LIQUID LOAD POINT DETERMINATION IN A REACTIVE DISTILLATION PACKING BY X-RAY TOMOGRAPHY

S. Aferka[a], A. Viva[a,b], E. Brunazzi[b], P. Marchot[a] , M. Crine[a], D. Toye[a]

[a] *Laboratory of Chemical Engineering, University of Liège, Sart-Tilman, B6, B4000 Liège, Belgium,* *s.aferka@ulg.ac.be*

[b] *Department of Chemical Engineering, University of Pisa, Via Diotisalvi 2, I-56126 Pisa, Italy*

Abstract: In this paper, we report on the use of X-ray tomography to determine the liquid load point in 0.1 m diameter modular catalytic distillation packings Katapak-SP11 and Katapak-SP12. The liquid load point corresponds to the overall packed bed liquid load above which there is an increment in the liquid flowing outside the catalytic baskets and the catalytic baskets themselves are saturated with liquid. From tomographic images, we show that several factors affect the wetting and filling of catalytic baskets. The complex hybrid structure of catalytic packings influences the liquid distribution inside the elements. The liquid preferentially fills the external catalytic baskets because they receive the liquid not only from the packing element situated above but also from the wall wipers. Moreover, liquid hold-up inside a catalytic basket section depends significantly on the vertical position in the packing element and on the position of the packing in the column packed bed. The counter-current gas flow speeds up the process of liquid filling of the baskets, also for low liquid loads. The non uniform distribution of liquid in catalytic basket which is observed experimentally makes the identification of a unique liquid load point not straightforward.

Keywords: X-ray tomography, Modular Packing, Hydrodynamics, Liquid load point.

1. INTRODUCTION

Reactive distillation is an example of process intensification that combines separation and chemical operations into one single apparatus. Such a hybrid process offers the potential for large savings of capital, energy and materials as well as a significant reduction of waste products.

The design of reactive distillation columns, especially for heterogeneously catalysed processes, is far more complex than that of separated conventional distillation columns and chemical reactors. The coupling of separation and

reaction zones leads to complex interactions between hydrodynamics, vapour-liquid mass transfer, internal diffusion and chemical kinetics. Various catalyst packing configurations based on a regular alternation of zones with weak permeability (catalytic zones) and of high permeability (separation zones) have been reported in the literature.

In Katapak-SP which is a catalytic distillation packing manufactured by Sulzer Chemtech, the solid catalyst particles are maintained in baskets constituted by metallic wire gauze, separated by corrugated sheets of metallic structured packing. The number of corrugated sheets between catalytic baskets may be varied allowing to adjust to the particular catalytic process.

In these hybrid structures, the liquid flows on the corrugated sheets and through the catalytic baskets, by partially or fully wetting the catalytic fixed bed. In catalytic baskets there is no gas or vapor flow. Gas-liquid interactions only occur outside the catalytic baskets. A controlled filling of catalytic baskets by flowing liquid can be used to improve the reaction side of integrated reactive separation processes. In this way, all the wetted catalyst becomes accessible to the liquid for reaction, while stagnant zones which reduce the efficiency of the catalytic bed are avoided (Moritz and Hasse, 1999; Hoffmann et al., 2004; Olujic et al., 2009; Viva and Brunazzi, 2009). Working at liquid load slightly above the saturation of catalytic baskets enhances also the separation performances, because the exceeding liquid pouring out from the catalytic baskets participates to vapour-liquid mass transfer as well as the liquid normally flowing through the separation zones (Behrens et al., 2007).

To take advantage of the flexibility of modular packings as Katapak-SP, a deep understanding of the multiphase flow phenomena imposed by the hybrid structure is required. Numerous hydrodynamic studies have been reported in literature during the last ten years. Except for Behrens et al. (2006, 2007, 2008) who worked on an industrial scale modular catalytic packing, most published studies (Ellenberger and Krishna, 1999; Moritz and Hasse, 1999; Goetze et al., 2001; Hoffmann et al., 2004; Ratheesh and Kannan, 2004; Kolodziej et al., 2001, 2004; Brunazzi and Viva, 2006; among others) are relative to experiments conducted on small diameter columns, despite it has been shown that flow behaviour depends on the size and on the structure of packings.

Based on these experimental studies, several hydrodynamic models have been proposed. In all of them, the knowledge of liquid load point represents a crucial issue. This concept was first introduced by Moritz and Hasse (1999) while studying flow in Katapak-S elements. From the analysis of liquid residence time distribution (RTD) curves at increasing liquid loads, the authors observed a flow regime transition at a given liquid load. This was attributed to an increment of liquid flow in the separation zones as a result of the saturation of catalyst baskets. As a consequence, these authors defined the liquid load point as the overall liquid load at which the catalytic baskets get saturated by liquid. RTD measurements are often used to determine the liquid load point because the dynamic hold-up measurement is not so sensitive in this respect. RTD data relative to flow in Katapak-S are provided by Moritz and Hasse (1999), Ellenberger and Krishna (1999) and Higler et al. (1999). Noeres et al (2002), Hoffmann et al. (2004), Kolodziej et al. (2005) and Gorak et al. (2006) used this method to analyse flow in the first hybrid modular packing, Multipak (Montz). Goetze et al. (2001) and Viva and Brunazzi (2009) also used RTD measurements to determine the liquid load point for Katapak-SP in packed columns of 250 mm and 100 mm, respectively.

In order to get a better understanding of the flow behaviour in catalytic baskets, Moritz and Hasse (1999), Hoffmann et al. (2004), Ratheesh and Kannan (2004) and Behrens et al. (2007, 2008) performed experiments on single baskets of different shapes. This way, they determined the catalytic liquid load (or saturation point) which is the liquid

superficial velocity in catalytic baskets at which they become saturated by liquid. All the measured values have been compared to the prediction of a model originally proposed by Moritz and Hasse (1999) for the calculation of the catalytic load point which is based on a force balance in the catalytic bed.

The relation between the catalytic load point and the overall liquid load point is not straightforward. It strongly depends on the packing geometry and on the liquid flow distribution. An a priori prediction of the liquid load point from the knowledge of the catalytic load point may be considered only if the liquid splitting between catalytic baskets and separation zones is known. Hoffmann et al. (2004), Ratheesh and Kannan (2004), Behrens (2006) proposed different methods to relate the overall liquid load point to the catalytic load point, based on different assumptions.

In the present work, we propose to measure liquid distribution in a pilot scale column packed with either Katapak-SP11 or SP12 elements using X-ray tomography, in order to get a better understanding of the relationship between liquid distribution and liquid load point. The results obtained by tomography are used to complement and to explain RTD results obtained on the same catalytic packed bed.

Using X-ray tomography is justified by the fact that this non intrusive technique has been shown to be an efficient visualization tool to provide insight into vapour-liquid contacting hydraulic fundamentals in multiphase systems (Green et al., 2007; Schmidt and Eldridge, 2004; Toye et al. 2005). This technique gives access to local information as it allows to visualize solid, liquid and gas phase distributions in all column cross sections. Moreover the technique has already been successfully used to measure static hold-up in Katapak-SP 12 (Aferka et al., 2007). In the present work, it is applied to determine the overall liquid load at which the baskets reach their saturation point.

2. EXPERIMENTAL SET UP

For this work, we have used a high energy (420 kV) large scale (0.45 m diameter and 4 m high) X-ray tomograph. The generator is a Baltograph CSD450 constant potential generator (Balteau NDT, BE). It may be operated between 30 and 420 kV. The X-ray source is an oil cooled, bipolar TSD420/3 tube (Comet, CH and Balteau NDT, BE), producing a 40° aperture fan beam. Its minimum focus size is 0.8x0.8 mm following norm IEC336 - EN12543. The detector is a X-Scan 0.4f2-512-HE (Detection Technology, FI), constituted by a linear array of 1280 photodiodes each coupled with a $CdWO_4$ scintillator. A detailed description of tomographic device and mechanical equipment is provided in Toye et al. (2005).

The experimental setup is made up of a 4 m high and 0.1 m inner diameter transparent PVC column. During the tomographic measurements, the column rotates slowly around its vertical axis. The packed bed of 1.6 m height (Figure 1c) is constituted by the superposition of, from the bottom to the top, one Mellapak Plus 752Y element (M1), four Katapak-SP elements (K1…K4) and three Mellapak Plus 752Y elements (M2…M4). The catalytic bed is placed between z = 200 mm and z = 1000 mm, being z the column axial coordinate. The conventional structured packings, i.e. Mellapak Plus 752 Y, placed above and below the catalytic bed have the aim of improving the liquid and gas distribution respectively. Two different configurations of catalytic packings have been investigated in the present study: Katapak-SP11 and Katapak-SP12 which are both made of an alternation of catalytic baskets and of corrugated layers of Mellapak Plus 752Y. The difference between Katapak-SP11 and Katapak-SP12 is the ratio of

baskets per corrugated metal sheets. For Katapak-SP11, there are four baskets and one sheet between baskets (Figure 1a), while, for Katapak-SP12, there are three baskets and two sheets between baskets (Figure 1b). Each packing placed in the column is 0.10 m in diameter and 0.2 m high. The catalyst particles inside the baskets have been replaced by glass spheres of diameter equal to 1 mm. Main geometrical properties of the catalytic packing elements are summarised in Table 1 (Viva, 2008).

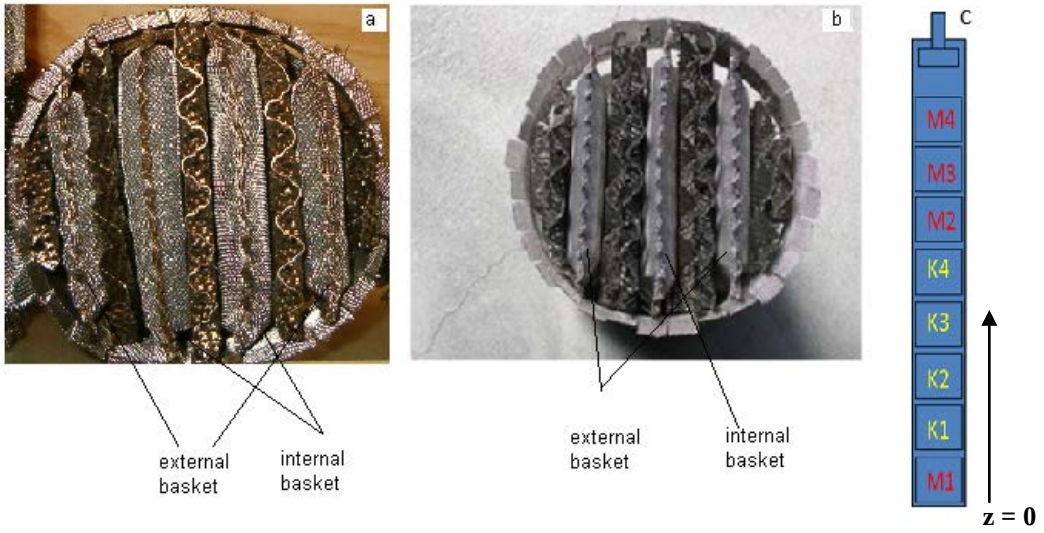


Figure 1 : Top view of Katapak-SP11 (a) and Katapak-SP12 (b). In (c) the position of the packings in the column is schematised.

Table 1: Geometrical properties of the packings used in the 100mm diameter column

	Katapak-SP11	Katapak-SP12
Packing element height (mm)	200	200
Packing element diameter (mm)	100	100
numbers of catalytic baskets	4	3
average height of catalytic baskets without seams (mm)	180	180
average thickness of catalytic baskets (mm)	13.5	13
numbers of corrugated layers	5	8
numbers of wall wipers per packing	3	3
height of wall wipers (mm)	35	35
specific geometric area (m^2/m^3)	210	282
overall packing porosity (-)	0.74	0.82
porosity of catalytic baskets, ε_{CB}, i.e. spheres bed + wire gauze (-)	0.399	0.399

The liquid distributor is a multiple point source distributor (approx. 4000 drip points/m^2). Before each measurement set, the packed column has been prewetted at high liquid load. Liquid (tap water) employed were from 4.6 to 23.1 m^3/m^2h. The gas superficial velocity ranged between 0 and 2 m/s.

3. RESULTS AND DISCUSSION

Tomographic measurements have been realised in packing cross sections situated at different heights between the top and the bottom of the packed column. Reconstructed images of these cross sections are obtained by a classical linear back projection algorithm adapted to the fan beam geometry implemented in the Fourier domain. The original methodology first proposed by Toye et al. (1996) for plastic random packings and used in subsequent works (Toye et al., 1998; Marchot et al., 2001; Aferka et al., 2007) has been adapted to analyse images of the metallic modular packings presented here. To obtain the sole liquid contribution, the projection data obtained on the dry column are subtracted, before reconstruction, from those obtained at the same height on the irrigated column. Resulting projection data are then used to reconstruct the liquid distribution image. In order to eliminate the background noise affecting reconstructed images, various numerical treatments such as masking, normalisation and thresholding are applied to raw images. To illustrate, as clearly as possible, the liquid and the solid phase distributions, we proceed as follows. First, dry packing images are recorded, reconstructed, thresholded and processed, leading to the grey part of the reconstructed image. Then, corresponding liquid distributions in irrigated cross sections are reconstructed and thresholded, leading to the blue part of the reconstructed image. Finally, grey and blue parts are superimposed (Figure 2a and 2b).

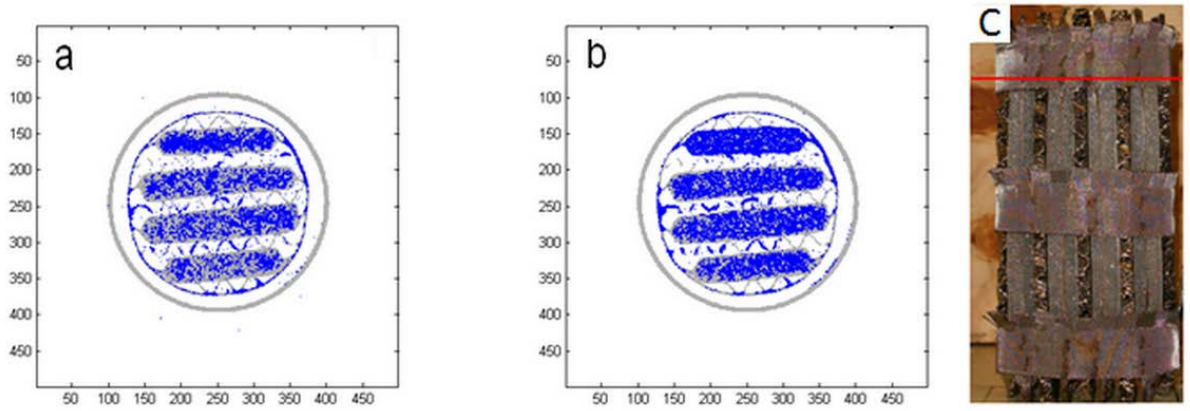


Figure 2: Reconstructed image of a cross section of Katapak-SP11 situated at a height of z=370 mm, irrigated by a liquid load of 4.62 m^3/m^2 h (a) and 12.7 m^3/m^2 h (b), and with no gas flowrate. In (c) the horizontal line shows the position of the cross section with respect to the packing element height (element K1 of Fig.1c).

Figures 2a and 2b show the liquid distribution in the same packed bed cross-section of Katapak-SP11 for two liquid load values (U_L = 4.62 m^3/m^2 h and 12.7 m^3/m^2 h). Liquid is not uniformly distributed in baskets. At high liquid

loads the baskets are progressively filled by more liquid. The same behaviour is observed in Figures 3a and 3b for Katapak-SP12. The wetting of wall wipers is clearly observed on tomographic images.

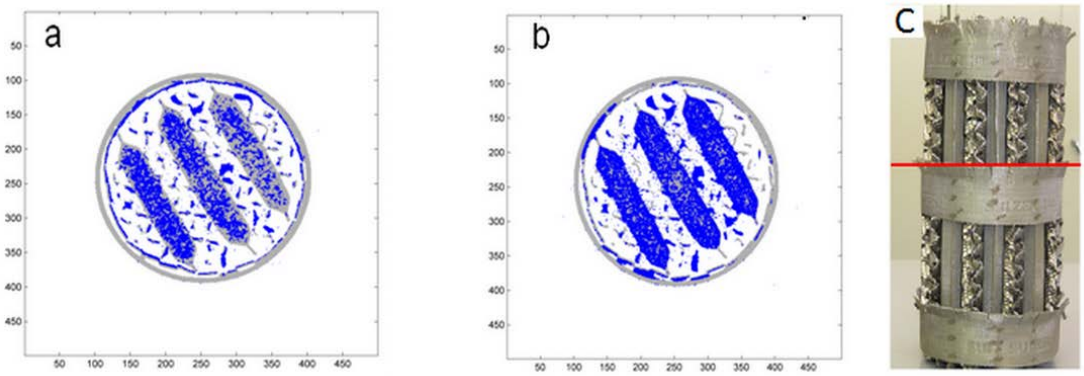


Figure 3: Reconstructed image of a cross section of Katapak-SP12 situated at a height of z=490 mm, irrigated by a liquid load of 4.62 m^3/m^2 h (a) and 12.7 m^3/m^2 h (b), and with no gas flowrate. In (c) the horizontal line shows the position of the cross section with respect to the packing element height (element K2 of Fig.1c).

Liquid hold-up in catalytic baskets obtained by tomography, hl_{CB}, is defined, in each section, as the ratio between the surface occupied by liquid inside the catalytic baskets and surface occupied by the catalytic baskets. Figure 4 shows the axial profile of liquid hold-up in external and internal baskets in Katapak-SP11 for the lowest liquid load value and without gas (U_L= 4.62 m^3/m^2 h). Vertical bars represent the junctions between the four Katapak packing elements. We can observe that there is more liquid in the external baskets than in the internal ones. This difference can be explained by the presence of wall wipers near the external baskets. The three wall wipers per packing are used to redistribute the liquid towards the inside of the packing in order to limit the flow along the column wall.

It is worth to note that, even at very low liquid loads, there is a liquid head at the bottom of each catalytic basket which corresponds to the capillary part of static liquid hold-up, as already observed by Aferka et al. (2007), Viva and Brunazzi (2007) and Behrens et al. (2008). From the hold-up profiles presented in Figure 4, one can say that baskets are not totally filled with liquid, as some liquid hold-up values in baskets are smaller than the basket porosity (equal to 0.399).

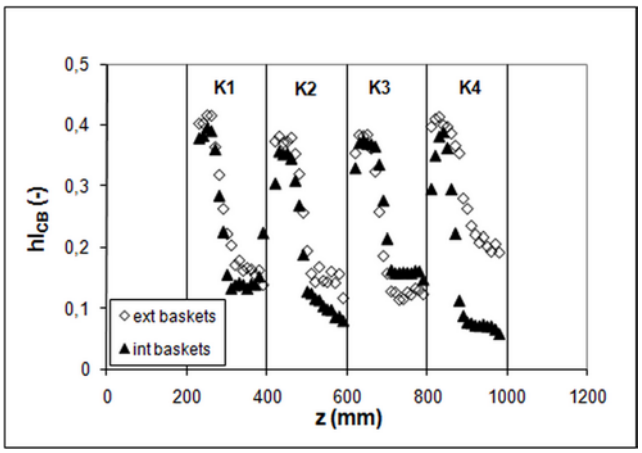
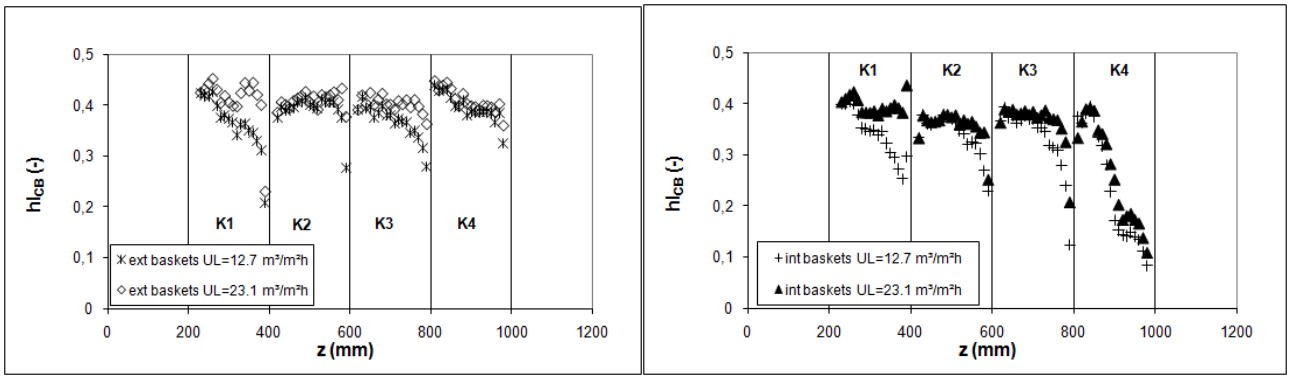


Figure 4: Liquid hold-up in the external and internal baskets of Katapak-SP11 for U_L =4.62 m^3/m^2 h, and with no gas flowrate.

The axial profile of liquid hold-up for two high liquid loads is shown in Figure 5. Liquid hold-ups in external and internal catalytic baskets are shown in Figures 5a and 5b, respectively. Hold-up values confirm that there is more liquid in external baskets than in the internal ones and that this maldistribution is observed in all Katapak-SP elements. For the Katapak-SP11 element located at the top (K4), the significant difference of hold-up values between external and internal baskets is probably due to problems of distribution from the above positioned Mellapak Plus packing.

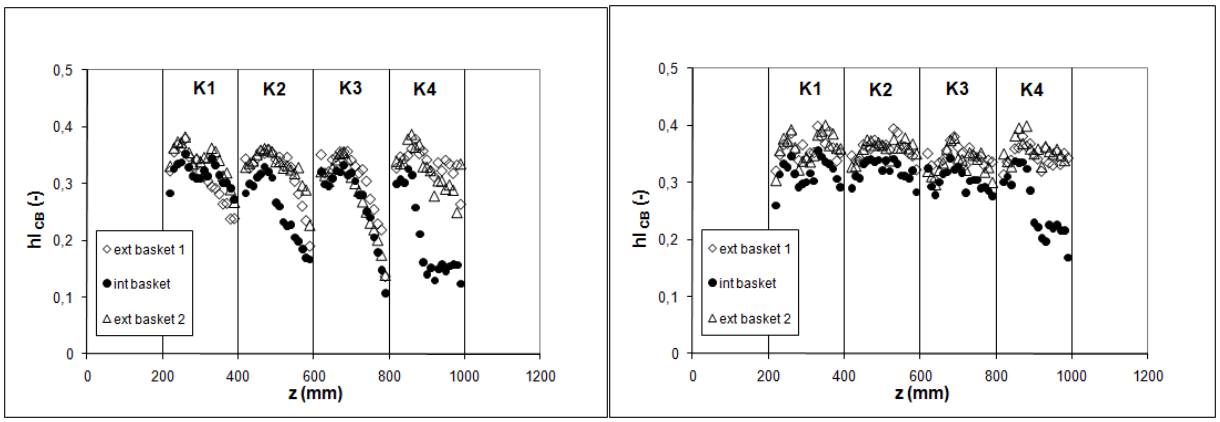
The influence of liquid load on the hold-up profile is more significant in internal baskets than in external ones, as the last get saturated for liquid loads as small as to 12.7 m³/m² h.



(a) (b)

Figure 5: Liquid hold-up in external (a) and internal (b) baskets of Katapak-SP11 for U_L =12.7 m^3/m^2h and U_L =23.1 m^3/m^2h, and with no gas flowrate.

Figures 6a and 6b show that hold-up axial profiles in Katapak-SP12 present the same behaviour as in Katapak-SP11. In fact, for a low liquid load (Figure 6a) the baskets are not saturated above all at the top of the spheres bed. Moreover the two external baskets show the same profile, due to the symmetric structure of the modular packing. At high liquid load (Figure 6b) the liquid hold-up approach the porosity value along all the catalyst baskets height.



(a) (b)

Figure 6: Liquid hold-up in external and internal baskets of Katapak-SP12 for U_L =9.24 m³/m²h (a) and U_L =23.1 m^3/m^2h (b), with no gas flowrate.

The influence of a counter-current gas flow on the liquid distribution in packed bed has been investigated for Katapak-SP11 and Katapak-SP12. Figure 7 shows the liquid hold-up in the external and internal baskets of Katapak-SP12 for the same liquid load, U_L = 9.24 m^3/m^2 h, as in Figure 6a and for a gas superficial velocity equal to 1.7 m/s. It shows that the gas flow induces an increase of liquid hold-up, due to the additional resistance it exerts on liquid descending along the walls of catalytic baskets. The counter-current gas flow thus speeds up the process of filling catalytic baskets. This affects mainly the upper part of baskets, because in lower part, liquid flow is driven by the liquid head in the basket. Figure 7 also shows that the effect of gas flow on liquid hold-up profile is more pronounced in the internal basket than in the two external ones. Even with no gas flow, the last ones are already saturated by liquid and the hold-up values approach the porosity of catalytic baskets. On the contrary, the liquid hold-up values in the internal basket measured with a counter-current gas flow (Figure 7) are higher than those reported with no gas flow (Figure 6a), but they are still far from the saturation.

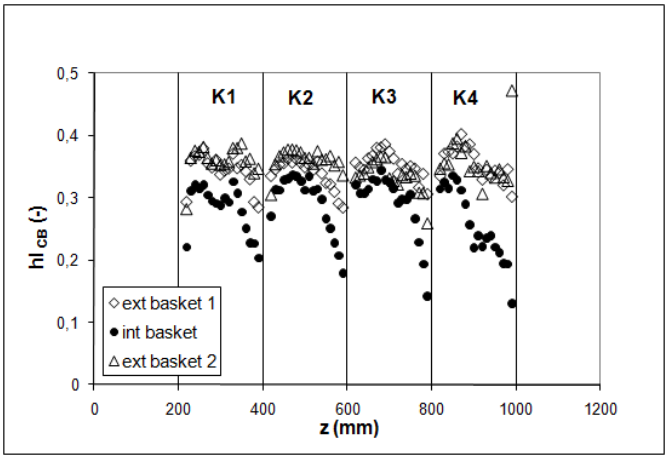


Figure 7: Liquid hold-up in the external and internal baskets of Katapak-SP12 for U_L=9.24 m^3/m^2 h and U_G=1.7 m/s.

The quantitative analysis of tomographic images has allowed to access to the liquid distribution in packing cross sections, and more particularly to the liquid distribution in catalytic baskets as a function of operating conditions. One may observe that liquid does not uniformly distribute in the packing modular structure and that catalytic baskets do not get saturated with liquid simultaneously. It is thus not straightforward to determine the liquid load point from this analysis.

Figure 8 presents the liquid hold-up in external baskets, $hl_{CB\_ext,}$ per unit of basket porosity, ε_{CB}, as a function of the overall liquid load in Katapak-SP12 and in Katapak-SP11. It may be used to determine the catalytic load point in the external baskets: as, for both packings, the ratio $hl_{CB\_ext}/\varepsilon_{CB}$ becomes almost constant for liquid loads higher than 12.7 m^3/m^2h, one may say that the external baskets get saturated and reach their catalytic load point for a liquid load equal to 12.7 m^3/m^2h.

This result may be compared to liquid load point values obtained from RTD curves analysis by Viva and Brunazzi (2009) on the very same Katapak-SP11 elements of 0.1 m nominal diameter. The RTD curves show a transition of the flow regime in the liquid range between 10 and 20 m^3/m^2h. Moreover, the liquid load point determined from tomographic images agrees also with the value approximately equal to 10 m^3/m^2h proposed by Goetze et al. (2001) for Katapak-SP12 of 0.25 m nominal size.

One may thus suppose that the saturation of external baskets by liquid induces a significant change in the liquid flow morphology, as more liquid is forced to flow in the separation zones. This flow regime transition occurs even if internal baskets are not yet saturated with liquid.

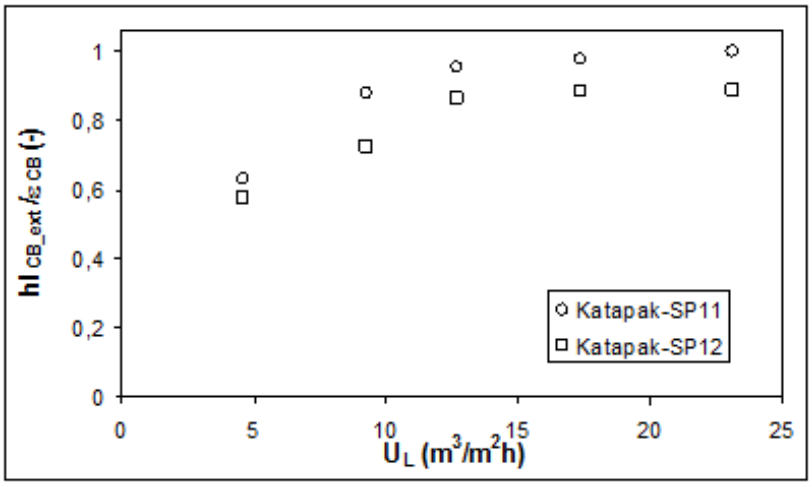


Figure 8: Increment of liquid hold-up in the external baskets, hl_{CB_ext}, over the catalytic basket porosity, ε_{CB}, as a function of the overall liquid load.

CONCLUSIONS

The present study shows that X-ray tomography allows to visualize and quantify the liquid distribution in modular metallic packings, such as Katapak-SP11 and Katapak-SP12. It is thus an appropriate technique to get a better understanding of the mechanisms responsible for the flow regime transition observed at the liquid load point in these complex packings.

From the obtained results, one can say that the liquid load point is strongly dependant on the liquid distribution inside the modular structure. Non uniformities in liquid distribution are observed along the column axis, as well as in packing cross sections. The former affects the liquid hold-up profile along the column height, while the latter leads the external baskets to be preferentially wetted and saturated by the liquid compared to the internal ones. This is due to the vicinity of the external baskets to the wall wipers which redistribute the liquid flowing along the column wall inside the packings.

A value for the liquid load point without gas can be estimated from the analysis of tomographic images, as the overall liquid load corresponding to the saturation of the external catalytic baskets with liquid. As this result agrees with experimental results obtained from RTD curves measured in a packed column filled by the very same Katapak-SP packing elements, we assume that this liquid load point corresponds to a change in liquid flow morphology, resulting in a flow regime transition in the packed bed.

A more accurate knowledge of the liquid distribution corresponding to the liquid load point could be used in models to predict fluid dynamic related quantities, such as pressure drop, mass transfer coefficients and effective interfacial area. These quantities are in fact implemented in simulation programs like Aspen with rate-based approaches to study the feasibility of an increasing number of reactive separation processes reported in the literature. Most often, these simulation models can only be validated with results obtained at a pilot scale (50 mm diameter column, sometimes 100 mm diameter column as it is very costly and complicated to run reactive separation columns at industrial scale). Meaningful fluid dynamic parameters for the same scale are thus needed. Once the simulation model is fully validated at a small scale, it can be considered as a confident tool for some reliable scale up.

But one must be aware that the behaviour observed by tomography in the present study has to be considered as strongly dependent on the structure and on the geometrical parameters of the packings under investigation. The extension of the used approach to packings of higher dimensions up to industrial scale is recommended in order to get a reliable scale up for the hydrodynamics description of these modular packings.

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