Investigation of suitable chemicals for coupled extraction and distillation at pilot scale

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Introduction

The liquid-liquid-extraction set-up and continuous distillation unit present in lab 319 are an important part of the process engineering course, because here the process engineer becomes familiar with continuous separation processes in the chemical industry. These set-ups originally worked separately with water, cyclohexane, 2-propanol, ethyl acetate and 1-propanol resulting in a large amount of chemical waste and an expensive experiment.

Figure 1: The distillation unit

Figure 2: The extraction unit

In this research, an attempt was made to find alternative chemicals through which the extraction and distillation set-ups could be paired so that the raffinate from the extraction feeds the distillation unit. For instance, ethyl acetate, 1- and 2-propanol were replaced by ethanol. Our objective was to replace cyclohexane with 1-hexanol, n-butyl acetate, D-limonene or linalool. For this purpose, the calibration curves and ternary diagrams of the water-ethanol-X mixtures were determined. Afterwards, the best candidates were tested and suitable settings for continuous operation of the set-up were examined.

Materials and methods

For the set up of the ternary diagrams the correlation between the density, refractive index and the composition of each mixture was determined from the literature based on calibration mixes. This relationship was used to determine the exact mass fractions in the equilibrated phase of water, ethanol and n-butyl acetate/D-limonene/linalool/1-hexanol allowing us to construct the ternary diagrams.

Eight mixtures were prepared to set up the calibration curves. The composition of the mixtures were predetermined (literature) to fall outside the two-phase range. This to prevent segregation of the mixtures for the calibration curves. Three pure substances, two binary mixtures and three ternary mixtures were used. Each of these mixtures were subjected to a density and a refractive index measurement. The exact composition of the mixtures was calculated using the total least squares method.

We use a set-up from Normag for the liquid-liquid extraction and as well as for the distillation. During the extraction, a two-phase system of immiscible liquids is used, resulting in the formation of a polar water layer and an apolar organic phase. The continuous distillation unit will separate the liquid mixture by difference in boiling temperature, creating two fractions: one rich of substance with a lower boiling point and one rich of substance with a higher boiling point.

Results and discussion

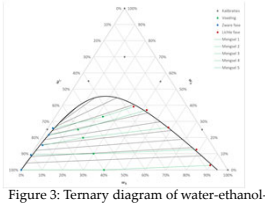

Figure 3: Ternary diagram of water-ethanol-1-hexanol

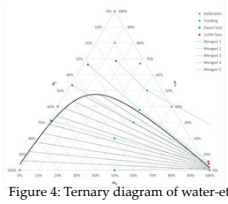

Figure 4: Ternary diagram of water-ethanol-D-limonene

1-hexanol was chosen as a good replacement for cyclohexane because:

- Binodal curve lies close towards the ethanol-water side
- Tie-lines don't converge into a point
- 1-hexanol has the least safety hazards of all possible substituents

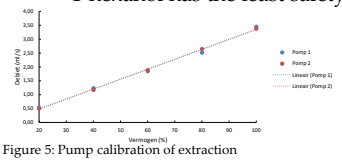

Figure 5: Pump calibration of extraction

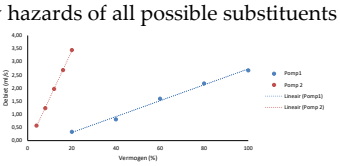

Figure 6: Pump calibration of distillation

Pump calibrations made continuous operation possible:

Extraction
- Pumps are equally strong
- P01 and P02 40% power

Distillation
- Pump P02 is much stronger
- P01 at 85% and P02 at 11% power

Table 1: Set-up for the extraction unit

Extraction with ethanol – water -				
	Before extraction		**After extraction**	
	Light fase	**Heavy fase**	**Light fase**	**Heavy fase**
Hexanol 1	50% hexanol 50% ethanol	100% water	3,35% ethanol 91,23% hexanol 5,42% water	27,30% ethanol 3,30% hexanol 69,40% water
Hexanol 2	50% hexanol 50% ethanol	100% water	6,16% ethanol 87,56% hexanol 6,28% water	20,34% ethanol 3,68% hexanol 76% water
N-butylacetate	50% n-butylacetate 50% ethanol	100% water	31,11% ethanol 55,78% n-butylacetate 13,11% water	15,99% ethanol 0,58% n-butylacetate 83,43% water

For the set-up of the continuous distillation the mass balance implies that the ingoing flow from the feed (20% ethanol and 80% water) is equal to the outgoing flow from distillate and soil residue. This gave the following settings: P01 = 64%, P02 = 9%, 3s reflux and 3s removal. False alarm when water in reboiler was too low. This interrupted the continuous working of the distillation column. Further optimalisation can be achieved by adjusting the reflux ratio and flow rate.

Direct coupling of the extraction and the distillation units is currently not possible. A buffer vessel would be needed to separate the extract. At the bottom an extra pump has to be installed so a relatively pure extract can be pumped to the distillation feed. Both the extract and the distillate can be purified by an extra cycle.

Conclusion

We set out on this project to find a combination of three chemicals that would work in a coupled extraction-distillation setup on a pilot scale. Two of these substances were already decided to be ethanol and water. A lab scale exploration of the problem was preformed using different solvent to determine their suitability for the final setup. These tests pointed to two solvents that showed the most promise : 1-hexanol and n- butyl acetate. Both were used in a pilot scale with ethanol and water to determine which mixture showed the most favorable results. The resulting extract from both runs was of appropriate purity, but the raffinate of the n-butyl acetate mixture contained significant ethanol content. As such the 1-hexanol mixture was decided on over the alternatives.

During this project, continuous distilation of an ethanol-water mixture was succesfully excecuted. Based on the purity of destilate and rafinate it was determined that the settings could still be further optimized, as the azeotrope was not reached. Increasing the reflux ratio may improve this.
While we did not achieve the coupled extraction-distillation setup as outlined in our hypothesis, using the conclusions reached in this research it will be possible to link both setups in the future. Further, we did achieve our goal to limit the volume of solvents used and hazerdous waste produced when operating the setup.

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