D_{tol} = 0.44$ mole/mole) and a high toluene/heptane selectivity ($S_{tol/hept} = 53$) at 10 (v/v)% toluene and 40 °C [17-19]. Therefore, this IL was selected for the extractive removal of toluene from a toluene/heptane mixture in a pilot scale Rotating Disc Contactor (RDC, Figure 1) [22].

3. Rotating Disc Contactor

The pilot RDC extraction column is shown in Figure 1. The column consists of five jacketed glass segments of each 360 mm in length and an inside diameter 60 mm, with each eight stirred compartments. Settlers of 240 mm (bottom) and 210 mm (top) with an internal diameter of 90 mm enclose the stirred segments. The internals of the stirred segments consist of alternating discs and doughnuts. The outside diameter of the doughnuts is 60 mm, the inner 22 mm and the thickness is 1.5 mm. The discs have a diameter of 40 mm and a thickness of 1.5 mm. The distance between two doughnuts is 32 mm, just as the distance between two discs. The internals were made of stainless steel.

Figure 1. Pilot Rotating Disc Column

The concentration profiles of toluene and heptane in both phases along the column were determined by taking samples at different column heights through the sample ports. The sample ports consisted of two metal tubes and inside one of the two tubes a Teflon tube was placed. The solvent was fed to the top of the column and the extract phase was collected from the bottom settler. The heptane/toluene phase was fed from the bottom and the raffinate phase was collected from the top settler.

The solvents were regenerated in the extraction column at 80^{o}C by bubbling nitrogen through the column for 16 to 22 hours. Total removal of heptane was possible, but total removal of toluene was not: 0.78 mole% was left in the sulfolane and 0.88 mole% in [mebupy]BF_4. The regenerated ionic liquid was used in the extraction experiments.

Until this date, no pilot plant scale experiments with extraction with ionic liquids have been carried out. The RDC was chosen because it is the most commonly used extractor in petrochemical processing. The pilot RDC provided good results for the toluene/heptane separation: small droplets (high mass transfer rates) were obtained and the column capacity was high. Lower solvent to feed ratios were required with the IL than with sulfolane to extract the same amount of toluene, while a 10% higher flux (volumetric throughput) and similar mass transfer efficiency were obtained. The best performance proved to be at the highest rotation speed used (643 rpm). In Figure 2, the number of equilibrium stages, N_S, as function of the rotating speed is depicted. The highest N_S is obtained with [mebupy]BF_4 at high rotor speeds and at a flux of 10.8 m^3/m^2.h. Excellent hydrodynamic behaviour was observed and about three equilibrium stages were contained in the 1.80 m high active section of the column.

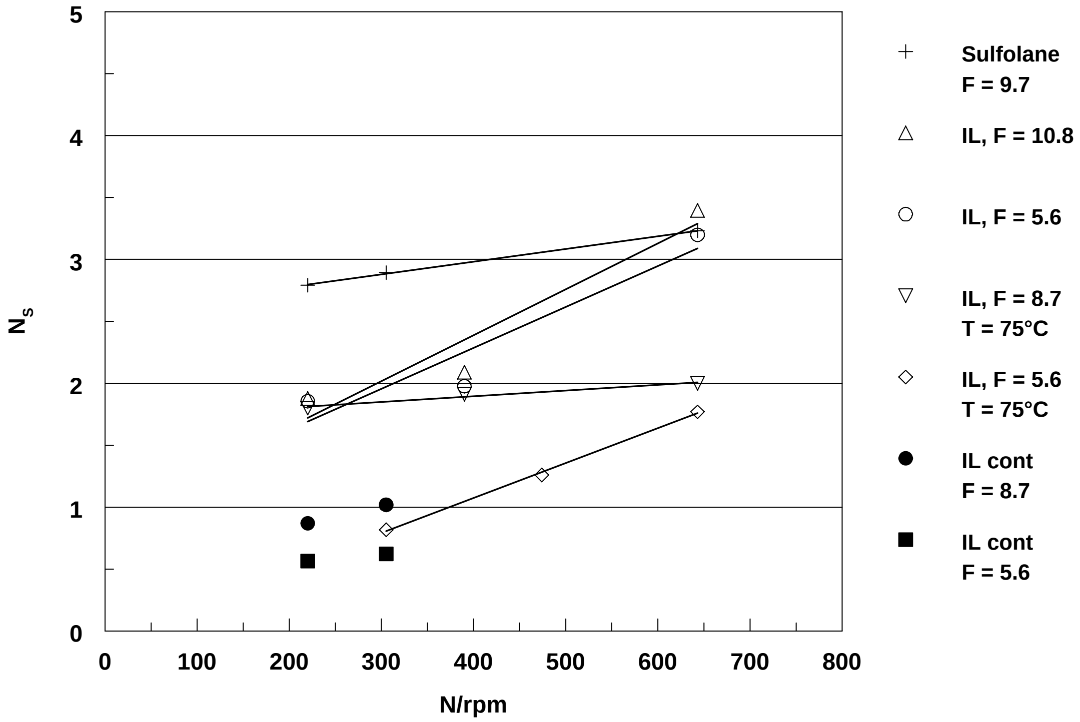

Figure 2. Number of equilibrium stages with sulfolane and [mebupy]BF_4 in the RDC, T = 40°C, ~ 10% toluene, flux in m^3/m^2.h

With sulfolane as the solvent, the feed flow was 5.8 kg/h with a total flux of 9.7 m^3/m^2.h and with [mebupy]BF_4 as the solvent, the feed flow was 10 kg/h with a total flux of 10.8 m^3/m^2.h. This means that more toluene can be extracted with the ionic liquid as solvent than with sulfolane.

4. Extraction of toluene from toluene/heptane with [mebupy]BF_4

In Table 2, the experimental and calculated data of the extraction with the ionic liquid [mebupy]BF_4 are shown. The number of stages in the pilot plant for this extraction is about three with the highest rotation speed and the highest flux. Therefore, this number of stages was also used in a process simulation with ASPEN Plus 12.1.
The ionic liquid used for the extraction of toluene contains a small amount of toluene, because regenerated ionic liquid was used. Comparing the experimental and calculated data in Table 2, it can be concluded that the measured and calculated data are in reasonable agreement with each other. The largest difference is the concentration of the ionic liquid in the raffinate phase. The average concentration of the ionic liquid in the raffinate phase measured with equilibrium experiments was 0.001 mole fraction or 0.24 wt% and this value compares very well with the one measured during the extraction in the column (0.26 wt%). The calculated ionic liquid concentration in the raffinate phase is 0.01 wt%. The experimental concentration of toluene in the extract phase is 1.38 wt% and is somewhat lower than the calculated value of 1.56 wt%. The calculated heptane concentration in the extract is 0.36 wt% and the experimental value is 0.14 wt%. The calculated concentrations of toluene and heptane in the raffinate phase are quite similar to the ones calculated with the flow

sheeting program. Therefore, the ASPEN Plus flow sheeting program can be used to generate reasonably accurate data for process evaluation.

Table 2. Experimental and calculated data for the extraction of toluene with [mebupy]BF_4 in three stages.

Parameters	Feed	[mebupy]BF_4	Raffinate	Extract
Pilot plant				
T, °C	40	40	40	40
P, atm	1	1	1	1
Flow, kg/h	10	20	n.a.	n.a.
Toluene, wt%	7.55	0.58	5.91	1.38
Heptane, wt%	92.45	-	93.83	0.14
[mebupy]BF_4, wt%	-	99.42	0.26	98.48
ASPEN				
T, °C	40	40	40	40
P, atm	1	1	1	1
Flow, kg/h	10	20	9.73	20.27
Toluene, wt%	7.55	0.58	5.71	1.56
Heptane, wt%	92.45	-	94.28	0.36
[mebupy]BF_4, wt%	-	99.42	0.01	98.08

Several process simulations were carried out with the ionic liquid [mebupy]BF_4 as the solvent, with the objective to establish the minimum solvent-to-feed ratio and the number of stages for a toluene recovery of at least 98% and a heptane purity in the raffinate of at least 98%. In order to achieve a toluene recovery of at least 98%, the number of stages must be higher than 6. For a purity of heptane in the raffinate of at least 98%, the S/F ratio must be above 1.65 with ten or twelve stages. Since the S/F ratio will be in the range of 2.3 on mole basis, or 5.5 on mass basis, with 12 stages in order to achieve a toluene recovery of at least 98%, the obtained heptane purity will always be higher than 99.5%. In Table 3, the results of the simulation of the extraction with [mebupy]BF_4 in 12 stages is given.

Table 3. Results of the extraction of toluene and n-heptane with [mebupy]BF_4 in 12 stages.

	Feed	IL	Raffinate	Extract	Split ratio, %	
					Raff.	Extr.
Temperature, °C	40	40	43.9	41.3		
Pressure, bar	1.013	1.013	1.013	1.013		
Mole flow, kmol/h	10	23.1	8.74	24.36		
Mass flow, kg/h	993.35	5475.85	875.49	5593.71		
Volumetric flow, m^3/h	1.45	4.66	1.31	4.80		
Mass flow, kg/h						
Toluene	99.34		1.95	97.39	1.96	98.04
N-heptane	894.01		873.49	20.52	97.70	2.30
[mebupy]BF_4	0	5475.85	0.05	5475.80	0.00	100.00

5. Economic Evaluation

If a major part of the aromatic compounds present in the feed to the crackers could be separated upstream of the furnaces, it would offer several advantages: higher capacity, higher thermal efficiency, and less fouling.

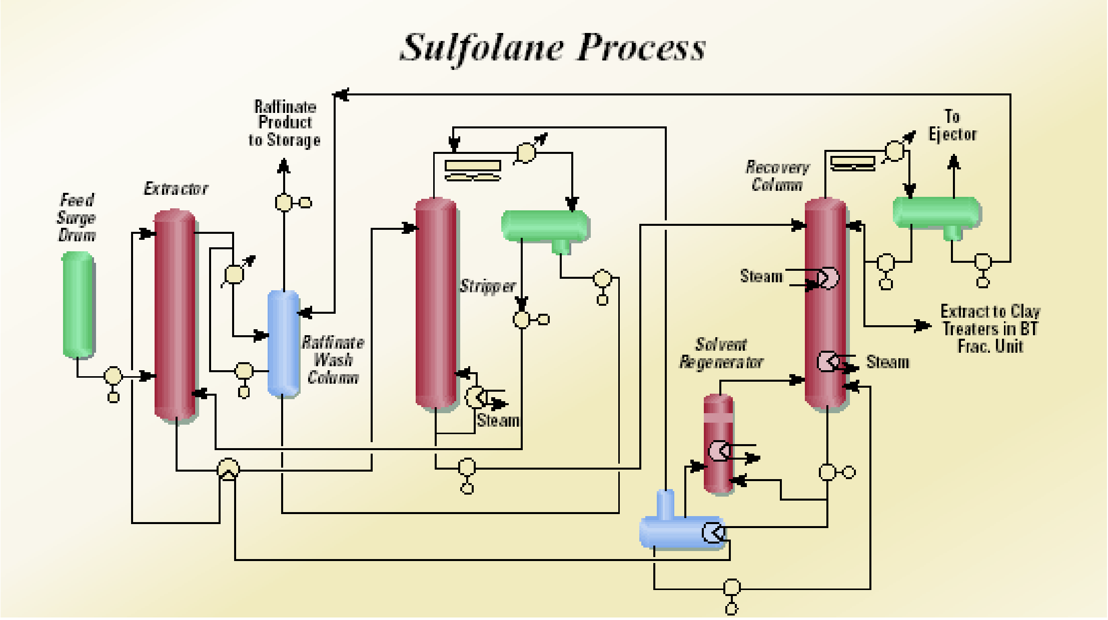

Figure 3. UOP Sulfolane process

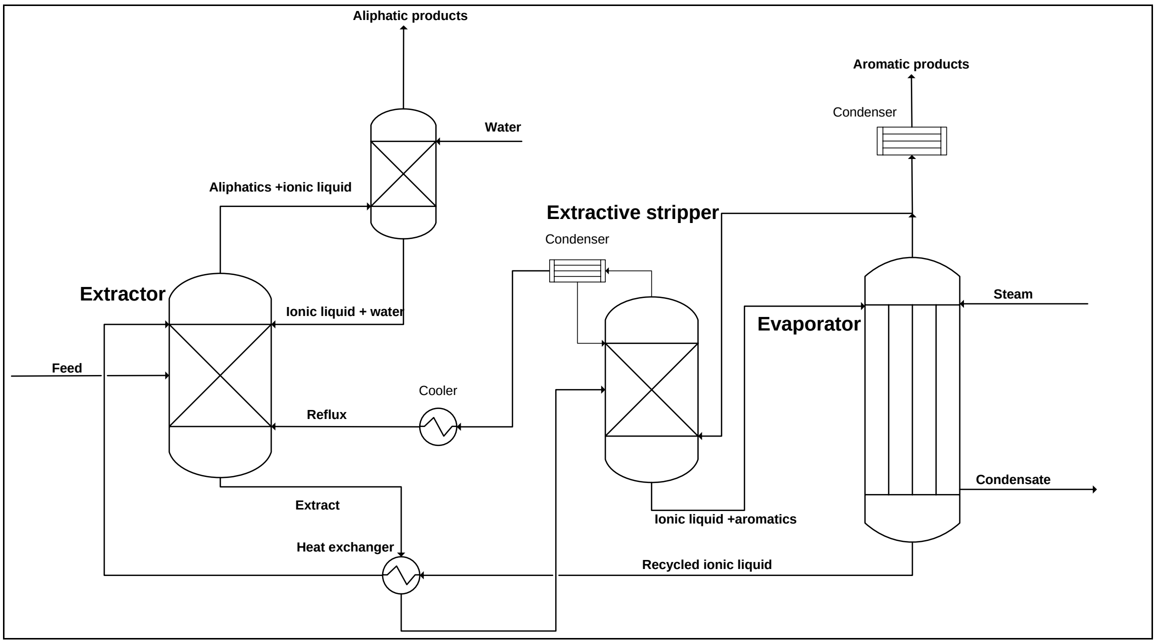

Figure 4. Conceptual flow scheme for the separation of aromatic and aliphatic hydrocarbons

The sulfolane process is one of the most used processes for the separation of aromatic and aliphatic hydrocarbons. Therefore, the cost of an extraction with an ionic liquid is estimated by comparing the sulfolane process with an ionic liquid process. According to UOP, the extraction of aromatics from naphtha cracker feed is feasible with the Sulfolane (Figure 3) or Carom process. Normally, this process is used for feed streams containing 68% aromatics or more (reformed petroleum naphtha, pyrolysis gasoline or coke oven light oil). UOP has made a rough estimate for the sulfolane process for a feed stream of 300 t/h with 10% aromatics in the feed.

Comparing the extraction processes with sulfolane (Figure 3) and with [mebupy]BF_4 (Figure 4), it can be stated that the extraction column will have about the same dimensions, because the flow of the ionic liquid is about the same as that with sulfolane. The solvent to feed ratio on mole basis is lower with the ionic liquid than for sulfolane, but the molecular weight of the ionic liquid is almost twice as high as that of sulfolane and, therefore, the about same amount of solvent is used on a weight basis.

The most expensive equipment in the sulfolane process is not the columns, vessels etc., but the heat exchangers, reboilers and coolers, which make up almost 65% of the total investment. The investment in the columns is about 28% of the total investment costs. The high share of the heat equipment is caused by the following reasons: the extraction is carried out at 100 °C, the extractive stripper and the recovery column operate at 190 °C and sulfolane, which has a boiling point of 287.3 °C, is taken from the top of the solvent regenerator column. These high temperatures require the use of high-pressure steam at several places, as can be seen in Figure 3. The high temperature has also another adverse effect: sulfolane decomposes at high temperatures and the decomposition products cause unacceptably high corrosion in the paraffin stripper and aromatics stripper if oxygen intrusion occurs [15].

Table 4. Estimated investment and variable costs in M€

	Sulfolane (UOP)	[mebupy]BF_4
Materials and labour	46	21
Engineering	15	6
Inside battery limits	61	27
Outside battery limits	20	9
Solvent inventory	5	20
Total investment costs	86	56
Annual costs, 40% of total I	34.4	22.4
Energy costs	24	5
Total annual costs	58.4	27.4

In Table 4, extraction with sulfolane and with the ionic liquid [mebupy]BF_4 is compared. Of course, the estimations for the extraction with [mebupy]BF_4 are crude in this stage and they will require more detailed investigation. But due to the fact that an ionic liquid has no vapour pressure, the regeneration of this solvent is much simpler than that of, for instance, sulfolane and low pressure steam can be used. Instead of two distillation columns required for the recovery of the aromatics and the regeneration of the solvent, only one flash column or stripper with for instance nitrogen is required for the ionic liquid process, see Figure 4. Therefore, also the process temperatures are lower in the ionic liquid process than in the sulfolane process. The amount of energy required in the ionic liquid process is calculated by the flow sheeting program of ASPEN.

Assuming that the investments in the heat exchangers, reboilers and coolers is proportional to the duty, the investment in heat equipment in the ionic liquid process will then be about 20 % of that of the sulfolane process. In the IL process, the recovery column and the solvent regenerator are replaced by one flash column and it is assumed that the investment in the extractor, extractive stripper and the flash column for the IL process will be lower than the investment in the extractor, extractive stripper, sulfolane regenerator column and recovery columns of the sulfolane process. The investments in the other equipment will be in the same range, because the flows will also be in the same range. The total investment in the IL process is then estimated to be about 65% of that of sulfolane.

The annual variable capital costs (depreciation, 10%; ROI, 20% and other investment related costs, such as maintenance, 10%) are estimated to be 40% of the investment costs. The energy costs for the sulfolane process are estimated by UOP to be € 10/ton feed, which will amount to M€ 24/year. The total annual costs for the extraction with sulfolane will then be M€ 58.4/year.

The improved margin for removing the aromatic hydrocarbons from the feed to the naphtha cracker will be around € 20/ton feed due to lower operational costs, which amounts to M€ 48/year for a naphtha cracker with a feed capacity of 300 ton/h. With the sulfolane process, this will result in an operational loss of around M€10/year and a profit of M€ 20/year for the ionic liquid process. Therefore, it is obvious that the sulfolane extraction process is not in operation anywhere for removing aromatic compounds from streams containing relatively low concentrations of aromatic compounds. Although the calculations for the investment and energy costs for the IL process are very crude, the margin between the sulfolane and the IL process is large enough to recommend further research in this process. In Figure 5, the investment and the total annual costs for the separation of 10% aromatics from a cracker feed with sulfolane and several ionic liquids are shown.

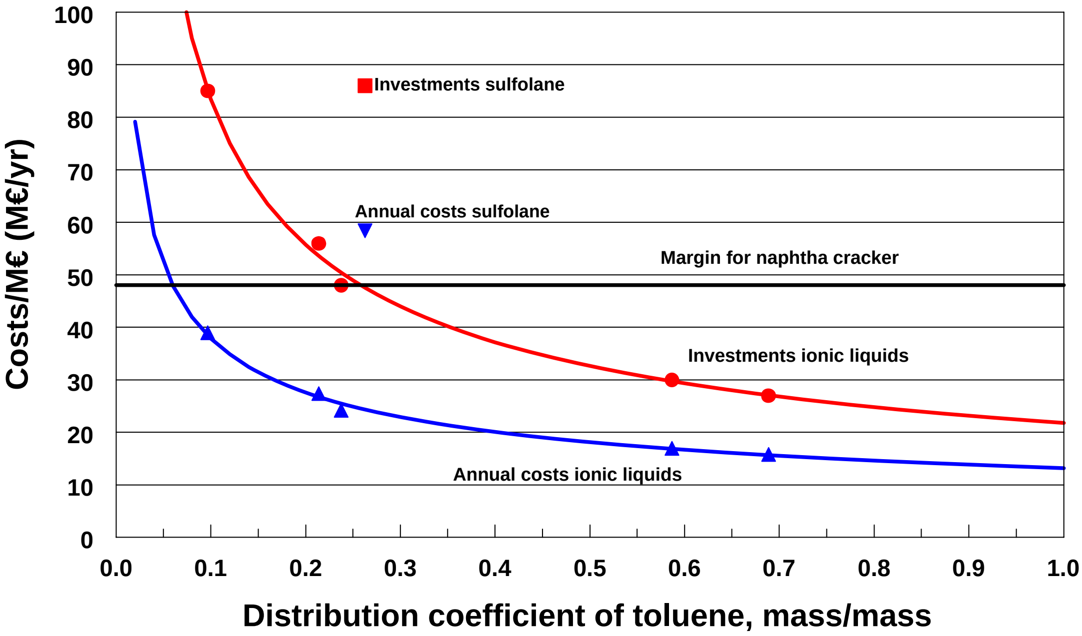

Figure 5. Investment and variable costs for extraction with ionic liquids

In the calculations, an ionic liquid price of € 20/kg was used and BASF, a major producer of imidazole, one of the primary products for ionic liquids, has indicated that it is indeed possible to reach a level of € 10 – 25/kg with production on a large scale [23-25]. The loss of ionic liquid to the raffinate phase is minimal, estimated to be 0.006%. However the ionic liquid can be recovered with an extraction with water.

6. Conclusions

A high aromatic distribution coefficient is the key factor for a feasible extraction process with ionic liquids, provided the aromatic/aliphatic selectivity is high enough, around 30 or higher, because the price of the ionic liquids will probably always be higher than that of sulfolane.

The toluene/*n*-heptane extraction can be simulated by the flow sheeting program in ASPEN Plus 12.1. The results of this simulation and the actual results obtained in a pilot plant extraction column are in reasonable agreement with each other. The complete removal of the ionic liquid from the raffinate stream is a prerequisite for an economically feasible aromatic/aliphatic separation. For water-soluble ionic liquids, washing with water is a possibility, but this must be experimentally proven.

The energy requirement for an extraction process with an ionic liquid as solvent is much lower than that with sulfolane, due to the lower process temperatures in the extraction with the ionic liquid process and the simpler recovery of the ionic liquid.

The investment costs are about 35 % lower for the extraction with [mebupy]BF_4 than with sulfolane. If ionic liquids are used with higher aromatic distribution coefficients, the investment costs can even be decreased by almost 70% compared to the sulfolane process.

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