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Effectiveness of Contact Devices on Ethanol Separation Efficiency in a Packed Distillation Column

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Authors' contributions

This work was carried out in collaboration between both authors. Author AOA designed the study, performed the experiments and statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Author EAT initiated, supervised, proofread, managed the literature searches and wrote the conclusion of the study. Both authors read and approved the final manuscript.

Research Article

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ABSTRACT

Aims: To obtain the distillation column efficiency as a function of the packing materials used at various feed compositions of binaries and a ternary system to validate the most effective packing arrangement.
Study Design: The design was based on the concentrations obtained from the fermentative production of ethanol.
Place and Duration of Study: Department of Chemical Engineering, Obafemi Awolowo University, Ile-Ife, Nigeria, between December 2009 and August 2010.
Methodology: Distillation experiments were carried out with three binary systems (ethanol-water, ethanol-ethylene glycol and water-ethylene glycol) and a ternary system (ethanol-water-ethylene glycol) in a 0.1m internal diameter glass column which was first packed with 8mm diameter Raschig rings (A) thereafter, same size of wire gauze rings (B) and later their combination (C). The experiments were performed under total reflux conditions and at atmospheric pressure.
Results: Out of the three packing arrangements investigated, Packing-B showed the best performances with the number of transfer units from 2.8818 to 1.9556 and 4.0723 to

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3.0845 corresponding to ethanol-water and ethanol-ethylene glycol binary systems respectively, in the direction of increasing most volatile component (*mvc*) in the charged compositions. Whereas, for water-ethylene glycol system, opposite trend was obtained. Also, the best efficiencies (0.5652 to 0.9842) were exhibited by Packing-B. The results obtained for the ternary system revealed an inverse relationship between the *mvc* in the charged composition and the ethanol component efficiencies for all the packing used. This showed that the component efficiencies were higher for Packing-B (3.0157 to 1.5038) than Packing-A (2.7393 to 1.4595) and Packing-C (2.8116 to 1.4721). T-Test showed significantly different efficiencies for only ethanol-water pair-wise comparisons as well as for Packing- A and Packing-B in the ethanol-ethylene glycol system.

Conclusion: The designed wire gauze ring packing (Packing-B) has offered higher separation for all the systems examined than the conventional raschig ring. Thus, offering research basis for improving upon locally fabricated packing devices for bio-ethanol industry.

Keywords: Distillation; binary system; ternary system; efficiency; MVC; ethanol; packing.

1. INTRODUCTION

Ethanol and gasoline mixtures can be used as fuels reducing environmental contamination. Blending gasoline with anhydrous ethanol has been reported to improve octane index [1,2]. During the production of ethanol by fermentation, effluent typically has an ethanol concentration of approximately 10 percent by weight [3]. Recovery of ethanol from the fermentation broth involves distillation of the dilute aqueous alcohol to its azeotrope (95.57 percent ethanol by weight); distillation using a third component, either an organic solvent or a strong salt component to break up the azeotrope and remove the remaining water [4] and distillation to separate water from the third component so that it can be recycled [5].

Separations by distillation is regarded as the most widely used separation technique in the process industries [6], accounting for approximately 95 percent of the total separation energy used in the refining and chemical processing industries in the U.S. (Eldridge et al., U.S. Department of Energy, Unpublished result, 2005). Research studies continue on the process due to its versatility and energy intensiveness.

Separation efficiency has fundamental importance in the design and performance evaluation of distillation column. The performance varies markedly with a number of parameters, among which are effective interfacial area, components concentrations, system properties, column design and operating condition parameters [7]. Also, hydrodynamics of operation has its effect on the packed column performance. This is characterised by fluid flow rates, liquid hold-up and pressure drop across the packed section. The degree of separation in packed column using distillation operation is represented by the number of transfer units.

This paper focuses on packed column technology on the separation of aqueous ethanol as a renewable energy resource. It evaluates the effectiveness of a locally fabricated packing material, including its combination with other conventional packing for separation in a packed distillation column.

2. MATERIAL AND METHODS

Motivated by the efforts of the Renewable Energy Division of Nigeria National Petroleum Corporation (NNPC) to grow bio-fuel industry in Nigeria and the need to develop local technology for adoption in bio-ethanol refinery to purify ethanol-water mixture from fermenters to pure fuel grade ethanol, wire gauze ring packing was fabricated (Fig. 1). The average nominal size of the fabricated packing is 8 mm. The performance was determined alongside a borosilicate raschig ring (A) and their combination (C). A quickfit visible flow packed distillation column (Corning Process Plant Engineering, Staffordshire, England) was employed for determination of column efficiency. The unit occupies a ground area of approximately 1.7 m^2 with overall height of 5.70 m and inside column diameter of 0.1 m. The height of packed section is 1.3 m. The reboiler system consisted of a boiler type heat exchanger fitted externally to a spherical vessel of nominal capacity of about 20 litres in a thermo-siphon loop. The region above the packed height but below the reflux divider and the region below the packed height but above the reboiler were chosen for accurate temperature measurement and sample collection. The raschig ring was supplied by ETA Ltd., Stafforshire, England, while the wire gauze ring packing was fabricated at the Department of Chemical Engineering, Obafemi Awolowo University, Ile-Ife. The feed composition range experimented were selected based on the concentration ranges of the product of fermentative production of ethanol which normally fall between 6 and 12 percent by weight. Fig. 2 is a diagrammatic representation of the experimental set up. The experiment was repeated three times.

The analysis of the vapour and liquid samples collected from the column for their compositions was carried out using a combination of the samples' refractive index (RI) and density. RI was determined using digital refractometer (RX-5000α, Atago, Japan) while density measurement was done using the density bottle. The data obtained was subjected to t-test statistical analysis.

The binary distillation efficiency for the systems was determined by solving Equation (1) using experimental data and Simpson's integration.

$$\int_{x_b}^{x_t} \frac{dx_1}{x^*_1 - x_1} = \frac{K_{x_1} aSZ}{L_m} = NTU_{x_1} \qquad (1)$$

For a total reflux condition and for a condition where the concentration change of the three components through the column is small as well as the situation where the driving forces do not change sign along the column, Qureshi and Smith [8] proposes the number of transfer units for a ternary mixture as:

$$NTU_{ox_i} = \frac{x_{i_T} - x_{i_B}}{x_{i_{LM}}} \qquad (2)$$

where

$$x_{i_{LM}} = \frac{\{(x_{iT}^* - x_{iT}) - (x_{iB}^* - x_{iB})\}}{ln\{(x_{iT}^* - x_{iT})/(x_{iB}^* - x_{iB})\}} \qquad (3)$$

where NTU_{ox} is the overall number of transfer units based on liquid phase, S is the cross sectional area of the column, Z is the height of the packed section, L_m is the liquid flow rate, K_x is the liquid phase mass transfer coefficient, a is the effective interfacial area, x_i and x_i* represent the liquid phase composition and the composition in equilibrium with the vapor in gas phase of component i, respectively, $x_{i,T}$ and $x_{i,B}$ represent liquid top and bottom compositions respectively.

3. RESULTS AND DISCUSSION

3.1 Binary System

The distillation column performance variation for the various packing experimented is presented in Figs. 3 to 6 with most volatile component (*mvc)* concentration in the feed mixtures for each of the three binary systems as a factor. The overall averaged distillation column performance was found to decrease monotonously with increase in the more volatile component in the feed for ethanol-water and ethanol-ethylene glycol systems (Figs. 3 and 4). The trend of the result is similar for the three packings (A, B and C) tested. This had been previously observed and reported by other workers using similar systems [9,10,11]. Generally, packing B showed the best performance for the three binaries and at various feed concentration. The performance follows the trend B>C>A, with packing B having between 15 and 45 percent enhanced performance over packing C. This trend reflects an increasing performance with increasing voidage. This enhancement is a result of improved vapour-liquid mixing characteristics which promotes high mass transfer. But, the result trend for packings A, B and C in Fig. 5 show an increasing performance with increase in the *mvc* of the feed for water-ethylene glycol system. This trend could be due to increasing vapour flow rate emanating from increased column temperature, hence, enhanced fluid turbulence in the column.

The feed composition effect on performance variation is attributable to response to system surface tension variation with *mvc*. This has tendency to significantly influence the wetting characteristics of the liquid phase. The reduction in surface tension of binary systems caused reduction in the liquid spread over packing surface, hence, drop in mass transfer rate. It is worth noting that the interfacial hydrodynamic effects subject to surface tension, depends on vapour flow rate, which in turn, influence system's turbulence and intimate mixing and hence mass transfer rate. Thus, when the more volatile component has the lower surface tension (positive system e.g. ethanol-water and ethanol-ethylene glycol systems) the froth is more substantial and more stable than when the more volatile component has the higher surface tension (negative system e.g. water-ethylene glycol system). It was observed that as the component with lower surface tension decreases in the binary feed composition, improved surface tension gradient of the liquid phase across the bed results. (The surface tension values at 20ºC for water is 72.86 mN/m, ethanol is 22.1 mN/m and 47.7 mN/m for ethylene glycol [12]). The liquid spread more readily over the packing surface and provides more interfacial area of contact (hence greater efficiency) for positive systems than for a negative system. This must have contributed to the observed trends in Figs. 3 to 5, which corroborates the report of Patberg et al. [13].

3.2 Ternary System

The ternary distillation efficiency for ethanol-water-ethylene glycol system was determined according to the approach of Qureshi and Smith [8]. The results obtained for comparison between the packing arrangements and *mvc in the charge* composition showed that the overall column efficiency is higher for Packing-B than others (Packing-A and -C). This is attributed to their corresponding pressure drop values obtained from their operations (as shown in Table 1). Fig. 6 depicts a decrease in the most volatile component (*mvc*) efficiency for all the packing arrangements with corresponding increase in the *mvc* composition in the feed. Equally, the plot shows an increase in the component efficiency of the *mvc* with a corresponding increase in the intermediate component concentration in the charge (Fig. 6). Further examination showed that at feed composition of x_{ET}=0.1024 and x_{WA}=0.7669, the component efficiency of ethanol for Packing-B arrangement recorded the highest value of 3.0157 which is 10 and 7 percents greater than both Packing-A and Packing C arrangement respectively. Likewise, when the ethanol concentration was doubled and that of water reduced by 18 percent, Packing-B recorded 12 percent decrease in component efficiency of ethanol. Furthermore, when the latter, most volatile component (*mvc*) concentration in the charged mixture was doubled and that of intermediate volatile component (*ivc*) was reduced by 100 percent, all the packing arrangement recorded more than 80 percent reduction in the ethanol component efficiency. This trend reflects a situation where an increase in the concentration of the intermediate volatile component (*ivc*) in the distilling ternary mixture increases the separability of the *mvc* in the mixture. Consequently, when *ivc* component increases in the charge, a much larger surface tension is presented for intermolecular interaction than that available when intermediate concentration reduces. These observations are in-line with the recorded values of the surface tension of interaction for ethanol and water, explained in Prausnitz et al. [14]. The value of the surface tension of interaction recorded for ethanol is less than that recorded for water. In addition to the above, the contribution of column temperature with *mvc* composition in the feed could be responsible for the obtained trend in the separation efficiency of *mvc*. According to Fig. 7, it can be observed that as the *mvc* in the charged increases there is a decrease in the average column temperature. Juxtaposing Fig. 7 with Fig. 6, decrease in average column temperature brings about a reduction in column performances for the packing arrangements investigated. This gave credence to the earlier assertion of Fasesan et al. [11] and Taiwo and Faesan [15] that decrease in average column temperature resulted into reduction in the average kinetic energy of the molecules which impedes the vapour flow rate and subsequently the liquid flow rate. Hence, an increase in the separation efficiency of the mvc with corresponding increases in the charge concentration of the ivc. Thus, it leads to decrease in turbulence that would have enhanced intimate contact. Hence, it resulted into decrease in column performances.

In this study, the azeotropic point of 95.57 percent by weight of ethanol was not reached by any of the packings (i.e. A, B and C) at various ethanol-water feed compositions tested. But with the ternary system, all the packing-types surpassed the azeotropic point with packing A having the highest top vapour composition of 98.73 percent ethanol from a feed composition (x_{Et} = 0.2 and x_{Wa} = 0.6477). Also, when a comparison of the top vapour composition of ethanol-water-ethylene glycol mixture ($x_{f,Et}$ = 0.1024, $x_{f,Wa}$ = 0.7669) is made with that from ethanol-water system ($x_{f,Et}$ = 0.1601) in order to identify whether changes in composition could be observed that may be associated with specific selective ethanol-ethylene glycol and ethylene glycol-water interactions, the result shows that the vapour composition from ethanol-water mixture is influenced by the addition of the ethylene glycol. This significantly shows water-ethylene glycol interaction in the systems that are stronger than those between ethanol and water.

3.3 T-Test Statistical Analysis Report on Column Performance

In order to compare the efficiencies of the contacting devices under each system, a paired sample t-test was carried out. The paired sample t-test is basically used to compare the means of outcomes in before/after experiments or to compare the means of similar observations on paired groups. The values of the test statistic (t) as well as the probabilistic significance (ρ) are reported in Table 2. The t-test was carried out at 95% level of confidence. For the ethanol-water system, all the pairwise comparisons revealed significantly different efficiencies (P = .05). The negative sign of the test statistic for A-B and A-C shows that B and C consistently have higher efficiency values than A. The positive t-value for B-C shows that B performed better than C. As a result, for the ethanol-water system the best performance was exhibited by packing B. This corroborates the trend reported in Fig. 3.

For ethanol-ethylene glycol binary system, the pairwise comparison of Packing A-B was significant and showed the dominance of Packing B arrangement over Packing-A. All other paired tests revealed insignificant differences in the obtained component efficiencies.

4. CONCLUSION

A new ring packing of wire gauze have been locally fabricated, experimented and compared with conventional borosilicate raschig ring packing. The experiments are conducted with both binary and ternary mixtures.

The designed wire gauze ring packing offered higher separation efficiencies for all the binary systems than the conventional raschig ring. But, its performances showed close proximity with the conventional one in the case of the ternary system.

Although the packing could not achieve separation pass the azeotropic point of aqueous ethanol, it has significantly provided better contacting device suppressing the azeotropic phenomenon with introduction of minimal amount of ethylene glycol as entrainer. Thus, offering research basis for improving upon locally fabricated packing devices for bio-ethanol industry.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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APPENDIX

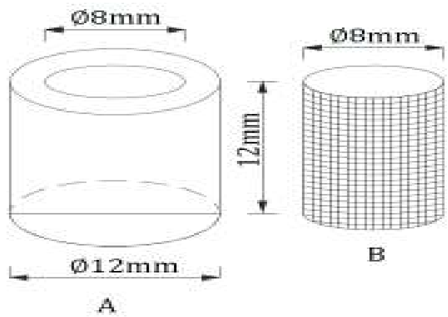

Fig. 1. Packing dimensions

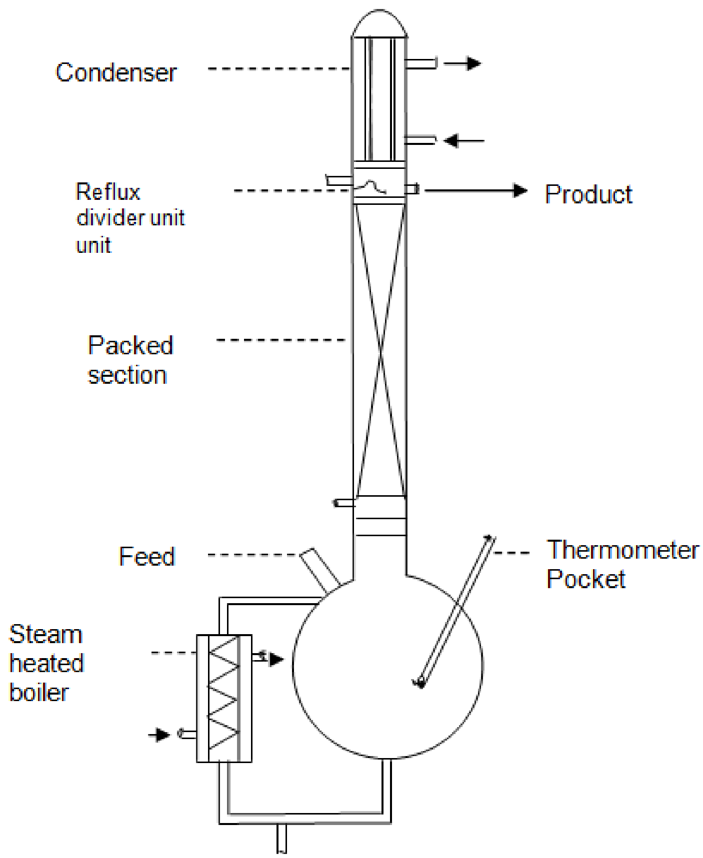

Fig. 2. Packed distillation column

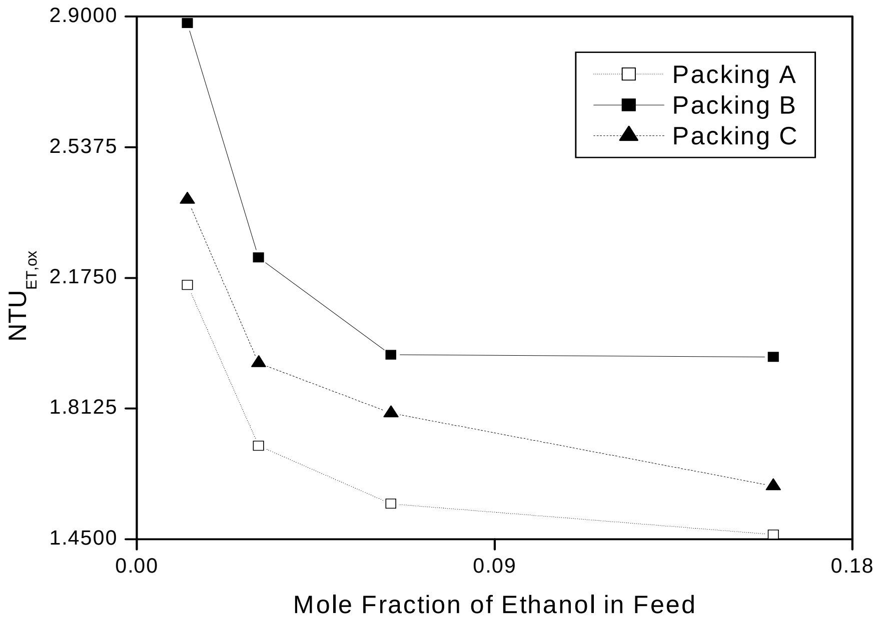

Fig. 3. Variation of Binary Efficiency with mole fraction of *mvc* in the Feed (Ethanol-Water system)

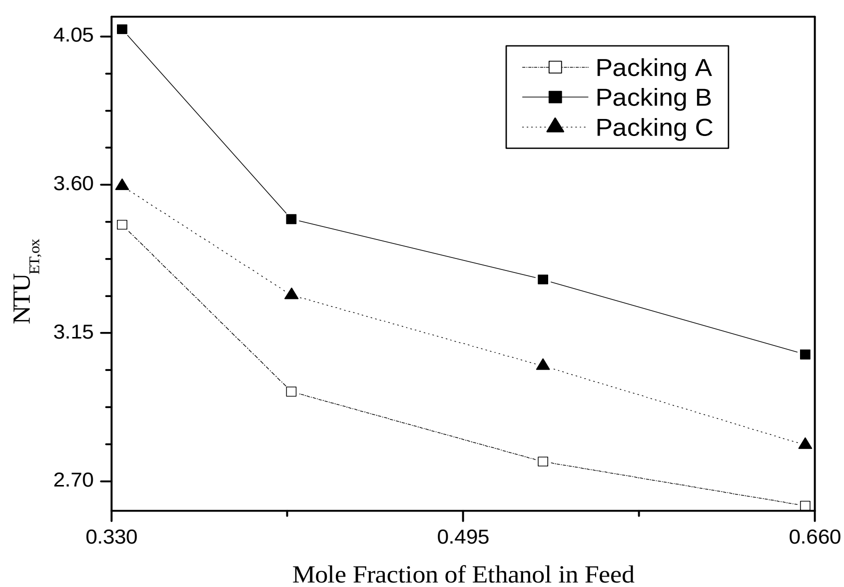

Fig. 4. Variation of binary efficiency with mole fraction of *mvc* in the feed (ethanol-ethylene glycol system)

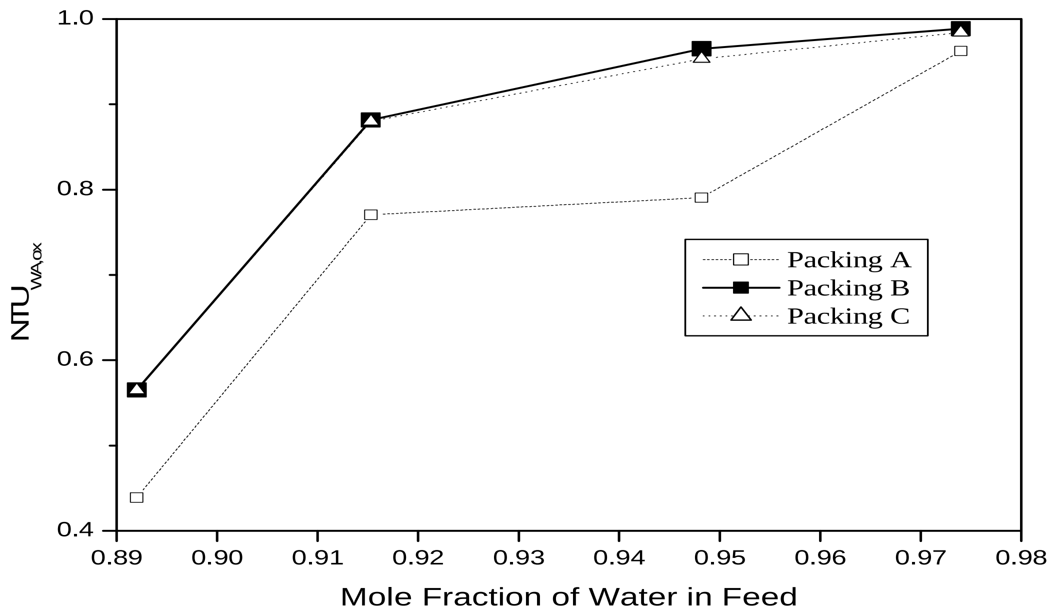

Fig. 5. Variation of binary efficiency with mole fraction of *MVC* in the feed (water-ethylene glycol system)

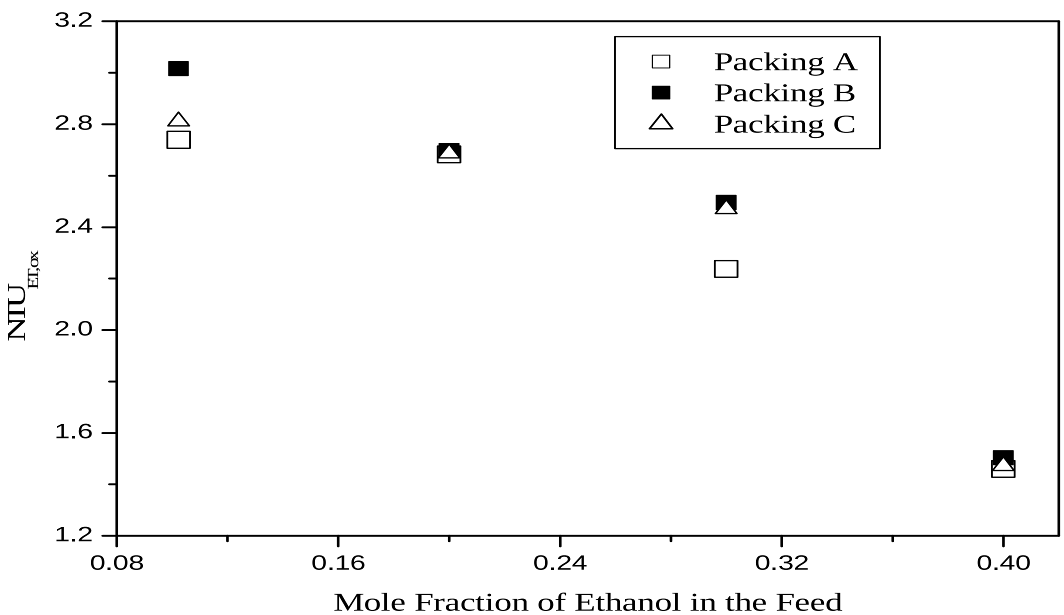

Fig. 6. Variation of *MVC* in the feed with ethanol component efficiency for ethanol-water-ethylene glycol system

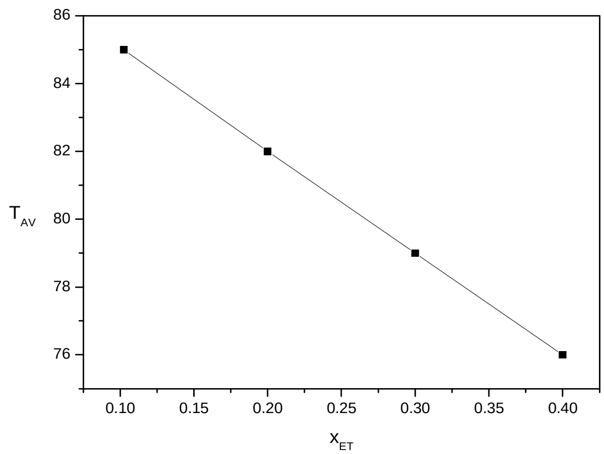

Fig. 7. Variation of average column temperature with the ethanol concentration in the ternary system feed

Table 1. Properties of packing bed

Type	Void fraction (%)	Pressure drop (cm. H_2O)
Raschig ring (a)	70.5	1.10-8.50
Wire gauze (b)	93.1	0.25-1.90
C= ½(a+b)	81.8	0.20-6.80

Table 2. T-test result

S/N	System	Pair-wise comparison	t	*P*
1	Ethanol-water	A-B	-8.076	0.004*
		A-C	-8.034	0.004*
		B-C	4.762	0.018*
2	Ethanol-ethylene glycol	A-B	-3.758	0.033*
		A-C	-0.916	0.427
		B-C	2.767	0.070
3	Water- ethylene glycol	A-B	-1.944	0.147
		A-C	-0.711	0.528
		B-C	0.851	0.458
4	Ternary system	A-B	-1.940	0.148
		A-C	-1.366	0.265
		B-C	1.428	0.249

**It is significant at P = .05*

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