HOW TO SEPARATE TWO NEAR AZEOTROPES: CASE OF THE SEPARATION OF THE MIXTURE PHENOL – HYDROCARBONS

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The separation of a mixture in its components is possible using distillation, on principle with admissible energy consumption, when relative volatility is greater than 1.05. When relative volatility is lower than 1.05 separation using distillation is possible with a greater number of tray and a greater reflux ratio. What happened when it have to separate an azeotropic mixture (relative volatility equal with 1.0)?

The paper presents a new process for phenol separation from the mixture phenol-hydrocarbons using azeotropic distillation with water. The mixture phenol-hydrocarbons is obtained at the top of one column in the separation system of an acetone-phenol plant. The process presented in this work was simulated with PRO/II from Simulation Science. Because the data about vapour-liquid equilibrium were not completed, it was necessary to obtain experimental values for vapour-liquid equilibrium for the system cumene-α-methyl styrene. The data were obtained on a Normag type apparatus and were regressed in order to obtain NRTL interaction binary parameters.

This paper proposes a new process with a very simple flow sheet. It is used only one column, at atmospheric pressure, with a small number of trays and like entrainer, water, easy to find and, practically, free. Using water like entrainer is an original proposal and it is the subject of a Romanian patent. Optimal variant was obtained after many simulations, the system water-hydrocarbons-phenol being strongly non-ideal (two liquid phases on the trays in the column). After these simulations we concluded:
- 12 theoretical trays are enough to obtain a phenol content of 100 ppm weight in the top product;
- the water flow must be sufficient for the two azeotropes, and a supplement to obtain top product with only 100 ppm weight phenol.

The results of the computer simulation were experimentally verified. At the very beginning, we choose to use Sulzer packing in the real column. These systems, with two liquid phases, present low trays efficiency for the valve trays. To have a good design of the real column it is necessary to have the separation efficiency of the Sulzer packing in such systems (HETP). The date about HETP of the Sulzer packing are missing and, to obtain them, we used an experimental column with 1 m Sulzer packing, 30 mm diameter, operated at infinite reflux ratio. The composition of the product was compared with the results of computer simulation for different increasing number of theoretical trays. The result of this originally procedure is that 1 m Sulzer packing is equivalent with six theoretical trays. This result permit us to verify the computer simulations with experimental data obtain on a continuous operated column. The column has two zones, each of them with 1 m height Sulzer packing and with 30 mm diameter.

These results show, without doubts, that the process is feasible. Computer simulations data are confirmed by the experimental data obtain on lab column. In this way are validated:
(1) simulation model;
(2) vapour liquid-liquid experimental data for the system cumene – α-methyl styrene;
(3) thermodynamic data supply by PRO/II;
(4) efficiency of the Sulzer packing obtained on the lab column with originally procedure.

This process was verified on an industrial column operated in continuous system and the results confirm all above. This was a strong argument for the validity of the new process that passes all validation steps: experimental, regression, computer simulation, verification on lab and industrial scale.

Also, an economically analyze on industrial experiment shows that this new process is more advantageous than the old processes.