CHEMICAL ENGINEERING TRANSACTIONS

VOL. 69, 2018

A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet

Guest Editors: Elisabetta Brunazzi, Eva Sorensen
Copyright © 2018, AIDIC Servizi S.r.l.
ISBN 978-88-95608-66-2; **ISSN** 2283-9216

DOI: 10.3303/CET1869009

Evaluation of the $k_L a_e$ of Structured Packings in Small Diameter Columns - The Key Role of Wall-Wipers

John F. Roesler*[a], Pascal Alix[a], Aude Royon-Lebeaud[a], Lukas Valenz[b], Jan Haidl[b], Frantisek Rejl[b]

[a]IFP Energies Nouvelles Lyon, Rond-point de l'echangeur de Solaize, Solaize 69630, France
[b] Department of Chemical Engineering, Prague Institute of Chemical Technology, CZ-166 28 Prague 6, Czech Republic
john.roesler@ifpen.fr

Wall wipers are commonly used in columns filled with structured packings in order to reduce the liquid flow along the column walls. In small diameter columns wall regions are prominent and wall flow, if not well tended to, can adversely affect measured mass transfer parameters, particularly the liquid side mass transfer coefficient $k_L a_e$. Wire gauze wall wipers provided by some manufacturers do not properly prevent wall liquid flow. In this work, specific wall wipers designed to re-direct liquid wall flow onto packing surfaces were tested against standard wall wipers that do not always properly reduce wall flow. Tests were run on two distinct facilities each with a column diameter of about 150 mm and with bed heights from 0.42 to 1.68 m of Mellapak™ 250.Y packing. The results demonstrate that with the more effective wall wipers it is possible to measure intrinsic height independent values of $k_L a_e$ that match those obtained in a 300 mm diameter column with standard wall wipers.

1. Introduction

The design of industrial absorption or distillation columns is increasingly based on rate based models that require knowledge of the intrinsic mass transfer parameters of the packing materials with which they are to be filled. The determination of these parameters is often performed by global absorption or desorption experiments with adapted chemical systems as reviewed recently by Hegely et al. (2017). Such measurements can be affected by end effects, height and wall flow effects. End effects are related particularly to the gas and liquid distributor set-ups and can be accounted for by specific measurements that generally involve changing the bed height. Height effects can be due to variations in liquid distribution along the bed and require making measurements at different bed heights, either through sampling probes (e.g. Valenz et al., 2011), which tend however to be intrusive, or by changing the bed height. Wall flow, in the case of structured packings, develops in the small gap between the packing and the wall. In order to reduce this flow, metal strips called wall wipers are attached around the packing elements to drive liquid build-up at the walls back into the packing.

In small diameter columns that are often used as a first means of evaluating packing performances the contribution of wall effects are of more concern due to the larger surface to volume ratio. Wire gauze wall wipers provided by some manufacturers do not seem to fulfill their purpose. Their ineffectiveness (at least for the aqueous systems) manifests itself through significant visible liquid wall-flow overcoming the wall-wipers and intensifying such as to alter the measured parameters along the column. In particular, wall flow will generate considerable liquid by-pass and will reduce the film velocity within the packing. Both of these flowfield alterations will tend to reduce the mass transfer rate and consequently the measured volumetric liquid side mass transfer coefficient $k_L a_e$.

The present study investigates the role of this wall flow on the evaluation of the intrinsic $k_L a_e$ of a structured packing, specifically here the Mellapak™ 250.Y (M250Y), by comparing measurements with and without additional wall wipers of a specific design that strongly reduces liquid flow along the walls.

Please cite this article as: Roesler J., Alix P., Royon-Lebeaud A., Valenz L., Haidl J., Rejl F., 2018, Evaluation of the klae of structured packings in small diameter columns - the key role of wall-wipers, Chemical Engineering Transactions, 69, 49-54 DOI: 10.3303/CET1869009

2. Experimental Methods

Measurements were performed to determine the $k_L a_e$ in two different 150 mm diameter columns (UCTP-DN150 and IFPEN-DN146) with beds consisting of 2 to 8 elements of stainless-steel M250.Y packing thus providing bed heights from 0.42 to 1.68 m. The chemical systems were however different: at UCTP $k_L a_e$ were measured by O_2 desorption from water into nitrogen at very low gas flowrates, while at IFPEN the measurements were based on the absorption of CO_2 mixed with air into a dilute MDEA/water solution (Roesler et al. 2016).

The packing elements from the manufacturer are provided with two wall wipers of a gauze material, the function of which is to prevent liquid build-up along the walls Observation in a transparent column shows however that, below the wall-wiper, a significant fraction of liquid stays in the gap between the packing and the wall. A more effective wall wiper was designed, made with plain sheet metal and with a saw-tooth structure at the bottom, the ends of which were bent to penetrate into the packing so as to re-inject the liquid well into the bed. Figure 1a shows a picture off a packing element with one extra wall-wiper (EWW). A recent study (Rejl et. al, 2017) implemented two layers of such wall wipers to perform $k_L a_e$ measurements.

(a) (b)

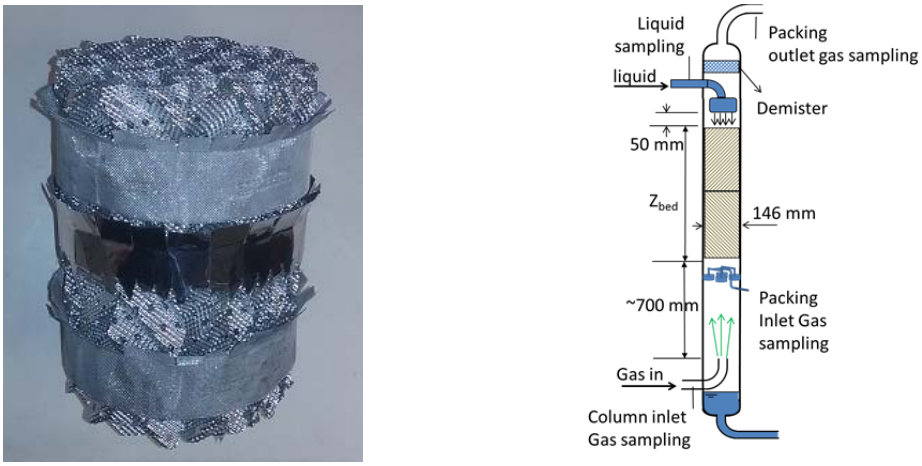

Figure 1: Specifics of the set-up in the IFPEN-DN146 column: (a) packing element with one extra saw-tooth wall-wiper (EWW) and (b) schematic of the column with two packing layers.

2.1 UCTP-DN150 Facility and Procedure

The experimental set-up and procedure on the UCTP-DN150 have been described in detail elsewhere (Rejl et al., 2017). The 150 mm ID column operates at atmospheric pressure and near ambient temperature in a counter-current flow regime. The liquid is tap water saturated in oxygen at 20°C and ambient pressure in a 0.5 m^3 tank. The liquid is pumped around into the column which it enters at the top and is injected 4 cm above bed through a 25 drip-point liquid distributor (1400 per m^2). The gas is nitrogen and is set to a superficial gas velocity of 0.02 √Pa. The liquid is sampled either from the inlet of the column or from the liquid collector bellow the packing for analysis by an optical oxygen probe. The volumetric global mass transfer coefficient, $k_L a_e$ of the bed for molecular oxygen as the solute is then evaluated as

$$k_{L,O_2} a_e = \frac{u_L}{Z} \ln\left(\frac{C_{O_2,in}}{C_{O_2,out}}\right) \quad (1)$$

where u_L is the liquid superficial velocity, Z is the bed height and C_{O2}, is the bulk liquid oxygen concentration measured by the oxygen probe.

2.2 IFPEN-DN146 Facility and Procedure

The IFPEN-DN146 facility (Figure 1b) is a stainless steel 146 mm ID column that can operate under pressure at ambient temperatures. The absolute pressure in this study was 1.5 bar. The column height can be adjusted in sections to hold bed heights of up to 6 m. The liquid is recirculated from a 0.5 m^3 tank and injected at the top of the bed through a distributor with 16 drip points located 50 mm above the top of the packing bed. The

gas is injected through a 50 mm diameter 90° elbow along the centerline axis of the column approximately 700 mm below the packing bed bottom section. The carrier gas in this study is air.
The chemical system used for determining $k_L a_e$ is the reactive absorption of 0.9 %vol CO_2 air into a solution of 3 %wt MDEA partially loaded between 0.1 and 0.2 mol_CO_2/mol_MDEA in order to operate with stable kinetic. At lower loadings the absorption kinetics are too fast due to reactions of CO_2 with OH^- while at higher values the driving force becomes altered by equilibrium considerations. Under the conditions mentioned above, this system meets the film model criteria for a pseudo-first order irreversible reaction given by Danckwerts and Sharma (1966). The rather weak absorption fluxes and consequently slow depletion rates of the gas phase solute make this method well suited for columns with a number of transfer units on the liquid side, NTU_L, greater than 2, where physical desorption methods may lead to high uncertainties (Hegely et al., 2017). Measurements with this system give results comparable to physical absorption methods (Roesler et al., 2016) provided the acceleration factor remains below 1.4 (or k_L > 3×10^{-5} m/s). The volumetric global mass transfer coefficient, $k_L a_e$ of the bed is then evaluated by assuming a 1-D system with negligible changes in liquid phase properties:

$$k_{L,CO_2} a_e = \frac{He_{CO_2} \cdot u_G}{Z_{bed} RT} \ln\left(\frac{y^*_{CO_2} - y^o_{CO_2\,in}}{y^*_{CO_2} - y^o_{CO_2\,out}} \right) \cdot \frac{1}{E} \qquad (2)$$

where u_G is the superficial gas velocity, R the gas constant, T the gas temperature, He_{CO_2} the Henry constant of CO_2 for the loaded solution, $y°_{CO_2}$ is the mole fraction of CO_2 in the bulk of the gas phase and $y*_{CO_2}$ the gaseous CO_2 mole fraction at equilibrium with the loaded liquid solution. Finally E is the acceleration factor given by:

$$E = \frac{Ha}{\tanh(Ha)} \qquad (3)$$

with

$$Ha = \frac{\sqrt{k_2 D_{L,CO_2}}}{k_{L,CO_2}} \qquad (4)$$

where k_2 is the pseudo first order reaction rate constant of CO_2 in the solution, and D_{L,CO_2} is the liquid phase diffusivity of dissolved CO_2. Since E is a function of k_{L,CO_2} the solution is found iteratively. The thermodynamic and kinetic parameters are calculated from a proprietary code. For reference the values are specified here for a 3 %wt MDEA solution at 20°C, 1.5 atm and a CO_2 loading of 0.1 : $y*_{CO_2}$ = 1.74 × 10^{-4}, He_{CO_2} = 2625 Pa/(mol/m^3), D_{L,CO_2} = 1.543 × 10^{-9} m^2/s and k_2 = 0.612 s^{-1}. Under the recommended experimental conditions the ratio $y*_{CO_2} / y°_{CO_2}$ is less than 0.02, E does not exceed 1.15 at the lowest $k_L a_e$, and He_{CO_2} is within less than 5% of its value for water. Therefore the results are rather insensitive to the thermo-kinetic uncertainties of the CO_2-MDEA system.

2.3 Measurement values and units

Gas samples are analyzed for CO_2 with an on-line NDIR analyzer. The amine solution is collected at the column inlet and outlet and its composition is analyzed by titration in terms of amine content and CO_2 loading.
In order to compare results from other studies using other solutes, all values are corrected back to their equivalent for oxygen which is used as the reference solute with a temperature of 20°C. The correction is given by:

$$(k_{L,O_2} a_e) = (k_{L,i} a_e) \left(\frac{D_{L,i}}{D_{L,O_2}} \right)^n \qquad (5)$$

where the power coefficient is 0.5 according to the penetration theory which is rather well verified experimentally (e.g. Rejl, 2017).

3. Results and Discussion

In a first series of measurements in the UCTP-DN150, only the original wall wipers were used. The bed $k_L a_e$, also referred to as the “brut” values, are presented in Figure 3a as a function of the liquid load B and are seen to decrease with bed height. In order to remove possible entrance effects, a net $k_L a_e$ was calculated by removing the solute mass flux in the first two elements of the bed which represents the minimum repetitive

structure and length to reach a well distributed liquid film owing to the orientation of the metal sheets and to the typical 90° rotation between the packing elements. The net $k_L a_e$ is given by:

$$(k_{L,O_2} a_e)_{net} = \frac{(k_{L,O_2} a_e) Z_{bed} - (k_{L,O_2} a_e) \cdot Z_{2-ELEMENTS}}{Z_{bed} - Z_{2-ELEMENTS}} \quad (6)$$

Figure 3b shows that the net $k_L a_e$ have lower values that still decrease with the bed height.

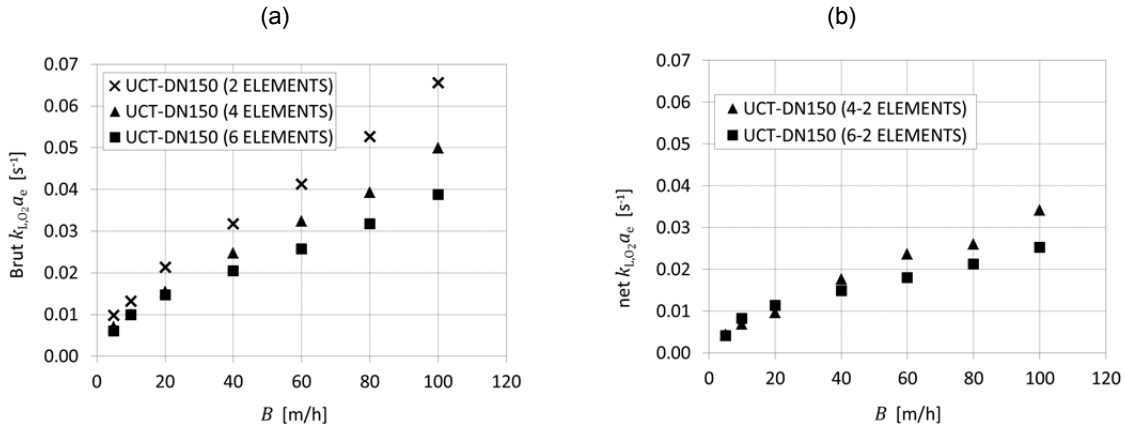

Figure 3. $k_L a_e$ measured with original wall wipers only for different bed heights in the UCTP-DN150 column at F_S = 0.02 √Pa for (a) brut values and (b) net values.

Under the above conditions, the UCT transparent column showed significant wall flow to develop as early as the first packing layer and to intensify along the entire bed. To reduce this wall flow, specific saw tooth wall wipers were added to each packing elements. These wall wipers largely reduce observable liquid flow at the walls. The measured $k_L a_e$ are reported in Figure 4. In this configuration the brut $k_L a_e$ still decrease in value along the bed (Figure 4a), however, after removing entrance effects, the net $k_L a_e$ (Figure 4b) are independent of the packing height. In addition, the net values are now up to 60 % higher than without the extra wall wipers (EWW) which indicates that the packing is operating more efficiently.

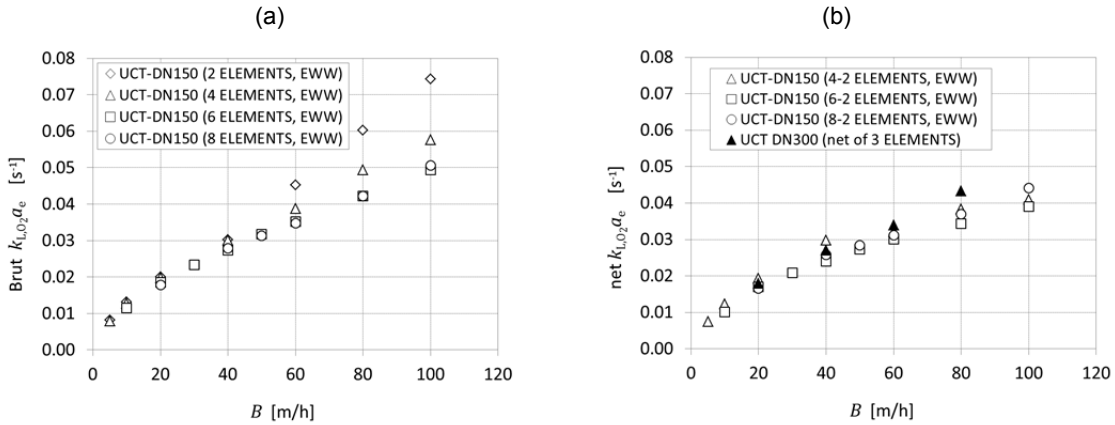

Figure 4. $k_L a_e$ measured with extra wall wipers (EWW) different bed heights in the UCTP-DN150 columns at F_s = 0.02 √Pa (a) brut values and (b) net values.

Figure 4b also displays results from a 300 mm ID column (UCTP-DN300) with a bed of 4 packing elements having only original wall-wipers. In this facility the liquid sampling is performed by probes inserted within the packing elements about 10 cm below the top of the bed and 10 cm from the bottom of the bed, thus removing end-effects and providing a net value for an equivalent bed height of 3 elements (Valenz et al., 2011). The values match well with those from the smaller diameter column, indicating that with the extra wall wipers have

been removed wall effects. A small deviation is observed at the highest liquid load of 80m/h due possibly to liquid inlet effects that propagate further than the first 10 cm of bed height.

To confirm these results, the same type of wall wipers were added to a different set of M250Y packing elements and tested in the IFPEN-DN146 column. The results are shown in Figures 5a and 5b respectively for the brut and net $k_{L,O_2}a_e$. The absolute values are lower by 25% relative to those from UCTP for reasons that are not well identified at this time. Nonetheless, the relative trends are comparable and the same conclusions are reached regarding the absence of bed height variations once the entrance effects are removed. Figure 5b shows rather large scatter for the net $k_L a_e$ with a bed of 4 elements. The uncertainties are due low extents of CO_2 depletion in the gas phase which lead NTU_G values for beds of 2 and 4 elements that fall below 0.05. These errors are cumulated when removing the entrance effect.

Brunazzi and Paglianti (1997) investigated bed height effects on $k_L a_e$ measurements of structured packings, of which M250Y, in a 100 mm diameter column. They observed significant reduction in mass transfer rate as the bed height was increased from 2 to 9 elements. The variation was associated with rivulet flow and incomplete mixing at the packing junction points through the Graez number. However, from the perspective of this work, properly removing wall flow and accounting for end effects should eliminate bed height effect. Therefore the theory of incomplete mixing does not seem appropriate for this packing.

(a) (b)

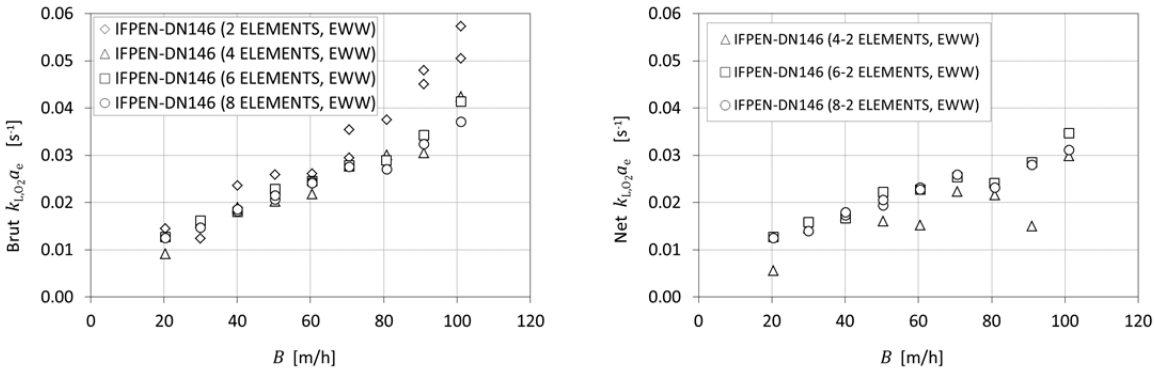

Figure 5. $k_L a_e$ measured with extra wall wipers (EWW) for different bed heights in the IFPEN-DN146 column at F_s=0.65 $\sqrt{Pa}$ (a) brut values and (b) net values.

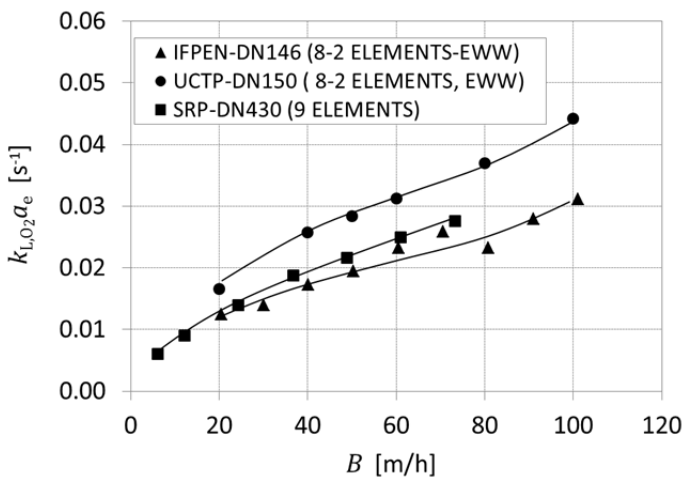

Figure 6: Comparison of $k_L a_e$ measured in different facilities with M250Y packing after consideration for wall flow with extra wall wipers in the small diameter columns.

The brut $k_L a_e$ are compared in Figure 6 to those measured by Wang (2015) with a 1.87 m bed height in a 430 mm diameter column (hereafter SRP-DN430). At this size the wall effects are minor and no extra wall wipers were used. The bed height corresponds to 9 elements of packing so entrance effects, which were not evaluated by Wang, could account for up to 15 % of the reported value based on this work. The values of Wang were obtained by toluene stripping at a gas factor of F_s = 1 √Pa and were corrected back to their equivalent value for oxygen at a temperature of 20 °C and F_s = 0.65 √Pa. This last correction was performed by assuming the independence of k_L to the gas flow and using the reported effective area measurements. The $k_L a_e$ values are different for each column. Since wall effects have been removed the observed differences are mainly due facilities and methodologies and re-inforce the need for standardizing protocols for mass transfer parameter evaluations.

4. Conclusions

The use of appropriate wall wipers along with considerations for entrance effects allow to measure intrinsic height independent liquid side mass transfer coefficients of structured packings in small diameter columns. Eliminating wall effects is seen to increase the net $k_L a_e$ by 60% for a 6 element bed (0.63 m) relative to a situation without these extra wall wipers. This suggests that height effects as characterized by Brunazzi and Paglianti (1997) could be due to incomplete mixing from wall flow instead of incomplete mixing at junction points within the packing. For larger columns, with diameters above 300mm, the wall effects become negligible. Even with high density drip point liquid distributors larger $k_L a_e$ are measured at the packing top elements. As a consequence, for bed heights of less than about 1.7 m end effects can contribute up to 20% of the measured $k_L a_e$. Despite taking into account these end and wall characteristics, different facilities using also different chemical systems show values that diverge by 30%, suggesting that standardization may be required to further improve measurement of mass transfer parameters from different laboratories.

References

Brunazzi E. and Paglianti A., 1997, Liquid-Film Mass-Transfer Coefficient in a Column Equipped with Structured Packings, Ind. Eng. Chem. Res., 36, 3792-3799.

Danckwerts P. V. and Sharma M. M., 1966, The absorption of carbon dioxide into solutions of alkalis and amines (with some notes on hydrogen sulphide and carbonyl sulphide), Chem. Eng., Oct. 1966, 244.

Hegely L., Roesler J., Alix P., Rouzineau D. and Meyer M., 2017, Absorption Methods for the Determination of Mass Transfer Parameters of Packing Internals: A Literature Review, AIChE J., 63(8), 3246–3275.

Rejl F.J., Haidl J., Valenz L., Marchi A., Moucha T., Petříček, Brunazzi E., 2017, Liquid-phase mass-transfer coefficients of Mellapak structured packings under desorption of oxygen from primary alcohols, Chem. Eng. Res. Des. 127, 1-9.

Roesler J., Royon-Lebeaud A. and Alix P., 2016, Liquid side mass transfer coefficient measurements in tall structured packing beds by reactive absorption with dilute MDEA solutions. AIChE Annual Meeting, San Francisco, CA, USA, November 13-18, Paper n° 8467941.

Valenz L., Rejl F. J., Sima J. and Linek V., 2011, Absorption Mass-Transfer Characteristics of Mellapak Packings Series, *Ind.Eng.Chem.Res.*, 50, 12134–12142.

Wang C., 2015, Mass Transfer Coefficients and Effective Area of Packings, PhD thesis, University of Texas at Austin, Austin, USA.