

COMPARISON OF MASS-TRANSFER EFFICIENCIES OF SCC AND STRUCTURED PACKING

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Abstract In a previous paper, pressure drop, flooding and mass-transfer characteristics of a novel pilot-scale distillation column called spinning cone column (SCC) were presented. Here, we present the result of comparison of mass-transfer efficiencies of SCC and structured packing. Comparison of SCC and structured packing mass-transfer characteristics show that the gas and liquid-side height of transfer units (HTUs) of the SCC are on the average 20% and 50% lower than the corresponding values for structured packing respectively. This in turn results in lower HETPs of the SCC for practical applications. Predicted HETPs in ethanol distillation using SCC are 30% lower than those of structured packing. Predicted HETPs for fusel oil and orange oil distillations are also 45% and 35% lower respectively than the experimental values obtained for structured packing. From these results, it is concluded that SCCs are more efficient with respect to mass-transfer than this particular structured packing, but to draw a general conclusion, more specific data on pressure drop per NTS, and liquid hold-up and residence time of the two columns should be available.

Key Words Spinning Cone Column, Mass-Transfer Efficiency, Pressure Drop, Flooding, Structured Packing, Food Flavor, Distillation

چکیده در مقاله قبلی، ویژگیهای افت فشار، طغیان و انتقال جرم یک دستگاه تقطیر جدید به نام ستون مخروطی چرخان (اس سی سی) در مقیاس پایلوت ارائه شد. در این مقاله نتایج مقایسه بازدهی انتقال جرم این برجها با برجهای پر شده با آکنه های منظم ارائه می شود. مقایسه ارتفاع مراحل انتقال در دو برج نشاندهنده این است که بطور متوسط ارتفاع مراحل انتقال در برجهای ستون مخروطی چرخان و در فاز گاز و مایع به ترتیب 20% و 50% کمتر از مقادیر مشابه برای برجهای پر شده منظم است؛ که این امر به نوبه خود باعث کاهش ارتفاع معادل با یک سینی تعادلی (اچ ای تی پی) در برجهای ستون مخروطی چرخان می شود. مقادیر اچ ای تی پی بدست آمده برای تقطیر آب/ اتانول توسط اس سی سی، 30% کمتر از برجهای پر شده منظم است. مقادیر اچ ای تی پی بدست آمده توسط برج اس سی سی برای سیستمهای روغن پرتقال و روغن فیوزل به ترتیب 35% و 45% کمتر از مقادیر بدست آمده آزمایشگاهی توسط برج پر شده با آکنه های منظم است. از این مقایسه چنین نتیجه گیری می شود که برجهای اس سی سی از نقطه نظر بازدهی انتقال جرم کاراتر از برجهای پر شده با آکنه منظم است؛ اما برای یک نتیجه گیری کامل در این زمینه، اطلاعات بیشتری در باره افت فشار و ماندگی مایع در دو برج لازم است.

INTRODUCTION

Figure 1 shows a schematic diagram of the SCC. The spinning cone column (SCC) is a gas-liquid

contacting device consisting of a vertical countercurrent flow system, which contains a succession of alternate rotating and stationary metal cones, whose upper surfaces are wetted with a thin

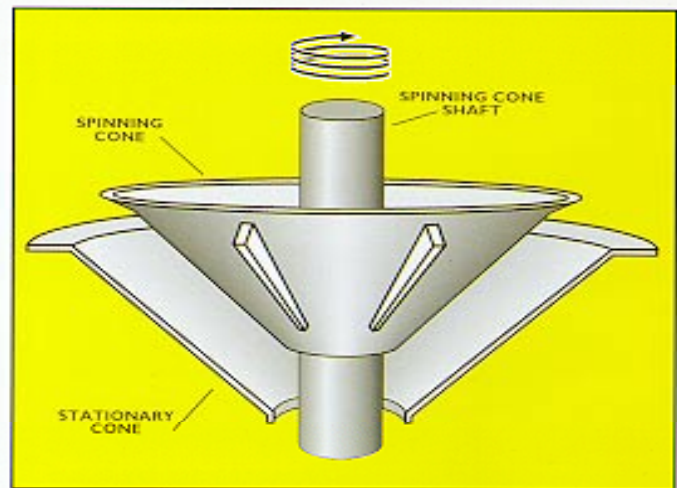
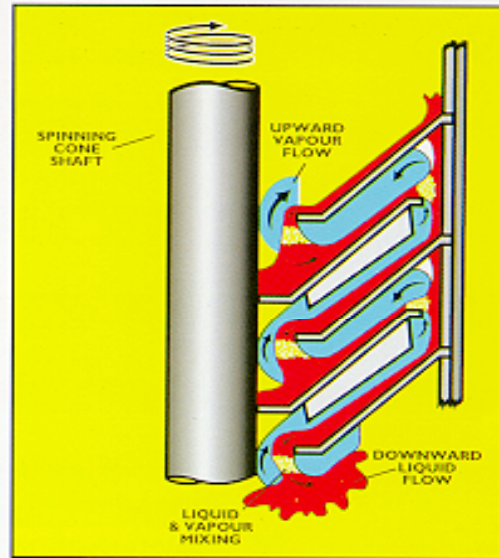
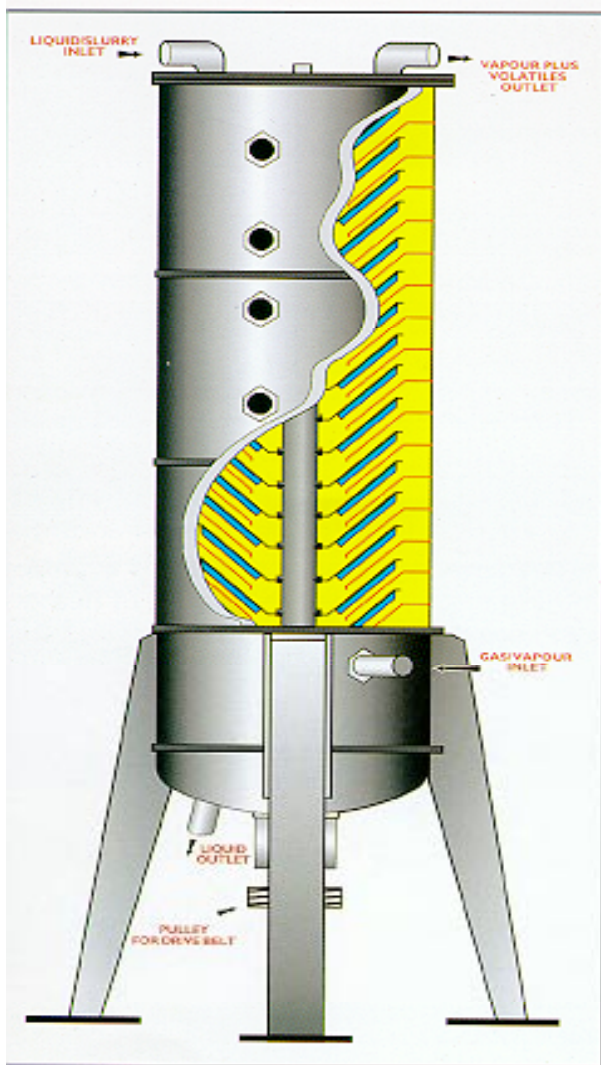


Figure 1. Schematic diagram of the SCC (Figure provided by Flavourtech).

film of liquid. Liquid flows down the upper surfaces of the stationary cones under the influence of gravity and moves up the upper surfaces of the rotating cones in a thin film by the action of the applied centrifugal force. Vapour flows up the column, traversing the successive fixed and rotating cones.

Pressure drop, flooding and mass-transfer characteristics of a pilot-scale SCC have been presented in the previous paper by Zivdar et al. [1]. Flooding data were compared with the data taken from a laboratory-sized SCC and an

industrial-scale SCC. It was shown that a single packing factor (see appendix A) can bring flooding data of these SCCs into a single curve, where plotted in a Sherwood-Leva-Eckert (SLE) diagram. The packing factor was not obtained experimentally but calculated from the definition of packing factor, which is the ratio of the wetted area by the volume. Ethanol/water and acetic acid/ethanol/water were used as a test mixture in measuring the mass-transfer efficiency of the column, expressed as Murphree vapour efficiency, E_{MV} , (see appendix B for sample calculation). The range of efficiencies

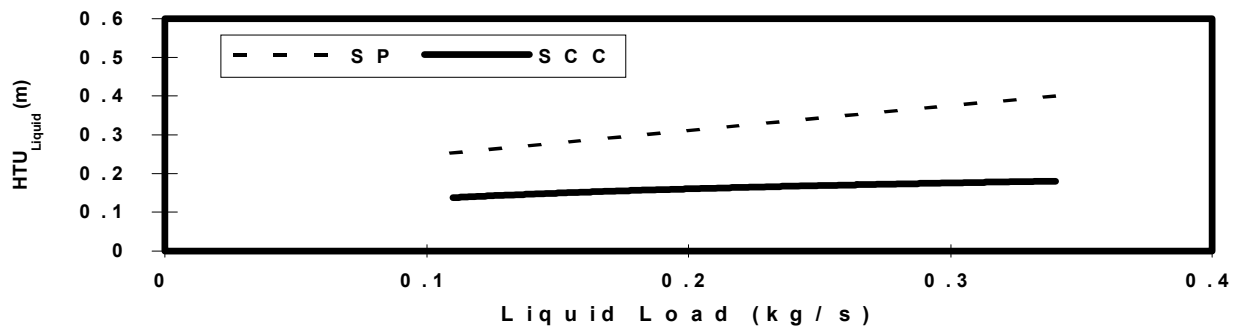


Figure 2. Comparison of liquid-side HTUs of the two columns.

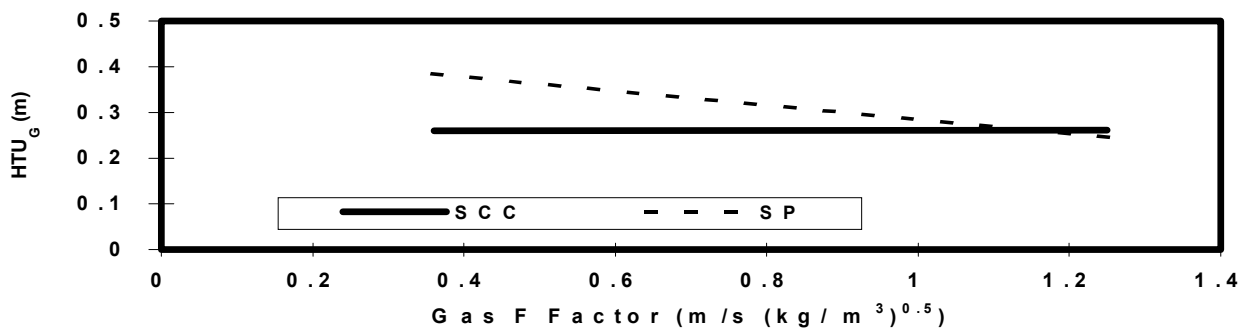


Figure 3. Comparison of gas-side HTUs of the two columns.

obtained was from 5% to 20% and 3% to 8% respectively for the ethanol/water and acetic acid/ethanol/water test mixtures. These efficiencies were lower than expected, which were attributed to the construction and maintenance of this particular SCC. VLE data for the calculations of efficiency were selected by extensive analysis of the data reported in the literature by thermodynamic consistency tests [11]. (See appendix C for details). Factors influencing the performance of the SCC were also discussed.

RESULTS AND DISCUSSION

Gas and Liquid-Side Mass-Transfer Coefficients

Comparison has been made in order to relate mass-transfer characteristics of structured packing to the predictions from the SCC. The individual transfer coefficients of the SCC were taken from Desho

et al. [2] and Prince et al. [3], where experimental gas and liquid-side transfer coefficients of the SCC were measured by humidification of air and desorption of oxygen respectively. It should be pointed out that the SCC data were taken at RPM=550, the most common value used in practice.

Figures 2 and 3 illustrate the comparison of individual liquid and gas-side HTUs of the two columns. The liquid and gas-side HTUs of the SCC are on the average 50% and 20% lower than those of the structured packing. The possible reason for higher mass-transfer coefficients in the SCC could be related to the turbulence and presence of waves on the liquid surfaces in the SCC. Prince et al. [3] have shown quantitatively that the enhancement of mass-transfer due to the presence of waves on the liquid surfaces is significant.

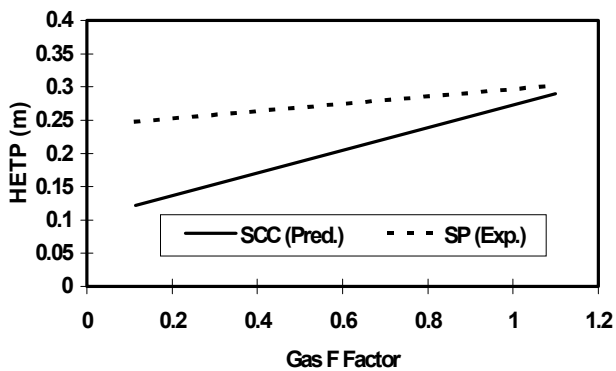


Figure 4. Comparison of HETPs of structured packing and SCC in ethanol/water distillation.

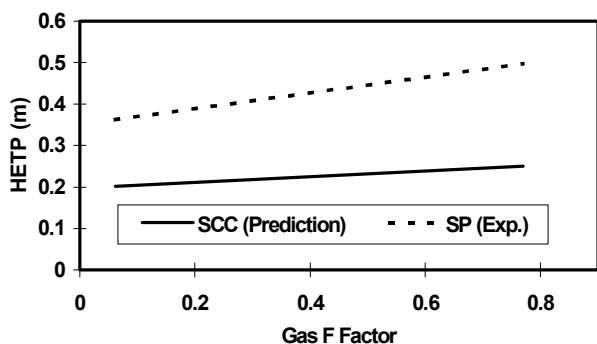


Figure 5. Comparison of HETPs in SP and SCC for distillation of fusel oil.

The comparison of the gas-side HTUs of the two columns shows different trends. The structured packing trend as explained by Zivdar et al. [4] is related to the gauze-type nature of the structured packing, which shows an increase in interfacial area with an increase in gas load. The comparison shows that gas-side HTUs of the SCC are on the average 20% lower than those of structured packing, however, both columns show comparable gas-side HTUs at F factor of about 1.2. This is related to the increase in the mass-transfer area of the structured packing at high gas loads.

Ethanol Distillation Experimental HETPs from ethanol/water distillation, (Zivdar [5]), using structured packing were compared to the predictions using the SCC's individual phase

mass-transfer data. Figure 4 illustrates the HETP as a function of the gas F factor for ethanol/water distillation. The HETPs of the SCC are on the average 30% lower than those of structured packing. This is expected, because individual gas and liquid-side transfer coefficients of the SCC were lower than those of the structured packing, however, the comparison again shows comparable HETPs at a gas F factor of about 1.1.

Fusel Oil Distillation Fusel oil is a by-product of the distillation step in ethanol manufacturing. It is mainly composed of higher alcohols. It is an undesirable fraction, and is removed from the products, but one of the components of fusel oil, 2-methyl-butanol, is a valuable product. The process of separating 2-methyl-butanol from fusel oil is carried out in two steps. The first step is to separate 2-methyl-butanol, 3-methyl-butanol and other heavier components from lighter components. Then, 2-methyl-butanol is separated from 3-methyl-butanol. The latter separation is difficult because these two components have a similar chemical structure and physical properties.

Figure 5 illustrates the variation of the HETP against the gas F factor for the two columns in fusel oil distillation. The predicted HETPs of the SCC are on average 45% lower than those of structured packing. This means that for the same separation, the required column height for the SCC is nearly half that required by structured packing. This makes the separation of the 2, 3-methyl-butanol more feasible practically.

Orange Oil Distillation Orange oil is obtained from the pressed peels of the fruit. Typically it contains around 96 wt% terpene hydrocarbons (mainly d-limonene), around 3% oxygenates (aldehydes, alcohols and esters), and about 1% non-volatile high boiling components, (Maarse [6]). Terpene hydrocarbons have little odour value and are susceptible to oxidation, giving rise to unpleasant off notes. The oxygenates, particularly the aldehydes, are the main flavour components.

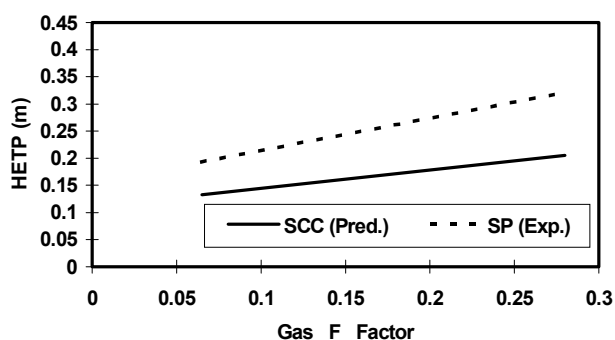


Figure 6. Comparison of HETPs of structured packing and SCC in orange oil distillation.

In order to reduce the problem of oxidation and produce more stable oil during storage, some or all of the terpenes should be removed. This is usually done by vacuum distillation under conditions leading to some loss of desirable material, particularly octanal and lighter components. We therefore investigated whether the high separation capability of structured packing under vacuum (high number of stages per unit pressure drop) would allow a sharper separation of the flavour compounds, octanal, from the terpenes, limonene.

Figure 6 illustrates the variation of the HETP against the gas F factor for the two columns in orange oil distillation. The HETPs of the SCC are on average 35% lower than those for structured packing. It should be pointed out that, for orange oil distillation, both the gas and liquid-side contribute nearly equal resistance to mass-transfer, while in the ethanol and fusel oil distillations, most of the resistance comes from the gas-side.

CONCLUSIONS

Pressure drop, flooding and mass transfer characteristics of spinning cone column have been studied, (Zivdar et al. [1]). From the results obtained it was concluded that Sherwood-type diagrams for representing flooding data are a reliable tool for predicting the capacity of the SCC, and these diagrams can then be used for the

design of new columns with respect to capacity, using an appropriate packing factor. The distillation (Murphree) efficiencies were lower than expected, due to construction and maintenance features of this particular column. However the trends with changing variables fitted our knowledge of these columns; efficiency increased with rotational speed, and decreased with increasing entrainment due to higher vapour rates.

The comparison of SCC and structured packing mass-transfer characteristics revealed that the gas and liquid-side HTUs of the SCC are on the average 20% and 50% lower respectively than the corresponding values for the structured packing. This in turn results in lower HETPs of the SCC for practical applications. Predicted HETPs in ethanol distillation using SCC are 30% lower than those for structured packing. Predicted HETPs for fusel oil and orange oil distillations are 45% and 35% lower respectively than experimental values obtained for structured packing.

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NOMENCLATURE

E_{MV}	Murphree vapour efficiency.
F	Gas F factor, $=U_G(\rho_G)^{0.5}$, $\text{m/s}(\text{kg/m}^3)^{0.5}$.
HETP	Height Equivalent to Theoretical Plate, m.
HTU	Height of Transfer Unit, m.
NTS	Number of theoretical stages.

- RPM Revolution per minute.
 y_{n*} Actual vapour mole fraction leaving stage n.
 y_n Vapour mole fraction in equilibrium with the liquid on plate n.

Subscripts

- L Liquid
 G Gas
 SCC Spinning Cone Column.
 SP Structured Packing.

APPENDIX A: MINIMUM FLOW AREA AND PACKING FACTOR CALCULATIONS

A schematic diagram of a rotating and stationary cone is shown in Figure A.

$$A_{\min} = \pi t (R_o + R_i)$$

$$A_{\min} = 3.14 \times 0.01414 (0.05 + 0.06) = 0.004886 \text{ m}^2$$

$$\text{Packing Factor} = \frac{\text{Wetted surface area}}{\text{Total volume}}$$

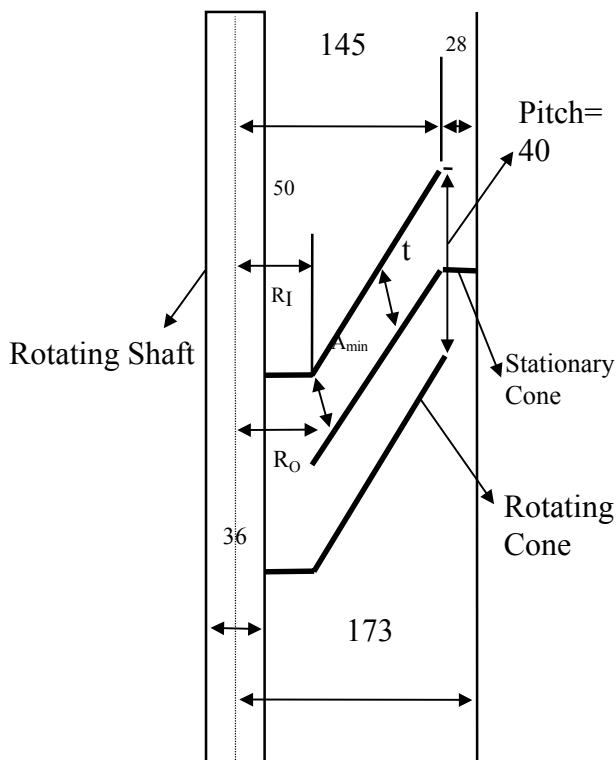


Figure A: Schematic diagram of the spinning cone column. All units in mm.

Area of a stationary cone =

$$\frac{\pi(0.145^2 - 0.05^2)}{\sin 45} + \pi(0.173^2 - 0.145^2) = 0.117 \text{ m}^2$$

Area of a rotating cone =

$$\pi(0.05^2 - 0.036^2) + \frac{\pi(0.145^2 - 0.05^2)}{\sin 45} = 0.095 \text{ m}^2$$

$$\text{Total surface area} = 30 \times (0.117 + 0.095) = 6.36 \text{ m}^2$$

$$\text{Packing factor} = 6.36 / (1.2 \times \pi \times 0.173^2) = 56.4 \text{ m}^{-1}$$

Note that the number of cones is 30 and the column height is 1.2 m.

After dismantling the column the pitch was measured accurately and based on this value new minimum flow area was calculated.

$$A_{\min, \text{new}} = 3.14 \times 0.0849 (0.05 + 0.056) = 0.00283 \text{ m}^2$$

$$\text{Percent reduction in flow area} = (1 - 0.00283 / 0.004886) \times 100 = 42\%$$

Please note that the pitch for the second case was measured 24mm.

APPENDIX B: MURPHREE VAPOUR EFFICIENCY CALCULATION

McCabe-Thiele method was used for binary steam-stripping calculations. A computer program was written which takes into account the effect of heat loss on the vapour and liquid flow rates at each stage in the column, and applies a Murphree vapour efficiency so that the output of the calculation matches the experimental data.

Murphree vapour efficiency is defined as:

$$E_{MV} = (y_n - y_{n-1}) / (y_{n-1}^* - y_{n-1}) \quad (\text{B.1})$$

It is a measure of deviation from ideal equilibrium behaviour of vapour streams

leaving each tray, e.g., a Murphree vapour efficiency of one means that the vapour leaving the tray is in equilibrium with the liquid leaving the same tray.

Heat loss from the column has a significant effect on the slope of the operating line at each stage. As a result of heat loss from the column, the vapour rate is going to decrease as the vapour flows from the bottom to the top of the column. In addition, the liquid is going to increase down the column due to the addition of condensed vapour. If we define "Internal Reflux", Z , to be the total moles of vapour condensing on the inside wall of the column with 30 actual stages, we can write

$$V_n = V_{top} + [(30-n)/30] Z \quad (B.2)$$

$$L_n = L_{top} + [(30-n)/30] Z \quad (B.3)$$

where:

V_n = the vapour molar flow rate at stage n ($n = 1$ is bottom stage),

L_n = the liquid molar flow rate at stage n ,

V_{top} = vapour flow rate at the top of the column (equivalent to distillate molar flow rate),

L_{top} = liquid flow rate at the top of the column (equivalent to feed molar flow rate).

From McCabe-Thiele analysis, the operating line is given by

$$x_{n+1} = x_n + V_n/L_n (y_n - y_{n-1}) \quad (B.4)$$

Substitution from Equations B.1, B.2 and B.3 results in

$$x_{n+1} = x_n + \frac{D + \frac{30-n}{n} Z}{F + \frac{30-n}{n} Z} [E_{MV}(y_n^* - y_{n-1})] \quad (B.5)$$

where:

D = molar flow rate of distillate,

F = molar flow rate of the feed.

We can then calculate the liquid composition on each stage by calculating each stage in sequence starting at the bottom of the column, with the following starting values:

$$n = 1 ; y_{n-1} = y_0 = 0$$

$x_n = x_B$ = measured bottom mole fraction of ethanol.

By using the above equations, the Murphree vapour efficiency was altered until the calculated distillate compositions matched those that were measured experimentally.

Multicomponent tray by tray calculations, starting from the bottom, were used for ternary mixture calculations. In this case Murphree vapour efficiency for the two components have to be specified and altered until the distillate compositions match those obtained experimentally.

APPENDIX C: THERMODYNAMIC CONSISTENCY TESTS

More than 250 references on experimental vapour-liquid equilibrium data were collected. Particular attention was made in order to find the data on systems formed by ethanol, acetic acid and water. The best set of experimental VLE data in each case was chosen. Two thermodynamic consistency tests have been considered in order to choose the best set of VLE data, first the point test of VanNess [7], and second the integral test of Redlich -Kister, [8]. The criteria for the first test is $\Delta Y \leq 0.01$. This means that data with an average absolute deviation of less than or equal to 0.01 are assumed to be consistent. The criteria for the second test is $[D-J \leq 10\%]$. In which

$$D = \frac{\langle A' - B' \rangle}{A' + B'} \times 100 \quad (C.1)$$

$$J = \frac{\langle \Delta T_{MAX} \rangle}{T_{MIN}} \times 150 \quad (C.2)$$

A' and B' are the area above and below the x-axis, where plot of $\ln \gamma_1/\gamma_2$ vs. X has been sketched. This test is exclusive to isobaric VLE data.

For the ethanol/ water system the data published by Stabnikov [9] is recommended. This data includes data for very dilute ethanol concentration.

For acetic acid/ water binary mixture the data published by Chalov [10] or the use of recommended parameters published in DECHEMA [11] is recommended.

For the system acetic acid/ ethanol, the data published by Amezagas [12] is recommended.

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