Data Article

Data on conceptual design and simulation of reactive distillation process

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ABSTRACT

The simulation of the reactive distillation for the esterification of acetic acid with methanol is carried out using the equilibrium stage model. The pseudo-homogeneous kinetic rate equation is used in equilibrium stage model to perform simulation in Aspen plus Version 7.3. The different parameters like the reflux ratio, number of stages, feed location of the acetic acid are used to obtain the data of mixture composition and acetic acid conversion. The non-ideal behavior of the system is accounted by NRTL, Wilson and UNIQUAC methods. All the thermodynamic models are able to generate data of compositions very well. The composition profiles with different activity based models compared and there is little deviation of water and methanol mole fractions. The optimum number of stages for the present system is 30 for achieving the higher conversion as well as the purity of the distillate. The optimum reflux ratio is 1.9, feed flow rate of acetic acid and methanol is 10.2 mol/hr, location of acetic acid above the reactive zone and methanol below the reactive zone gives the 99.5% by mole of the methyl acetate and 99% acetic acid conversion.

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1. Data

The experimental data on reactive distillation is scarcely available in the literature. The data article presents data on the comparison of experimental and simulation data for the esterification of acetic acid with methanol in the presence of Indion 180 solid catalyst. The data generated for simulations data of reactive distillation by ASPEN PLUS software. The experimental data along with simulation data by accounting the non-ideal behavior of the system is presented for the esterification of acetic acid with methanol. Very few studies have the experimental and simulation data for this system in presence of solid catalysts like Indion 180 [1,2]. Table 1 and Table 2 show the feasibility of the system and azeotropic conditions. The simulations and experimental data for the temperature are given in Table 3. The compositions data of simulations and experimental is shown in Table 4. Fig. 1a and b shows the ternary plot of the reactive distillation for the methyl acetate/methanol/acetic acid and water/acetic acid/methyl acetate. Fig. 2 shows the temperature profile as function of the stage numbers. Whereas Fig. 3 shows the liquid composition profiles as function of the stage numbers for different thermodynamic

| Table 1 | Mixture investigated for Azeotropes at a pressure of 101325 N/m². |
|---------|---------------------------------------------------------------|
| Comp ID | Component Name      | Classification   | Temperature |
| -----   | -----------------   | ---------------- | ----------- |
| METHA-01 | METHANOL        | Stable Node      | 64.53°C    |
| METHY-01 | METHYL-ACETATE  | Stable Node      | 57.05°C    |

| Table 2 | Azeotropic composition at a pressure of 101325 N/m². |
|---------|--------------------------------------------------------|
| Number Of Components: 2 | Temperature 53.64°C |

|                | Classification: Unstable Node | MOLE BASIS | MASS BASIS |
|----------------|-------------------------------|------------|------------|
| Homogeneous    |                              |            |            |
| Methanol       | 0.3341                        | 0.1783     |            |
| Methyl acetate | 0.6659                        | 0.8217     |            |
models for both experimental and simulation data. The presented data is useful to the further esterification reaction process as a basic system.

2. Experimental design, materials, and methods

The kinetic rate equation is obtained by conducting the experiments in a batch reactor under different temperatures and catalyst concentrations [1]. The experimental data for the esterification of acetic acid and methanol in presence of Indion 180 catalyst in a batch reactor under different temperatures, catalyst concentrations and mole ratio of acetic acid to methanol are investigated. From that experimental data the kinetic parameters are obtained by calculating the error between the experimental data and model predictions of pseudo homogeneous kinetic model.

The simulations have been carried out for the reactive distillation column by incorporating the developed kinetic rate equation using Aspen Plus. The parameters used for simulation of reactive distillation column are total height of the column has the 3 m and 50 mm diameter. The total operating pressure is 1 atm. The reactive zone contains 10 equilibrium stages whereas non reactive zone contains 20 equilibrium stages including condenser and reboiler.

All the packing characteristics are assumed as per the Katapak-S commercial packing in the reactive zone and wire mesh packing characteristics in the non reactive zone; that is in rectifying and stripping zones.

Fig. 1a and b shows the conceptual design of the methyl acetate synthesis by the esterification of acetic acid and methanol. The mixture azeotropic temperature at 101325 N/m² is shown in Table 1. The azeotropic composition of the multi-component is shown in Table 2. The methanol/methyl acetate

| T_{model} | T_{experimental} |
|-----------|------------------|
| 1         | 330.07714        | 328            |
| 2         | 330.121285       |                |
| 3         | 330.141982       |                |
| 4         | 330.152321       |                |
| 5         | 330.158783       | 333            |
| 6         | 330.165264       |                |
| 7         | 330.181987       |                |
| 8         | 330.268162       |                |
| 9         | 330.789109       |                |
| 10        | 333.714044       | 337            |
| 11        | 344.640483       |                |
| 12        | 344.675501       |                |
| 13        | 344.724665       | 338            |
| 14        | 344.803033       |                |
| 15        | 344.93169        |                |
| 16        | 345.111302       |                |
| 17        | 345.18586        | 339            |
| 18        | 344.486431       |                |
| 19        | 344.367541       | 339            |
| 20        | 344.525245       | 339            |
| 21        | 346.626655       |                |
| 22        | 347.079443       |                |
| 23        | 347.222137       |                |
| 24        | 347.390824       |                |
| 25        | 347.82194        | 339            |
| 26        | 349.082241       |                |
| 27        | 352.634392       |                |
| 28        | 360.269712       |                |
| 29        | 368.589481       |                |
| 30        | 372.593451       | 358            |
forms azeotropes of composition 0.3341/0.6659 at a temperature of 53.64°C. The methyl acetate/water forms the azeotropes at composition of 0.92/0.08 and at a temperature of 56.2°C [3].

The variation of the temperature from top of the column (starting stage) to the bottom of the column is shown in Fig. 2. The temperature is increasing stage 1 to stage 3 and then maintains constant till 15th stage. The temperature suddenly rose from stage 15th to 18th stage and then falls. It is because, the reaction is taking place in the reactive zone and heat is liberated due to slightly exothermic reaction. Then the sudden fall of temperature is happening due to supply of the cold methanol at 20th stage. In the stripping zone, 21st to 30th stage there is raise in temperature due to supply heat generates more vapors which are mixed with liquid [8–10]. Hence the overall temperature is decreasing from the reboiler to condenser stage. The temperature data by simulation and experimental data is shown in Table 3.

Fig. 3 shows the composition profiles with respect to stage number for different thermodynamic activity models at a reflux ratio of 1.9 and at equimolar ratio of feed flow rates to the column. The methyl acetate composition is increasing from the bottom to the top of the column due to the high volatility of the methyl acetate where as the composition of the water is increasing from top to bottom of the column [8–10]. The reactants should have the high concentrations in the reactive zone to give high conversion of the acetic acid. Acetic acid has high concentration from stages 10–20 and methanol have high concentration from stages 20–30. The reactants concentrations are almost negligible in distillate and reboiler. Hence the reactive distillation is able to give almost 99.6% of the distillate and bottom products at stoichiometric ratio. Among the activity models UNIQUAC model is able give more water mole fraction in the reboiler and methyl acetate in the distillate almost in greater than 99%. Hence this

| Liquid compositions by UNIQUAC model | Liquid compositions by experiment |
|--------------------------------------|---------------------------------|
| Acetic acid | Methanol | Methyl acetate | Water | Acetic acid | Methanol | Methyl acetate | Water |
| 1 | 0 | 0.001535 | 0.991163 | 0.007302 | 0 | 0.04909 | 0.95091 | 0 |
| 2 | 0 | 0.000742 | 0.993979 | 0.005279 | 0 | 0.09 | 0.87 | 0.04 |
| 3 | 0 | 0.000498 | 0.995172 | 0.004329 | 0 | 0.0003546 |
| 4 | 0 | 0.000423 | 0.995688 | 0.003886 | 0 | 0.00356 |
| 5 | 0 | 0.0004 | 0.995905 | 0.00368 | 0 | 0.003773 |
| 6 | 0 | 0.000393 | 0.995928 | 0.003585 | 0 | 0.004998 |
| 7 | 0.000602 | 0.000391 | 0.995462 | 0.003546 | 0 | 0.008498 |
| 8 | 0.003829 | 0.000391 | 0.992221 | 0.00356 | 0 | 0.015551 |
| 9 | 0.023761 | 0.000394 | 0.972073 | 0.003773 | 0 | 0.025645 |
| 10 | 0.128522 | 0.000412 | 0.866069 | 0.004998 | 0 | 0.040785 |
| 11 | 0.428232 | 0.000436 | 0.562833 | 0.008498 | 0 | 0.065055 |
| 12 | 0.428232 | 0.000761 | 0.555456 | 0.015551 | 0 | 0.107868 |
| 13 | 0.42832 | 0.001219 | 0.544817 | 0.025645 | 0 | 0.119153 |
| 14 | 0.428475 | 0.001872 | 0.528868 | 0.040785 | 0 | 0.208892 |
| 15 | 0.428228 | 0.002845 | 0.503872 | 0.065055 | 0 | 0.208892 |
| 16 | 0.428489 | 0.004431 | 0.462811 | 0.107868 | 0 | 0.208892 |
| 17 | 0.40637 | 0.007808 | 0.391021 | 0.194801 | 0.09654 | 0.2806 | 0.51517 | 0.1077 |
| 18 | 0.320267 | 0.021864 | 0.24375 | 0.41412 | 0 | 0.320267 |
| 19 | 0.080518 | 0.101316 | 0.047474 | 0.770693 | 0 | 0.080518 |
| 20 | 0.003696 | 0.457887 | 0.014882 | 0.523536 | 0 | 0.003696 |
| 21 | 0.003691 | 0.468449 | 0.002736 | 0.525124 | 0 | 0.003691 |
| 22 | 0.003691 | 0.469832 | 0.000492 | 0.525895 | 0 | 0.003691 |
| 23 | 0.003693 | 0.468298 | 0 | 0.527921 | 0 | 0.003693 |
| 24 | 0.0037 | 0.462483 | 0 | 0.533801 | 0 | 0.0037 |
| 25 | 0.003726 | 0.444569 | 0 | 0.551702 | 0.06487 | 0.44458 | 0.07137 | 0.41918 |
| 26 | 0.003815 | 0.393142 | 0 | 0.603042 | 0 | 0.003815 |
| 27 | 0.00409 | 0.2737 | 0 | 0.72221 | 0 | 0.00409 |
| 28 | 0.004606 | 0.118975 | 0 | 0.87642 | 0 | 0.004606 |
| 29 | 0.00555 | 0.033322 | 0 | 0.961128 | 0 | 0.00555 |
| 30 | 0.008837 | 0.007302 | 0 | 0.983861 | 0.10083 | 0.20889 | 0.02107 | 0.66921 |

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Fig. 1. a) Ternary plot (Conceptual design) of the reactive distillation for the methyl acetate/methanol/acetic acid and b) water/acetic acid/methyl acetate.
Fig. 2. Temperature profile with respect to stage number.

Fig. 3. Composition profiles with respect to stage number.
model is suggested for the esterification process. The liquid composition of multi-component mixture by simulation and experimental data is shown in Table 4.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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