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ESTABLISHING A FACILITY FOR THE HYDRODYNAMIC CHARACTERISATION OF DISTILLATION COLUMN INTERNALS

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Abstract: A better understanding of the hydrodynamics related to flooding conditions in distillation columns is required in order to provide optimised designs for new plants and plant retrofits. Current flooding prediction correlations are predominantly based on air/water systems and tend to diverge at high vapour capacities. In this work an experimental setup was designed and constructed to test the hydrodynamic characteristics of random – and structured packing as well as sieve – and valve trays. The experimental setup is used to develop a better understanding of the influence of gas and liquid properties on entrainment and column flooding. The experimental setup was tested with an air/water system and the tray entrainment data followed the trends from Bennett *et al.*[1] quite well. The packed column yielded the correct trends on both pressure drop and liquid hold-up data which correlated reasonably well with the general model proposed by Piché *et al.*[2].

Keywords: Hydraulic capacity, Liquid hold-up, Entrainment, Sieve – and valve trays, Random – and structured packing.

1. Introduction

Significant contributions[1-18] have been made to increase our understanding and knowledge of the hydrodynamics in both packed and tray columns. The influence of gas and liquid flow rates, column geometry, and gas and liquid physical properties on entrainment, liquid hold-up, and pressure drop has been investigated previously. However, a larger portion of the measured data consisting of air/water data and the effect of physical properties has been largely neglected.

For packed columns some of the recent hydrodynamic models developed include those by Olujic[9], Brunazzi and Pagliante[10], Rocha *et al.*[12], Verschoof *et al.*[13], Billet and Schultes[14], Kister and Gill[15], Robbins[16], Maćkowiak[17], and Piché *et al.*[2] Many of these hydraulic models were derived from either: 1) a range of system-specific experiments or 2) a pooled database of industrial data. Some of the models that predict the pressure drop and liquid hold-up in structuredly packed columns, predict a conservative dependency on the viscosity[19]. Thus, further investigation is required with fluids that have varying physical properties.

For tray columns most of the data and correlations found in literature focus on the onset of entrainment (L'/G < 5 %) and the prediction the flooding velocity[3,4]. These correlations are based on data obtained from a variety of experimental setups and from various institutions. Entrainment correlations from different authors[1,6,7,8] were compared with data from literature over a range of gas and liquid flow rates, tray spacings, and tray geometries to investigate possible deviations. The Colwell[18] correlation was used to calculate the liquid hold-up required for some of the entrainment correlations[6,7,8]. From the investigation an increase in gas velocity resulted in the most significant deviation between the trends, as shown in Figure 1. The prediction from Kister and Haas[8] (K & H), showed a strong dependency on gas velocity and compared well with the prediction of Bennett *et al.*[1] up to 1.4 m/s in Figure 1(a) and 2 m/s in Figure 1(b), whereafter a significant deviation was noted. The trends from Hunt *et al.*[6] and Thomas and Ogboja[7] (T & O) in Figure 1 (a) did not agree with those of Bennett *et al.*[1] and Kister and Haas[8]. This was expected since Hunt *et al.*[6] developed their correlation from stagnant liquid data while the measurements made by Thomas and Ogboja[7] were much lower than the entrainment rates presented here. With possible deviations in up or downstream processes,

and increased capacity demands from old or retrofitted columns, higher fractions of the liquid on the tray is expected to be entrained (L'/G > 5 %). Ultimately one would thus like to extent the current database by testing over a range of gas and liquid physical properties as well as gas and liquid flow rates at higher entrainment (L'/G > 5 %) rates. It is therefore important to conduct testing at these rates. A need therefore exists to conduct hydrodynamic characterisation at higher flow rates and for systems with varying gas and liquid properties/ the aim of this paper is to present an experimental setup that is able to conduct the required hydrodynamic characterisation and, using an air/water system, prove that the setup produces reliable results.

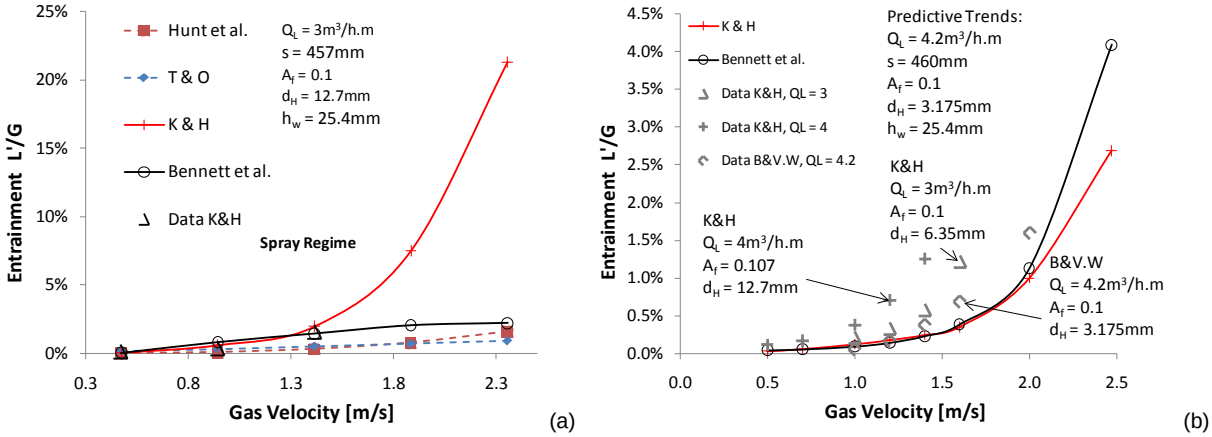

Figure 1 Comparison of the effect of gas velocity on entrainment for the different entrainment prediction correlations[1,6,7,8] with existing data from Kister and Haas[8]. Dotted lines indicate extrapolation beyond recommended range of application.

Table 1. Sensor specifications.

	Differential Pressure Transmitters	Absolute Pressure Transmitters	Liquid Flow Meter (High Flow)	Liquid Flow Meter (Low Flow)
Supplier	Endress & Hauser	Endress & Hauser	Flowmec	Flowmec
Measuring Range	0 – 10 kPa	0 – 200 kPa abs	1.8 – 27 m^3/hr	0 – 2 m^3/hr
Turn-down Ratio	15:1	15:1	Adjustable span	Adjustable span
Measuring Accuracy	0.075 % MV	0.075 % MV	0.25 % FS	0.25 % FS

2. Experimental Setup

Motivated by the shortcomings in the literature a pilot plant was designed to conduct hydrodynamic characterisation of packed and tray columns. To test the hydrodynamic capacity of the tray- and packed columns, the following should be measured: gas and liquid flow rates, tray and packed bed pressure drop, absolute column pressure, gas and liquid temperatures, entrainment (tray column only), weeping (tray column only), and liquid hold-up. The following parameters can be varied to test their effect on the hydrodynamic capacity: gas and liquid physical properties (viscosity, density, surface tension), gas and liquid flow rates, tray spacing, tray type (sieve and valve), and packing material. The insides of the columns are visible to identify different states and to correlate the states with the data. Sensors are placed at appropriate locations to generate accurate and reliable data to characterise the system. The columns were designed so that the influence of selected variables can be tested while other variables are kept constant. To accurately test the hydraulic capacity the effect of mass transfer has to be eliminated by using liquids with low vapour pressures and high boiling points with non-reacting gasses. The columns were placed in parallel using the same utilities to reduce capital costs. The packed column can accommodate both random-and structured packing. Random packing (IMTP® 40) was used to verify the preliminary results regarding accuracy and reproducibility of the packed column. A sieve tray was used to verify the tray column setup. Table 1 represents the sensor specifications used in both columns. A schematic representation of the experimental set-up is given in Figure 2.

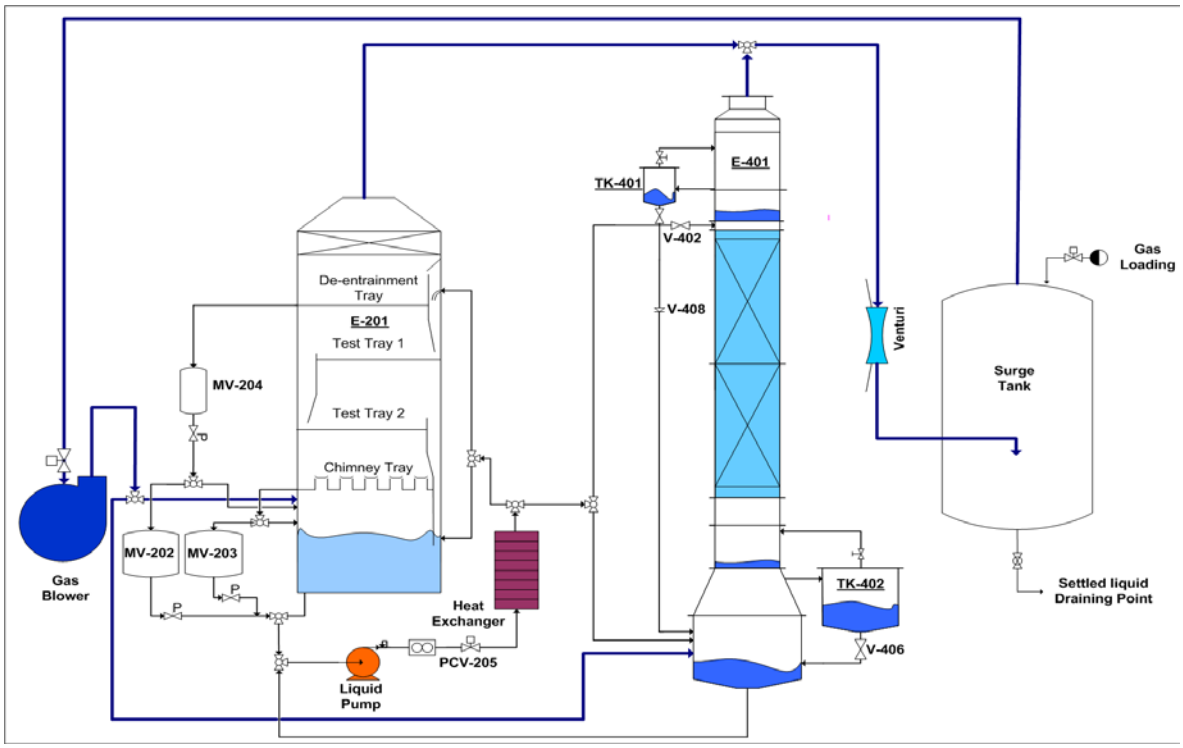

Figure 2 Process flow diagrams for the tray and packed column.

2.1 Tray column design

The tray column (E-201) has a rectangular shape (0.175 × 0.635 m) to eliminate expansion and constriction of the liquid flow path at the downcomer outlet and exit weir. The expansion and constriction of the liquid flow path occurs in circular columns at varying degrees, depending on column diameter. Gas enters the column through a chimney tray and then passes through the two test trays. Two test trays are used to accurately represent hydrodynamic conditions experienced during weeping and entrainment. A de-entrainment tray is located above the top test tray and a demister fitted at the top of the column. Window sections are placed on the downcomer sides and on the front to create a view of the flow path. The liquid that weeps through the bottom test tray is collected on the chimney tray and transferred to either the sump or the hold-up tank (MV-203) through an isolated pipe line. A closed downcomer is therefore used to transfer the liquid from the bottom test tray to the sump. The entrained liquid is separated from the gas in the de-entrainment section and transferred to the sump or hold-up tank (MV-202/4). The tray pressure drop is measured using differential pressure transmitters, as shown in Table 1.

2.2 Packed column design

The packed column has an inner diameter of 387-393 mm. This size was chosen as one of the industrial suppliers has a standard 387 mm structured packing size which will be used in future experiments. The size is as economically close as possible to the suggested diameter of 400 mm. At this size the wall effects become less significant. The column sections are made of borosilicate glass to allow visualisation of the different operational stages. The gas enters through the sump section and is distributed by a chimney tray before it reaches the packed bed (3000 mm height). This packed bed can be either structured or randomly packed material. Above the packed bed is the liquid distributing section where one of three different drip-point liquid distributors is used to cover the range of liquid flow rates. A de-entrainment section is placed above the liquid distribution section to remove most of the entrained liquid. The entrained liquid is then transferred to vessel TK-401 and measured using a differential pressure transmitter. The liquid hold-up in the column is measured with the same method using vessel TK-402 by cutting the feed to the column (V-402) and closing the pneumatic ball valve (V-406). In order to obtain accurate liquid hold-up measurements, the liquid on the chimney- and liquid distributor plates is measured by monitoring the liquid levels through viewing ports. The pressure drop over the packed bed is measured with a differential pressure transmitter. The total column pressure is monitored with an absolute pressure transmitter. Five PT-100 temperature probes are inserted in the column to ensure that the experimental conditions are well recorded during sampling. These probes

are positioned to measure the following: 1.) the temperature of the liquid entering the column. 2.) The temperature of the liquid being drained from the sump. 3.) The temperature of the liquid in the hold-up tank (TK-402). 4.) The temperature of the gas directly below the packing material. 5.) The temperature of the gas directly above the de-entrainment section.

2.3 System Operation

Liquid is circulated by means of a centrifugal pump. The liquid flow rate is controlled by a pneumatic control valve and the pump revolutions can be changed using an inverter. Gas is circulated through the system using a centrifugal gas blower. The gas flow rate is controlled with both an inlet radial vane control valve placed on the blower inlet and an inverter. The system pressure is controlled using a pneumatic control valve connected to a gas cylinder. In the hold up vessels differential pressure transmitters monitor the liquid level while PT-100 temperature sensors monitor the liquid temperature. To measure the gas flow rate for a range of gasses a venturi meter is used. The liquid volumetric flow rate is measured with 2 interchangeable positive displacement flow meters (for low and high flow rates respectively). A plate heat exchanger with cooling water removes any excess energy, generated by the gas blower and pump, from the system. The surge tank acts as a droplet settling tank as well as a dampener to prevent system pressure oscillations.

3. Experimental Results

3.1 Entrainment in Sieve Tray Column

The experimental results obtained for an air/water system at atmospheric conditions are shown in Figure 3 (a) and compared with trends from literature[1,5] for 5 % entrainment (L'/L) in Figure 3 (b).

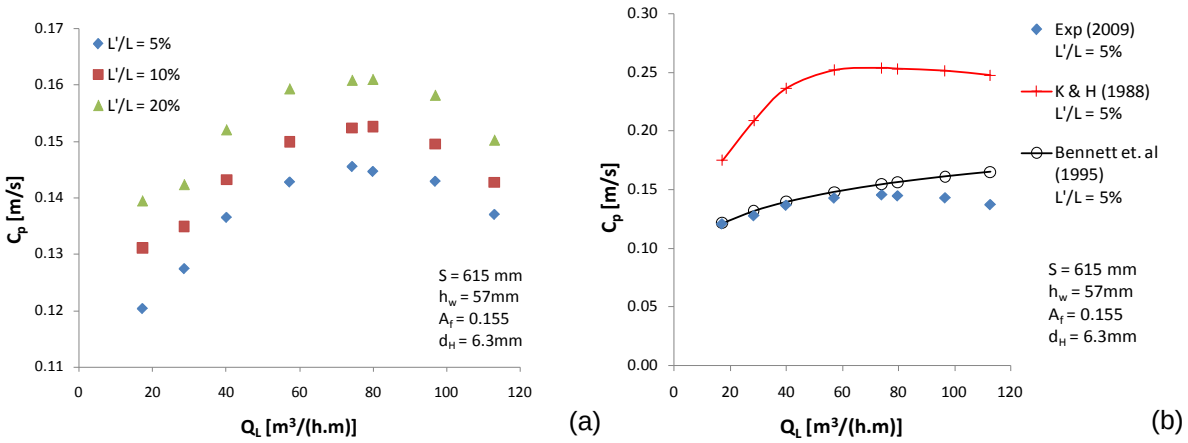

Figure 3 (a) Entrainment results of an air/water system at 25 °C and 1 atm as a function of gas and liquid flow rate. (b) Comparison of entrainment predictive trends[1,5] with experimental data for 5 % entrainment.

Figure 3 (a) shows that the gas velocity had to be increased as the liquid flow rate increased from 17-75 m^3/(h.m) to maintain constant entrainment. For liquid flow rates exceeding 75 m^3/(h.m) the gas velocity was reduced to maintain constant entrainment. This changeover is caused by the high liquid flow rate and short (455 mm) flow path length which creates a non-uniform (undeveloped) froth that pushes up against the column wall at the exit weir. This increase in liquid hold-up at the exit weir results in a significant increase in entrainment. The experimental data is compared with trends from Bennett *et al.*[1] and Kister and Haas[8] in Figure 3 (b). The data follows the trend from Bennett *et al.*[1] very well up to the point where the froth is no longer uniform (75 m^3/(h.m)). Although the gas velocities are within the recommended range of application for their correlation, Kister and Haas[8] based their correlation on lower gas velocity ($C_{p,max}$ = 0.1 m/s) data.

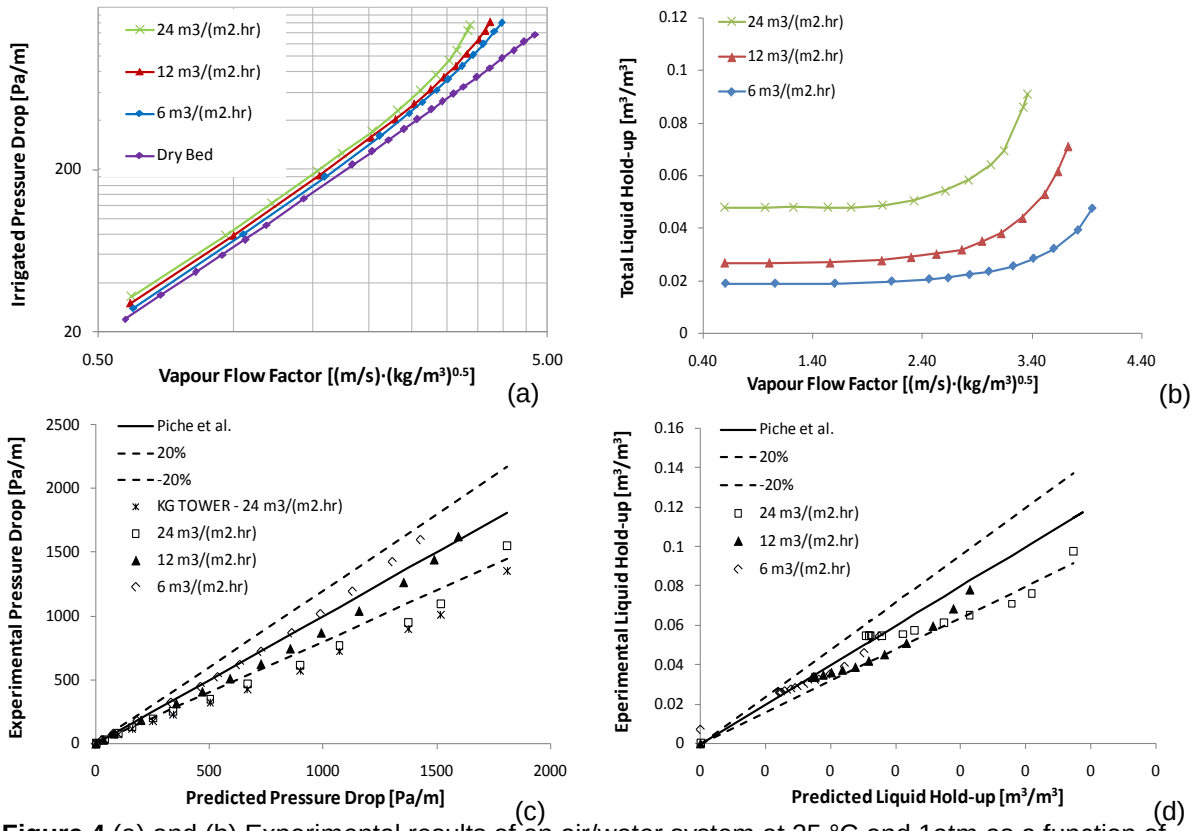

Figure 4 (a) and (b) Experimental results of an air/water system at 25 °C and 1atm as a function of vapour flow factor compared to pressure drop and liquid hold-up. (c) and (d) Parity plots of the experimental data compared to the predictive model found in the literature[2].

3.2 Randomly Packed Column

The experimental results obtained from the randomly packed column, in an Air/water system at atmospheric conditions and with 40 mm IMTP® as packing, are shown in Figure 4. From Figure 4 (a) and (b) it can be seen that the correct trends were obtained for both pressure drop and hold-up data. The model proposed by Piché *et al.*[2] was chosen as no packing constants are needed and it has a mean relative error of 20 %. Most of the data fits well within these limits on the parity plots. However, at higher liquid loads a deviation is noticed on the pressure drop. To verify the higher load pressure drop data, the software package KG-TOWER[20] was used to predict the pressure drop trend. It can be seen that the two curves are nearly identical and fall within 10 % of each other. From the hold-up data it seems that the model over-predicts the loading region hold-up for all the flow rates investigated. This could be due to the fact that the model used did not include any experimental data on IMTP® 40 even though it should be accurate over the flow rates investigated. Thus further validation will be done on a different random packing with data commonly found in the literature, as well as higher liquid loads than those investigated in this work (up to 120 $m^3/m^2 \cdot hr$).

4. Conclusions

A literature survey showed that more data at higher entrainment rates and gas velocities (> 2 m/s) is required to determine accurate hydrodynamic behaviour in distillation columns. In order to understand the influence of gas and liquid physical properties on hydrodynamic behaviour, a range of gasses and liquids should be used in related experiments. An experimental setup that is capable of generating hydrodynamic capacity data for a range of gasses and liquids was successfully constructed. Air/water entrainment data was generated in a sieve tray column and compared with trends from literature. The data followed the predictive entrainment trend from Bennett *et al.*[1] well for liquid rates up to 75 $m^3/(h.m)$. At higher rates a deviation due to non-uniform froth formation was noted. The experimental data generated with the 40 mm IMTP® packing correlated reasonably well with the data from the predictive models. Based on these results the accuracy of the experimental data generated with this packed column setup is partially verified.

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Nomenclature

A_h	Total hole area, m^2	h_w	Weir height, mm
A_f	Fractional hole area (A_h/ A_p) or tray free area	L	Liquid mass flow (Entering the tray from the downcomer), kg/s
A_p	Perforated (Active) area (including blank areas), m^2	L'	Entrained liquid mass flow, kg/s
C_p	Capacity factor based on A_p, $C_p = u_p\sqrt{\rho_g/(\rho_L-\rho_g)}$, m/s	Q_L	Liquid flow per weir length, m^3/(h.m)
d_H	Hole diameter, mm	s	Tray spacing, mm
G	Gas/Vapour mass flow, kg/s	ρ_g	Gas/vapour density, kg/m^3

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