Suitability of Using Brass Ferrules as Packing in a Glass Distillation Column and Presenting a New Relationship between HETP and Pressure Drop

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ABSTRACT: *Brass Ferrules have been used as packing in a laboratory- scale glass distillation column approaching pilot plant size because of their availability in all sizes and low cost. The available size range is such that these ferrules could also be used in industry and the results of this study could be scaled up. The performance of the new packing has been compared with the performance of glass packing, and has proven to be more satisfactory with lower HETPs (Height Equivalent to a Theoretical Plate). Three systems were studied in this work, namely, ethanol/water, 1-propanol/water, and 1-butanol/water and the HETP of the brass packing in these systems was experimentally measured. Pressure drop and flooding have been studied together in this work and a new relationship has been presented that relates efficiency in terms of HETP to pressure drop, a parameter that can be measured readily in any column or predicted.*

KEY WORDS: *Packed columns, Packing, HETP, Efficiency, Pressure drop.*

INTRODUCTION

Distillation is the oldest and most widely used separation method that dates back to ancient Egypt [1]. However, distillation in packed columns is relatively new and the first patent in this regard belongs to the German scientist Dr. *Raschig* in 1907 [2]. The advantages of packed columns over tray columns have attracted more attention to them. These advantages include: lower pressure drop, higher efficiency, higher corrosion resistance of plastic and ceramic packings, better resistance to clogging, and less foaming.

Metal, ceramic and plastic are the three common materials of construction and their selection depends mostly on their corrosion and heat resistance. Metal packings are mostly made of cast iron and are the first choice in non-corrosive service. They are cheaper than ceramic and plastic packings and have higher capacity and efficiency, a larger range of geometries, more free space for liquid flow; they do not break easily and stand high pressures. Plastic packings are relatively cheap, weigh very little and can be easily sucked into or blown out of the column, but soften easily at relatively low temperatures, are not resistant to solvents, are hydrophobic, decompose under ultraviolet light, and react with oxygen, are fragile at low temperatures, and have a

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high foaming tendency when their additives are dissolved, the heat of dissolution of which could cause hot spots. Ceramic packings are fragile, have low capacity, and are not available in a wide range of geometries. Their advantage is being non-reactive and heat- resistant which makes them suitable for operations such as sulfuric acid absorption [3].

SYSTEMS STUDIED

Three alcohol-water systems, namely, ethanol/water, 1-propanol/water, and 1-butanol/water were used in this study because of the abundance of highly precise experimental equilibrium data on these systems that provides for better comparison between different packing materials.

The chemicals used were purchased from Merck with purity in excess of 99.9 %. The water used was distilled and de-ionized water produced in the laboratory with purity in excess of 99 %.

EXPERIMENTAL SETUP

The column used in this work is shown schematically in Fig. 1. All of the components used for determining HETP are shown: A 4-liter reboiler equipped with a 2.5 kW heater possessing three legs, one for liquid intake, one for vapor exit, and one used both for sampling and pressure measurement, a column with an inside diameter of 7.5 cm, a packed bed of ferrules with an equivalent diameter of 1 cm, a brass condenser with a heat transfer area of 1570 cm^2, a water manometer, and a pipe-type liquid distributor with a distribution point density of 226 points per square meter.

A typical brass ferrule used in this work is shown in Fig. 2.

EXPERIMENTAL PROCEDURE

Three liters of solution with a mole fraction between 0.10 and 0.15 is prepared and poured into the glass flask used as the reboiler vessel. Lower concentrations would cause low boil-up rates and could cause errors in experimental results in view of the sensitivity of HETP to vapor flow rate. The above concentrations fall in the appropriate region of the equilibrium curve indicating high relative volatility where the distance between the equilibrium curve and the operating line (the 45° line in the case of total reflux) is large, thus allowing better precision in reading the experimental data.

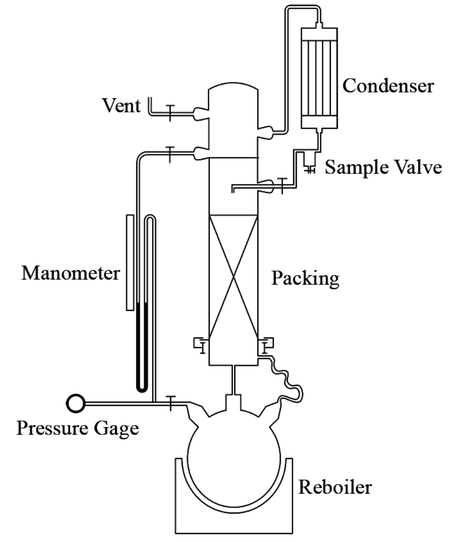

Fig. 1: Schematic diagram of the experimental setup.

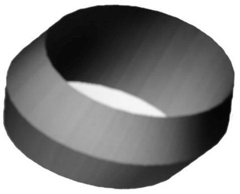

Fig. 2: A typical brass ferrule used as column packing in this work.

All valves are closed except for the sampling valve. The heater is turned on and one waits until the existing air in the column is vented, indicated by a few exiting distillate droplets. Improper venting could force the fittings to open or could cause the column to explode upon heating. The sampling valve is then closed and one waits until a few centimeters of liquid accumulate behind the reflux valve that is then opened such that a constant liquid level is maintained.

The internal column pressure and the pressure drop are measured periodically so that sampling could begin once steady state is reached. One waits for half an hour after the above parameters and the liquid reflux are constant to make sure that concentration in the overhead sampling reservoir is also constant, and then begins sampling.

The overhead liquid is taken from the sampling valve and the reboiler sample is taken by inserting a pipette into the reboiler, both actions are carried out in a few seconds and consecutively.

The experiments were carried out using both a conventional Raschig-type glass ring and the new brass packing.

The measured specifications of the new brass packing and Raschig-type glass ring packing are listed in tables 1 and 2 respectively. The column is first calibrated with typical Raschig-type glass packings and then operated with the new packing.

ADVANTAGES OF THE NEW PACKING

There is still a tendency to use newer kinds of packing with better efficiency and lower pressure drop to achieve more satisfactory column operation. The main reason for carrying out research in this area is lowering operating costs and optimizing profit. The historical trend of the improvement in column packing performance is shown in Fig. 3.

The higher efficiency and lower pressure drop of the new packing can be justified in view of its geometry. The slanted edges of the packing provide high mechanical strength with the lowest possible weight. The tiny grooves existing on the walls of the packing caused by the manufacturing process result in better distribution of the liquid and thus increase the contact area leading to higher efficiency. The height to diameter ratio of 2/3 and the slanted edges provide for better hydrodynamic conditions and result in lower pressure drops. High corrosion resistance because of the material of construction (brass) and availability in a large size range (3 to 50 mm) are other advantages that make the new packing suitable for application in absorption and distillation services.

The packing factor is a factor independent of packing size. It is a good measure for comparison among different packing materials in order to determine which packing has better performance. Tables 3 and 4 show that the new brass packing is either better or comparable in performance with the available types of conventional packing and the newly fabricated ones. The new hollow fiber paking shows better performance (a_p / ε^3 =1357) than the packing introduced in this work (a_p / ε^3 = 1175), this different amounts to about 15 %, however, the packing manufacturing process is a lot more difficult and also column maintenance is very expensive [7]. This emphasizes the advantages of the proposed packing, namely, having lower weight and cost while providing a reasonable packing factor.

Table 1: Characteristics of the packing and the packed section for the brass packing.

Material of construction	Brass
Number of pieces per liter	1880
Weight of each piece (gr)	0.8
Height of each piece (mm)	6
Diameter of the mid section of each piece (mm)	9
Wall thickness (mm)	0.2

Table 2: Characteristics of the packing and the packed section for the glass packing.

Material of construction	Glass
Number of pieces per liter	403
Weight of each piece (gr)	0.89
Height of each piece (mm)	12
Diameter of the mid section of each piece (mm)	10.7
Wall thickness (mm)	0.9

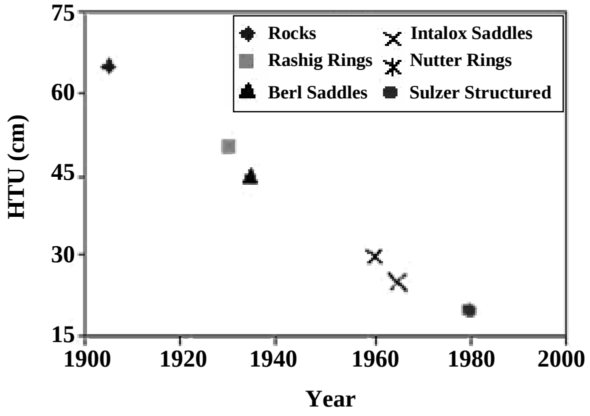

Fig. 3: Historical trend of HTU improvement through the use of different types of packing.

Why brass ferrules?

Low-diameter tubes are usually cut into pieces to make Raschig-type rings for laboratory purposes. Brass ferrules are available in various sizes and need no preparation as pieces used in tube fittings. They resemble conventional Raschig rings in shape but their apparent ratio (height to diameter) is 2 to 3 and their edges are slanted. This particular shape results in high mechanical resistance and has a direct effect on pressure drop.

Table 3: Comparison of the packing factor for different packing materials.

Packing Type	a_p (m^2/m^3)	Reference
Raschig Ring (D=6.35 mm)	700	[4]
Pall Ring (D=25 mm)	48	[4]
Berl Saddle (D=6.35 mm)	900	[4]
Euroform (plastic, D=34.9mm)	110	[5]
Hiflowring 50 (plastic, D=6.35 mm)	192	[5]
Impulsepac (Ceramic, D=32.5 mm)	102	[5]
Packing No. 1 (PVC, D=19 mm)	189	[5]
Packing No. 2 (PVC, D=9 mm)	360	[5]
Glass Packing (D=10.7 mm)	300	This work
Brass Packing (D=6 mm)	856	This work
Hollow fiber as new structured packing (D=8 mm, 50 fibers)	484	[6]

Table 4: Comparison of different packing materials based on the packing factor

Packing type	ε	Packing factor (a_p/ε^3)	Reference
This work's brass packing (D=6 mm)	0.9	1175	This work
This work's glass packing (D=10.7 mm)	0.88	440	This work
Sulzer BX	0.9	206	[6]
Mellapak	0.95	89	[6]
New hollow fiber (D=8 mm)	0.71	1352	[6]

Grooves existing on the surface of these ferrules as a result of machine finishing provide sites for better liquid distribution and increase the contact surface.

An important parameter used for comparing different packing materials is the interfacial area. *Onda* has presented a correlation based on dimensionless numbers for the interfacial area of the random packings in distillation columns [8]:

$$a_w = a_p \left\{1 - \exp\left[-1.45\,\mathrm{Re}_L^{0.1}\,\mathrm{Fr}_L^{-0.05}\,\mathrm{We}_L^{0.2}\,(\sigma/\sigma_c)^{-0.75}\right]\right\} \quad (1)$$

Where the dimensionless *Reynolds*, *Froude* and *Weber* numbers are defined as:

$$\mathrm{Re}_L = L/a_p\mu_L\,;\ \mathrm{Fr}_L = a_p L^2/g\rho L^2\,;\ \mathrm{We}_L = L^2/a_p\sigma\rho_L$$

And the parameters used for their calculation are:

a_w: wetted interfacial area (m^2 interfacial area/m^3 packing volume)

a_p: specific surface area of packing (m^2/m^3)

L: liquid superficial mass velocity ($kg/m^2.s$)

μ_L: liquid viscosity (Ns/m^2)

g: gravitational acceleration (m/s^2)

ρ_L: liquid density (kg/m^3)

σ: surface tension (N/m)

σ_c: critical surface tension (N/m)

In this relation the wetted area of the packing is taken to be the effective mass transfer area. The effective surface area of the packing is also a measure of effective mass transfer area. In low-diameter columns such as the one used in this work, the effective surface area (a_e) is almost equal to the specific surface area [9].

The results obtained from the *Onda* relation for the packings and the hydrodynamic conditions of the column used in this work are presented in table 5.

As can be seen in the mentioned table, the wetted and effective areas of the brass packing are larger than the respective values for glass packing and this could be the reason for the lower value of the HETP of brass packing as compared to the glass packing. *Higler* and *Repke* have shown in the modeling of distillation columns using non-equilibrium methods based on mass transfer rate that the interfacial area has a significant effect on the mass transfer rates between the liquid and vapor phases and the separation process as a whole [10, 11] The higher a_w and a_e which are measures of the interfacial area, the higher the mass transfer rate and separation power which in turn results in the higher efficiency of the packing in terms of lower HETPs.

RESULTS AND DISCUSSION

There are two approaches to calculating the efficiency of packing materials in packed columns called HTU/NTU and HETP. HTU is the height of a transfer unit and NTU represents the number of transfer units which are defined in the following manner:

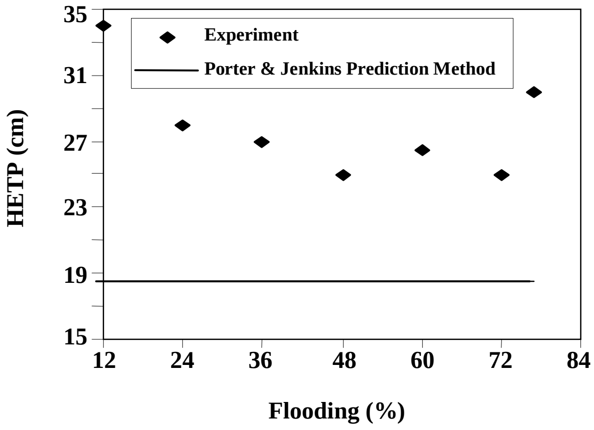

Fig. 4: Height Equivalent to a Theoretical Plate as a function of percentage of flooding for the glass packing (ethanol/water system).

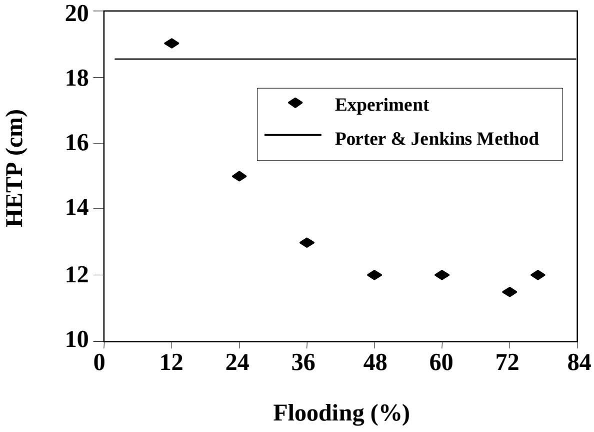

Fig. 5: Height Equivalent to a Theoretical Plate as a function of percentage of flooding for the brass packing (ethanol/water system).

$$HTU = V/ (K^* a ^*c ^*A) \quad (2)$$

$$NTU = \int dy/(y^*-y) \quad (3)$$

$$Hp = HTU * NTU \quad (4)$$

Where V is vapor flow rate, K is mass transfer coefficient, a is effective surface area of packing, c is concentration, A is cross sectional area, y* is equilibrium concentration and Hp is total height of the packed section.

Another approach (and the one used is this work) is HETP which represents height equivalent to a theoretical plate.

Many correlations have been proposed for calculating the efficiency of packed columns. The most dependable correlations such as the *Bravo* model (1982) and the *Wagner* model (1997) cover 90 percent of the data within an error range of 30 percent [12]. These rule of thumb correlations are based on large data bases and give similar results. The *Porter* and *Jenkins* method has been used in this work for calculating HETP because it is a general method requiring only the packing diameter and is simple to use. This simple correlation is given below [13]:

$$HETP(ft) = 1.5D_p (in.) \quad (5)$$

Where D_p is the packing diameter. Thus the HETPs calculated from the above equation for the glass packing and the new packing after conversion are 18.5 and 13.5 cm, respectively.

The measured HETPs are plotted in Figs. 4 and 5 for the two packings and show an average deviation of 30 % for the glass packing and an average error of –7 % for the brass packing in comparison with the correlation value. The lower HETP and higher efficiency of the new packing are apparent because the experimental HETP in Fig. 4 for glass packing is higher than the calculated value which is not desirable while the experimental HETP in Fig. 5 for brass packing is less than the calculated value. To determine the HETP, the available equilibrium data for three mixtures ethanol/water, 1-propanol/water and 1-butanol/water were used to construct the equilibrium curve. The 45 degree line was used as the operating line for both the rectifying and stripping sections since operation was at total reflux. Then the number of theoretical stages needed to achieve the same separation as that obtained in the experiments for a given set of top and bottom compositions was stepped off and the height equivalent to a theoretical stage determined. The average values obtained for the HETP of the brass packing are compared in table 6 with the more sophisticated hollow fiber packing which is a lot more expensive. The performance compares well with baffled hollow fibers and is a lot better than un-baffled hollow fibers.

As a matter of fact the model should be compared with the experimental data and evaluated against it, however, the correlation is used here as a basis for comparison. The correlation underestimates the HETP for the glass packing, which is conservative and safe.

Table 5: Hydrodynamic conditions of the column.

parameters	glass	brass
Re_L	0.391	0.137
Fr_L	6.96E-7	1.96E-6
We_L	1.38E-6	5E-7
a_w	71.16	153.84
$a_p = a_e$	300	856

Table 6: HETP comparison for the brass packing and the hollow fiber packing.

Packing type	HETP (cm)	Reference
Brass packing		This work
ethanol-water	12	
1-propanol-water	14	
1-butanol-water	14	
Hollow fiber		[6]
-Un-baffled	22	
-Baffled	11	

However, it overestimates the HETP for the brass packing. Both deviations are systematic because of the very simple horizontal nature of the correlation used. In this correlation HETP is only a function of the packing diameter and when the packing size is fixed a single value for HETP results.

The variation of pressure drop with liquid and gas flow rates is shown in Fig. 6 for the glass packing and in Fig. 7 for the brass packing. A liquid flow rate of zero indicates the condition in which the packings have just been wetted and liquid flow is about to begin. The pressure drop is lower at lower liquid flow rates and is not greatly affected by it. This could be due to the better hydrodynamic conditions at the cost of lower cross sectional area at lower liquid flow rates. There are many holes and pores available for gas flow in dry packing beds causing great pressure drops as a result of numerous abrupt changes in the flow direction. Once this space is filled with liquid, these changes in direction are eliminated. On the other hand, liquid flow restricts gas flow, thus increasing the pressure drop. These opposing effects tend to compensate for each other at low flow rates and cancel out but the decrease in cross section becomes dominant at higher flow rates. Another interesting observation is the occurrence of flooding at a pressure drop of about 10 cm of water per meter of packed bed regardless of the gas and liquid flow rates for both kinds of packing.

THE BEHAVIOR OF THE HETP CURVE

Liquid holdup, specific contact area, mass transfer coefficients, and the hydrodynamic behavior of the column (most often indicated by its pressure drop) are among the factors considered to play a major role in the performance of a column. In the past, each of these parameters has been studied separately. In newer models, the combined effect of these parameters has been considered. In this work a new relation is presented that relates efficiency (in terms of HETP), which is a parameter whose prediction is difficult, with pressure drop that can be measured easily and accurately or obtained from available correlations.

Pressure drop

Three theories are usually used for modeling pressure drop, and in general, the hydraulics of a column: the channel model, the particles model, and length of free path model. In the first model, pressure drop is considered analogous to the resistance of a number of parallel channels against the flow of gas. The channels could have internal curvature and could expand or contract. Liquid flows downward along the walls of the channels and gas flows upward through them. The wetting of the walls by the liquid decreases the cross section open to gas flow and increases the pressure drop [14]. This model has been used for both structured and random packings but the latter application has enjoyed more success.

In the particle model, one assumes that the drag force of the particles causes the pressure drop. The presence of liquid decreases the void fraction of the bed or in other words increases the size of the particles. Ergun has used this model for single-phase flow in fixed and fluidized beds. *Stichlmair et al.* have applied this model successfully for establishing a relationship between pressure drops and flooding [13].

The length of free path method could be considered an extension of the channel model in which the channels have a finite length called the length of the free path and

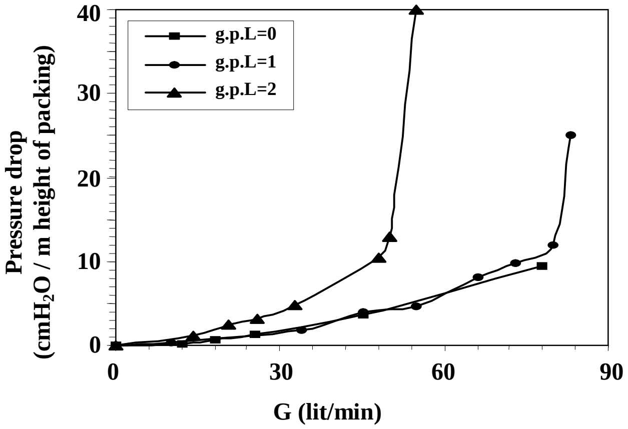

Fig. 6: Pressure drop as a function of the gas flow rate for different liquid flow rates for the glass packing (ethanol/water system).

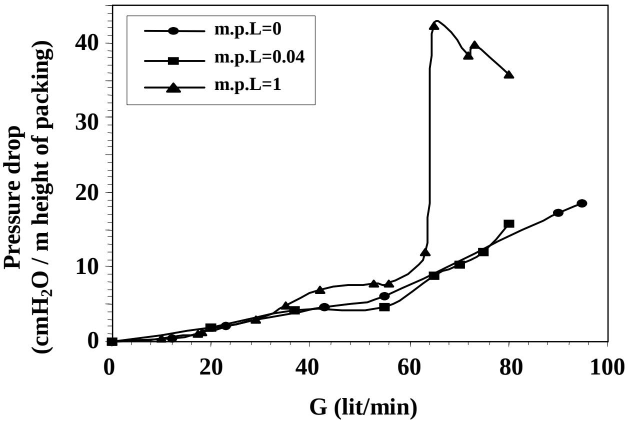

Fig. 7: Pressure drop as a function of the gas flow rate for different liquid flow rates for the brass packing (ethanol/water system).

all liquids exiting the channels, having traveled this path, are mixed and redistributed in channels located below them [15].

PRESENTATION OF THE NEW MODEL

HETP has a rather complex relationship with percentage of flooding. Investigating the efficiency diagrams, one can distinguish three distinct regions: 1- less than 24 %, 2- between 24 and 72 %., and 3- more than 72 %.

HETP decreases sharply in the first region, becomes stable at a minimum value in the second region, and increases sharply in the third region. How can this behavior be justified? So far, researchers have held mal distribution accountable for this behavior. But we propose the following justification that can be considered a theory based on dry and filled cells resulting in a relationship between efficiency and pressure drop.

One can consider the internal space of the bed to consist of small cells through which liquid and gas flow. These cells can be classified into three categories: 1- dry cells into which no liquid flows, 2- filled cells, i.e., cells filled with liquid into which no gas flows, 3- useful cells into which both liquid and gas flow. The first two groups play no role in mass transfer or the efficiency of the column and their minimization minimizes the HETP of the column.

In the first region of the efficiency diagrams of Figs. 4 and 5, since both liquid and vapor flow rates are low (column is at total reflux), most of the cells are dry playing no role in mass transfer and, therefore, HETP is high. Increasing the liquid and vapor flow rates decreases the number of dry cells and one reaches the second region. But this region, despite appearances is not homogeneous as far as internal flows are concerned, i.e., although the HETP at 24 % flooding is equal to the HETP at 72 % flooding, as an example, the internal conditions of the column are quite different. There are two opposing factors here: reduction in the number of dry cells and increase in the number of flooded or filled and plugged cells.

Thus, increasing the percentage of flooding causes a decrease in the number of dry cells and an almost equal increase in the number of filled and plugged cells till one reaches a loading point of 70 to 80 % of flooding where there are no more dry cells to counterbalance the increasing number of the filled cells and this is the beginning of the third region. Another justification for this claim is the trend of the flooded cells curve in Fig. 8.

The friction factor between air and water is not all that different from the factor between air and metal or air and glass, therefore, one can consider the variation in pressure drop to be only a function of the number of plugged cells that narrow the path of gas flow. Thus the monotonous increase in pressure drop can justify the presented theory of the counterbalancing effect of the dry and filled cells. It is clear from the diagram that the pressure drop increases monotonically from 0 to 100 % of flooding indicating an increase in the number of filled cells while the efficiency diagram presents a complex behavior in the same interval.

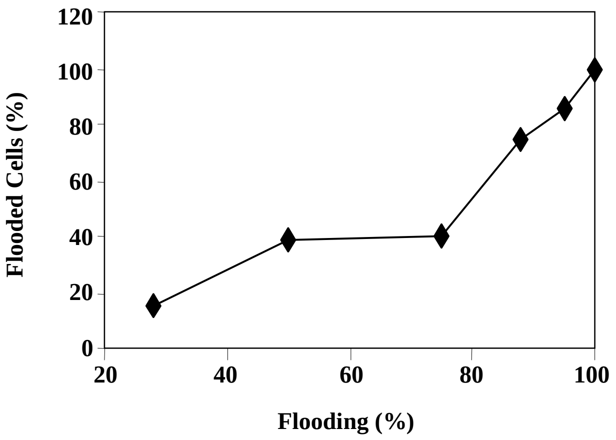

Fig. 8: Percentage of flooded cells as a function of percentage of flooding (ethanol/water system).

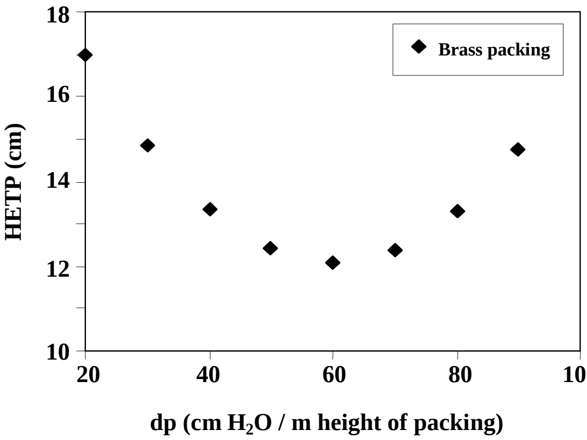

Fig. 9: HETP as a function of pressure drop for brass packing.

In order to justify the trend of the HETP curve in the diagram, we only need to find a relationship between the rate of increase in the number of filled cells and the rate of decrease in the number of dry cells.

It is obvious that both filled cells and dry cells are useless in mass transfer, therefore, their sum is inversely proportional to efficiency and directly proportional to HETP. One can conclude that HETP is minimized when the sum of the percentage of dry cells and the percentage of filled cells is at a minimum and this sum could be considered a criterion for efficiency.

HETP has been drawn as a function of pressure drop in Fig. 9. The following second degree polynomial fits the curve with a perfect coefficient of determination ($R^2=1$):

$$HETP=22.993 - 0.3614 (\Delta P) + 0.003 (\Delta P)^2 \qquad (6)$$

CONCLUSIONS AND RECOMMENDATIONS

The suitability of using brass ferrules that are readily available, relatively cheap, light, corrosion resistant, and have good mechanical strength, for pilot plant studies was indicated. This packing has a higher efficiency compared to traditional packings.

The existing theories on fluid flow in packed beds were investigated and extended based on the observations of the systems studied. The inutility of filled and dry cells for mass transfer was recognized and the minimization of their total number by adjusting the operating conditions, most importantly flow rates, as a means of optimizing the efficiency was established.

A relationship was presented that relates efficiency in terms of HETP, which requires tedious experimental measurements, with pressure drop which is easily measured. This relationship can be used for calculating the satisfactory performance region of the column from measured or calculated pressure drops for the given packing. This satisfactory performance region helps in controlling the operating parameters such the flow rates to optimize efficiency.

We recommend conducting the experiments in larger columns and under conditions other than total reflux so that the results could be used in scale-up work.

Nomenclatures

A	Cross sectional area (m^2)
$a = a_e$	Effective surface area of packing (m^2 interfacial area/m^3 packing volume)
a_w	Wetted interfacial area (m^2/m^3)
a_p	Specific surface area of packing (m^2/m^3)
c	Concentration ($kgmol/m^3$)
g	Gravitational acceleration (m/s^2)
D_p	Packing diameter (in.) [in equation (5)]
HETP	Height Equivalent to a Theoretical Plate (ft) [in equation (5)]
Hp	Total height of the packed section (m)
HTU	Height of a Transfer Unit (m)
K	Mass transfer coefficient (m/s)
L	liquid superficial mass velocity ($kg/m^2.s$)
NTU	Number of Transfer Units
V	Vapor flow rate (kg/s)
y*	Equilibrium concentration

Dimensionless Numbers

Re_L	Liquid Reynolds number
Fr_L	Liquid Froude Number
We_L	Liquid Weber Number

Greek Letters

ε	Porosity
σ	Surface tension (N/m)
σ_C	Critical surface tension (N/m)
ΔP	Pressure drop (cm H_{2O}/m of packing)
ρ_L	Liquid density (kg/m^3)
μ_L	Liquid viscosity (N.s/m^2)

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