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Determination of liquid hold-up and flow distribution inside modular catalytic structured packings

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Abstract

This paper presents the results of a study carried out to examine liquid hold-up and flow distribution in a 0.1m internal diameter column filled with catalytic structured packing Katapak-SP.

Information has been gathered at local scale by means of a non-intrusive high energy X-ray tomograph. Measurements have been carried out in a large number of packing cross sections situated at different heights between the top and bottom of the packed column, giving access to the evolution of axial profiles of liquid hold-up in the open channels (separation zone) and in the catalytic baskets (reaction zone) as a function of the liquid load. The total hold-up, evaluated by averaging local tomographic values over the packed volume, was compared with global hold-up data obtained by traditional methods, like draining and RTD measurements.

A method was also proposed to deduce the distribution of liquid flowrate, between the reaction and the separation zones, from hold-up distribution measured by tomography. The methodology was validated by comparison with experimental data obtained by collecting separately the liquid flowing out of the two zones at the bottom of the packed bed. The obtained results are invaluable to improve the description of hydrodynamics in rate based performance models.

Keywords: Liquid hold-up; Catalytic structured packing; Katapak-SP; X-ray tomography; Flow distribution;

1. Introduction

Catalytic structured packings are used in an increasing number of reactive separation processes. The commercially available Sulzer Katapak-SP packing, under study in this work, is characterized by a hybrid structure made of corrugated sheets of conventional distillation layers (separation elements) and catalytic baskets, i.e. wire gauze envelopes filled with catalyst particles (reaction elements), assembled in alternate sequence. The modular design allows varying the relative size of the separation and reaction zones by placing one or more corrugated sheets in between. This feature provides a degree of flexibility, because it allows to arrange the packing to fit the requirements of each specific process, and makes Katapak-SP a highly competitive internal device for reactive distillation columns. The ratio between catalytic baskets and corrugated sheets (typically, 1:1, 1:2 or 1:3) identifies the packing configuration (Götze et al., 2001; Olujić and Behrens, 2006).

The hybrid structure of the catalytic structured packing determines the flows development inside the packed bed and consequently the overall column performances. The mesh size of the wire gauze envelopes ensures easy access of liquid, prevents the particles cross-over and makes the baskets practically impermeable to the gas flow. While the hold-up and the superficial velocity of liquid inside the catalytic baskets influence the reactive performance of the packing, the hold-up and the superficial velocity of liquid on the corrugated sheets are mainly responsible for the interactions with gas. Hence, knowledge of the distributions of liquid hold-up and of liquid superficial velocity between these two zones is very important for the prediction of fluid dynamic related parameters, such as pressure drop, capacity, interfacial area and mass transfer volumetric coefficients. Moreover, the dependence of these distributions as a function of packing design and of operating parameters must be taken into account for the development of accurate predictive models.

In catalytic structured packings both the static and dynamic liquid hold-up contribute significantly to the total liquid hold-up (Götze et al., 2001; Gorak et al., 2006; Behrens et al., 2006). The static hold-up is the volume fraction of liquid that remains within the packed bed after complete draining and it results from the action of capillary forces that hold some liquid on the packing mainly at

contact zones between corrugated sheets, between corrugated sheets and gauze envelopes and between the particles in the catalytic zone. The dynamic (free flowing-draining) hold-up consists of the flowing liquid and strongly depends on the liquid load. Both corrugated sheets and catalytic baskets contribute to the static and dynamic liquid hold-ups. Draining or volumetric methods and tracer based measurement techniques are commonly used to study liquid hold-up and liquid flow behaviour, and provide hydrodynamic parameters referred to the whole packed bed volume, i.e. global values. Dynamic hold-up measurements have been reported for Katapak-SP 12 of different nominal sizes, from pilot scale of 100 mm and 250 mm (Ratheesh and Kannan, 2004; Götze et al., 2001) to industrial scale of 450 mm (Behrens et al., 2006). As far as Katapak-SP 11 is concerned, dynamic hold-up data have been measured on packings of diameter equal to 50 mm and 100 mm (Brunazzi and Viva, 2006; Viva and Brunazzi, 2007) and on the industrial packing size of 450 mm (Behrens et al., 2006). The static hold-up contribution can be mainly attributed to the catalytic baskets. The drainage of single catalytic baskets taken from dismantled packings have been investigated (Viva and Brunazzi, 2007; Behrens et al., 2007, 2008). These studies are complementary to the measurements of the static hold-up on packing elements of Katapak-SP 11 and Katapak-SP 12 (Viva and Brunazzi, 2007; Aferka et al., 2007).

The present work first shows local and global values of liquid hold-up obtained by different techniques. It is worth to emphasize that the very same packing elements and prewetting procedures were used. Two modular configurations of Katapak-SP, i.e. 1:1 and 1:2, have been investigated and the experiments have been carried out at room temperature and atmospheric pressure using water as liquid phase. The catalytic baskets were filled with glass spheres of 1 mm of diameter.

In particular, local information on liquid hold-up inside Katapak-SP 11 and Katapak-SP 12 has been determined qualitatively and quantitatively by means of X-ray tomographic measurements. X-ray tomography is a non invasive technique capable of offering local information on the liquid hold-up distribution in the inhomogeneous packing structure (Aferka et al., 2007; Toye et al., 2005; Aferka et al., 2010). The technique consists in the acquisition, reconstruction and processing of packing

cross section images. Spatial variations of the liquid distribution inside the constitutive elements can be detected on irrigated packing. Quantitative information on liquid hold-up is obtainable from tomographic images when the appropriate image processing methodology is applied. Using X-ray tomography has been shown to be an efficient visualisation tool to provide insight into vapour-liquid contacting hydraulic fundamentals in multiphase systems (Schmit and Eldrige, 2004; Green et al., 2007). The total hold-up, evaluated by averaging local tomographic values over the packed volume, was then compared with global hold-up data obtained by traditional methods, like draining and RTD measurements confirming the soundness of the tomographic image processing and the consistency of the different techniques.

But the tomographic information on liquid hold-up distribution is not enough to predict performances of catalytic distillation devices. Indeed, performance of modular catalytic packing such as Katapak SP in terms of reaction as well as in terms of separation depends on the residence time of fluids in the catalytic and in the separation zones. So, logically, models which allow predicting product distribution as a function of operating conditions require not only the knowledge of the distribution of liquid hold-up in the separation and in the reaction zones of the packing but also the knowledge of the distribution of the liquid flowrate between the two zones (Hoffmann et al., 2004). This information is not directly accessible from tomographic images, but some authors have already proposed a method to deduce the distribution of liquid superficial velocity from hold-up distribution measured by tomography in classical, non modular packings (Toye et al., 1999; Boyer and Fanget, 2002). In this paper, we propose a similar methodology to compute the distribution of liquid flowrate between the reaction and the separation zones in modular catalytic distillation packing. In this method we use the well-established hydrodynamic correlation proposed by Suess and Spiegel (1992) to compute the liquid flowrate from hold-up values measured in the separation zone. The liquid flowrate in the catalytic zone is then evaluated by difference between the total flowrate and the flowrate of the liquid in the separation zone. The global methodology is

then validated by comparison with experimental data obtained by collecting separately the liquid flowing out from the two zones at the bottom of the bed.

2. Measurements and methods

2.1 Catalytic packing

In the present work, Katapak-SP configurations 1:1 and 1:2 (top view in Figure 1a and side view in Figure 1b, respectively) are investigated in the nominal size of 100 mm diameter and 200 mm height. Corrugated sheets are those encountered in MellapakPlus 752.Y, a large specific geometric area and high performance structured packing (Olujic et al. 2009; Aferka et al., 2011). Glass particles of 1 mm diameter fill the catalytic baskets for a bed height equal to h_{CB} (Figure 1c). Three wire gauze wall wipers (Figure 1b) for each packing hold together the modular structure and pour back into the packing the liquid which may flow along the column wall. Packings are arranged in the column rotated of 90° each others (Figure 1d).

Following the nomenclature introduced by Hoffmann et al. (2004) for Multipak, the predecessor of Katapak-SP, and adopted for Katapak-SP by different authors (Ratheesh and Kannan, 2004; Behrens et al. 2006; Olujic and Behrens 2006) the open channels, OC, correspond to the separation zones where the corrugated sheets are placed, outside the catalytic baskets, CB, and excluding the wall wipers circular section, WW.

Figure 1.

Katapak-SP geometrical parameters are strongly dependent on the packing size and configuration. An accurate geometrical characterisation includes the common parameters, i.e. specific area and void fraction, but also the overall dimensions of the modular elements. To evaluate the geometrical parameters, we have measured all the elements lengths on dismantled packings and calculated the

corresponding fractions. We refer to the effective diameter, L_{\max} , as to the length of the symmetric element in the packing, which is a corrugated sheet in Katapak-SP 11 (Figure 1a) and a catalytic basket in Katapak-SP 12 (Figure 1b). Table 1 summarizes the effective values obtained by geometrical characterisation. It is noteworthy that elements in the packing may occupy a surface fraction (i.e. ratio of the element section surface on the column section surface) which differs from the volumetric fraction (i.e. ratio between the element volume and the packing volume), as for example in the case of catalytic baskets and open channels. This difference is taken into account further on. Indeed, the global liquid hold-up analysis is based on the knowledge of volumetric fractions, whereas the surface fractions are needed to determine the superficial velocities of fluids in the different zones and also to interpret the tomographic measurements which are made on column cross-sections along the packing height. The void fraction (ϵ), as well as the void fraction of catalytic baskets with (ϵ_{CB}) and without (ϵ_{PB}) wire gauze envelope, have been measured by comparing the volumes of a measurement chamber filled with water before and after the immersion of the packing and of the catalytic basket, respectively.

Table 1

2.2 X-ray tomographic measurements

The X-ray computed tomography facility available at the Laboratory of Chemical Engineering of the University of Liège (Belgium) was used to measure liquid hold-up distribution at local scale as a function of liquid load. The facility is a high energy (420 kV) X-ray tomograph equipped with a fan beam X-ray source and with a 1280 photodiodes linear detector which are both fixed on an arm able to translate vertically along the scanned object height (Figure 2a). Scanned objects are put on a rotating plate which may perform a complete revolution around a vertical axis. Objects with diameters up to 0.45 m diameter and with height up to 4 m may be analyzed with a spatial

resolution equal to 0.36 mm. More details are provided in Toye et al. (2005). This X-ray CT may work in radiographic mode as well as in tomographic mode. When used in radiographic mode, the rotating plate of the scanner is deactivated and the scanned object remains fixed. 2D radiographic images correspond to X-ray attenuation horizontal profiles measured at different heights by vertically translating the arm. In tomographic mode, the vertical arm remains fixed while the scanned object is rotated in order to get attenuation profiles for all angular positions. From these attenuation data, one may obtain the image of the column cross-section corresponding to the vertical position of the arm. Images of cross sections situated at different heights may be obtained by repeating the measurement procedure for different positions of the arm supporting the X-ray source and the detector. X-ray tomography is thus a time consuming measurement technique as, for each operating condition, a large number of cross section images have to be reconstructed to get information on phase distributions relative to the whole bed.

In the set of measurements described in this work, four elements of catalytic packing (i.e. from the bottom to the top K1, K2, K3, K4) were stacked into a 0.1m internal diameter column, rotated of 90° each other. Four elements of conventional corrugated packing were also placed to improve the fluid distribution, three above and one below the catalytic bed. With reference to the packed column radiogram reported in Figure 2b, the catalytic bed is placed between $z=200$ mm and $z=1000$ mm, being z the column axial coordinate.

Figure 2.

Tomographic measurements have been performed in packing cross sections (77 for Katapak-SP 11 and 78 for Katapak-SP 12) situated at different heights between the top and the bottom of the column catalytic bed. Preliminary measurements have been carried out at dry condition (no liquid) and after complete filling with liquid in order to determine the attenuation factors corresponding to solid, gas and liquid phases. Afterwards, the column has been irrigated, by varying the liquid

flowrate. In the present paper, we show results with water as working liquid and liquid loads up to $25 \text{ m}^3/\text{m}^2/\text{h}$. Throughout the paper, liquid loads are defined as liquid volumetric flowrates per unit area of column cross section A_c .

A classical linear back-projection algorithm adapted to the fan beam geometry (Kak and Slaney, 1988) and implemented in the Fourier domain is used to reconstruct the solid and the liquid distributions inside the object for each section starting from the local X-ray attenuation values. Reconstructed images are squares of 512×512 pixels dimension and the spatial resolution in horizontal planes is around 0.36 mm. Images have to be post-processed in order to obtain quantitative values. The original methodology first proposed by Toye et al. (1996) for plastic random packings and used in subsequent works (Marchot et al. 2001, Toye et al. 1998) has been adapted to analyse images of metallic modular packings and can be summarised in four main actions:

1. thresholding of the images for the elimination of noises and artifacts;
2. drawing masks around the catalytic baskets, including the wire gauze envelopes, to separate the analysis on liquid hold-up between the separation and reactive domains;
3. reduction of corrugated sheets to their skeleton in order to overcome the instrument resolution;
4. normalisation of the attenuation values measured in each pixel, according to normalisation factors which correspond to pixels filled with gas, liquid or solid phases.

More details on the validated procedure for the post-processing of dry and irrigated Katapak-SP images are provided in Viva et al. (2011). In particular, post-processing of dry packing images has also provided information about void fraction and specific area on a number of different cross sections along the packed column. The averaged tomographic volumetric values are reported in Table 1 for comparison.

2.3 Conventional measurements

Packed bed scale, i.e. global values of liquid hold-up measured with conventional methods were used to validate the results obtained by tomography. The specifications concerning the hold-up gravimetric measurements carried out in a column filled with the same Katapak-SP 11 and Katapak-SP 12 packings are provided elsewhere (Viva and Brunazzi 2007, 2009; Brunazzi and Viva, 2006). It is worthy to note that measurements were repeated several times on different packings in order to assess the reproducibility of the measurements and that a standardized procedure was set and followed to assure the consistency of the results. In particular, a standard draining time of 1 h was set for the measurement of dynamic hold-up draining from the packed column and for the estimation of the static hold-up on single packings. Results were found (Viva and Brunazzi, 2009) in good agreement with the analysis at infinite draining time, which was first proposed by Urrutia et al. (1996) for trickle beds. Dynamic hold-up measurements were conducted with water loads up to $30 \text{ m}^3/\text{m}^2/\text{h}$, in a column filled with 10 Katapak-SP 11 packings, and $26 \text{ m}^3/\text{m}^2/\text{h}$, in a column filled with 5 Katapak-SP 12 packings.

For Katapak-SP 11 additional experiments were carried out to determine the liquid-phase residence time distribution (RTD) and the distribution of liquid flowrate between the open channels and the catalyst baskets as a function of liquid load. Procedures and results of RTD estimation by means of tracer experiments are described in Viva and Brunazzi (2009). The obtained RTD hold-up values agree with the total hold-up values given by the sum of static and dynamic contributions derived from draining experiments, for the whole range of liquid loads up to $30 \text{ m}^3/\text{m}^2/\text{h}$, thus confirming the consistency of the different techniques.

The liquid flow splitting has been determined using the 0.1m internal diameter column filled by 10 Katapak-SP 11 packings, by means of an experimental arrangement similar to the one suggested by Ratheesh and Kannan (2004). The experimental arrangement is sketched in Figure 3. The column was first pre-wetted at high liquid load and afterwards the liquid load was set at the desired value and maintained to reach the stationary flow regime inside the column. The liquid flowing in

catalytic baskets was collected from the packing at the bottom of the column in *ad hoc* designed boxes. The flow was discharged in a separate tank and the liquid flowrate was estimated on the basis of the temporal variation of the liquid level measured with a differential pressure transmitter. These collecting experiments were limited to low and medium liquid flowrates. As a matter of fact, at high liquid loads the contribution of the liquid film flowing outside the wire gauze and pouring inside the collecting boxes was not negligible and affected the measured splitting factor.

Figure 3.

3. Experimental liquid hold-up distribution results

3.1 Qualitative results from X-ray tomography

X-ray tomography detects the liquid distribution in the packings at different positions along the column height. Figure 4a shows a zoom on the radiogram reported in Figure 2b, where five sections are evidenced. Figures 4b,c,d,e,f show, for a liquid load of $5 \text{ m}^3/\text{m}^2/\text{h}$, the variation in these sections of the liquid morphology (in blue) on the solid structure images (in gray). In these figures, axes are given in pixels. One pixel has a length equal to 0.36 mm.

Figure 4.

At such low liquid load, we can observe that catalytic baskets at the top of packing K1 are not completely filled (Fig. 4b), while some liquid wets the corrugated sheets and the wall wiper. On the contrary, the liquid fills the catalytic baskets at the bottom of K2 (Fig 4c) but, moving upward (Fig 4d and 4e), the catalytic baskets get again progressively empty. Some wet spots are visible in Figures 4d and 4e, where the wall wipers touch the corrugated sheets and the liquid accumulates. In

Figure 4f, the liquid wets uniformly both the corrugated sheets and the seams closing the catalytic baskets.

Liquid distribution clearly changes by increasing the liquid load, as reported in Figures 5a-e for the section corresponding to $z=370$ mm. The liquid fills the catalytic baskets, up to the top of each packing, when the liquid load is above the so called liquid load point (Aferka et al. 2010). The latter occurs for Katapak-SP 11 of 100 mm diameter, above $15 \text{ m}^3/\text{m}^2/\text{h}$.

Figure 5.

3.2. Quantitative hold-up results from X-ray tomography

The observed qualitative results correspond to quantitative hold-up values, after the proper normalisation of liquid attenuation values during the image post-processing.

In each cross section, the catalytic basket contribution, hl_{CB} , is defined as the ratio between the surface occupied by the liquid inside the masks and the surface of the masks section. The open channel hold-up, hl_{OC} , is expressed as the ratio between the surface occupied by the liquid outside the catalytic baskets and the surface of the column section.

The superficial fraction occupied by CB masks on the column section, ψ_{CB} , varies along the column height and is determined from dry images (Figure 6a). The profile is quite constant where the catalytic baskets are fully developed, while the low values correspond to sections where the baskets approach the seams. The average tomographic value is 0.496, which is in good agreement with the value of 0.465 determined by geometrical characterisation (see $\psi_{CB,S}$ in Tab 1).

In Figure 6b and 6c, the local values of hl_{CB} and hl_{OC} are reported for low ($5 \text{ m}^3/\text{m}^2/\text{h}$), medium ($14 \text{ m}^3/\text{m}^2/\text{h}$) and high ($25.5 \text{ m}^3/\text{m}^2/\text{h}$) liquid loads.

Figure 6.

At low liquid load, hl_{CB} is not uniform along the packing height but it shows the same trend for the four packing elements. We can distinguish the liquid head at the bottom of catalytic baskets shown qualitatively in Figures 4c and 4d. Locally the catalytic baskets become almost saturated with liquid and hence hl_{CB} approaches the porosity value. The liquid head resembles the static capillary height observed on catalytic baskets of Katapak-SP 12 packing by Aferka et al. (2007) during static liquid hold-up measurement with X-ray tomography. hl_{OC} at low liquid load has instead a quite uniform profile, around a value of 0.017, with a slight increment at the transitions where the whole section liquid hold-up is only due to corrugated sheets and seams.

By increasing the liquid load at medium and high values, we observe the expected increment in both hl_{CB} and hl_{OC} contributions. The fraction outside the catalytic baskets shows pronounced peaks in the sections where the wall wipers are placed, because the liquid flowing along the column wall is collected and redistributed inside the packings.

The hl_{CB} values remain unvaried at the bottom of the baskets, while they increase in the sections above the capillary height due to a dynamic contribution dependent on the liquid load. At high liquid loads, the saturation of the catalytic baskets is reached and the nominal porosity value, equal to 0.399, is approached. The K4 packing shows a somewhat different behaviour with respect to the other three catalytic packings. This can be due to the altered liquid draining and distribution from the MellapakPlus packing above.

By adding up the local values of CB and OC hold-up contributions for each liquid load, the total hold-up can be obtained at each section, according to the following relation:

$$hl_{TOT}(z) = hl_{CB}(z) \cdot \psi_{CB-S}(z) + hl_{OC}(z) \quad (1)$$

The local total hold-up is therefore defined as the surface occupied by the liquid on the surface of the column section.

Figure 7a presents the total hold-up results obtained on Katapak-SP 11 packings. It can be observed that hl_{TOT} follows the trend of the main contribution, which is the catalytic baskets hold-up.

Figure 7.

The same measurements have been carried out on Katapak-SP 12 packings and Figure 7b shows total hold-up profiles similar to those obtained on Katapak-SP 11 for the same three liquid flowrates. As expected, the total hold-up values for Katapak-SP 12 are lower than those obtained on Katapak-SP 11, since the contribution of catalytic baskets is lower.

Global values of liquid hold-up may be computed by averaging local values relative to cross sections over the column height packed with the catalytic bed. This way, the global values are defined as volume of liquid per unit volume of column occupied by the catalytic bed. The averaged results are reported in Figures 8a and 8b as a function of liquid load for Katapak-SP 11 and Katapak-SP 12, respectively.

Figure 8.

Dynamic hold-up data, measured on the same packings by means of conventional draining measurements, are reported in Figures 8a and 8b for comparison, as well as the total hold-up values given by the sum of dynamic and static contributions. For Katapak-SP 11, also the total hold-up derived from RTD measurements is shown for comparison. A very good agreement can be observed, for both Katapak-SP 11 and Katapak-SP 12, and effectively validates the post-processing methodology of tomographic images and the calculation of local liquid hold-up. Of course, the comparison between the used techniques highlights their different advantages. Conventional measurements are characterized by simplicity in the procedures and in the required equipment, hence results can be obtained in short time but limited to global scale. Tomography is, on the contrary, a complex and very time consuming technique (during the measurements, the reconstruction and the post-processing analysis) but it allows to see inside the packed bed and to

provide invaluable quantitative local information on the liquid hold-up distribution in the packing elements and along the column height.

The collected experimental information have been used to determine whether liquid hold-up in Katapak-SP packings can be described by using correlations proposed in the literature for corrugated sheets and single-phase trickle beds, with the final aim of estimating the liquid flowrates distribution. This approach is described in the following section.

4. Liquid flowrates determination

Local experimental results have shown the evolution in the liquid hold-up distribution by increasing the liquid loads. Liquid hold-up correlations proposed for corrugated structured packings and single-phase trickle beds have been adapted to the Katapak-SP geometry and used to relate the measured liquid hold-up with the liquid flowrates outside and inside the catalytic baskets, respectively. Similarly to previous studies on catalytic packings (Moritz and Hasse, 1999; Hoffmann et al., 2004; Ratheesh and Kannan, 2004; Behrens et al., 2006, 2008), the present analysis has been differently approached for high and low liquid loads.

The starting point of the analysis is to write the total liquid flowrate as the sum of two contributions:

$$Q_L = u_{TOT} \cdot A_C = Q_{L,OC} + Q_{L,CB} = u_{OC} \cdot A_{OC} + u_{CB} \cdot A_{CB} \quad (2)$$

Where $Q_{L,OC}$ and $Q_{L,CB}$ stand for the amount of liquid flowing outside and inside the catalytic baskets, respectively. In terms of superficial fractions occupied by the modular elements on the column cross section the above equation becomes:

$$u_{TOT} = u_{OC} \cdot \psi_{OC_S} + u_{CB} \cdot \psi_{CB_S} \quad (3)$$

4.1 High liquid loads

At high liquid loads, the catalytic baskets get completely filled with liquid.

According to the approach firstly suggested by Moritz and Hasse (1999), the maximum liquid superficial velocity inside the catalytic basket, u_{CB_max} , can be determined on the basis of a balance between gravity and the resistance for liquid flow through the bed of spheres:

$$\rho_L \cdot g \cdot \delta = \Psi \cdot \frac{1 - \varepsilon_{CB}}{\varepsilon_{CB}^3} \cdot \rho_L \cdot u_{CB_max}^2 \cdot \frac{1}{dp} \quad (4)$$

Where the friction factor is expressed as:

$$\Psi = \frac{160}{Re_{CB_max}} + \frac{3.1}{Re_{CB_max}^{0.1}} \quad (5)$$

and the effective Reynolds number in the bed of spheres is:

$$Re_{CB_max} = \frac{u_{CB_max} \cdot \rho_L \cdot dp}{(1 - \varepsilon_{CB}) \cdot \mu_L} \quad (6)$$

We assumed that the contribution of the wire gauze envelope is negligible for the definition of the equivalent diameter which has been taken equal to the spheres diameter, while we used the catalytic baskets porosity for the determination of the liquid flowrate. It is also worth to note that in the used Reynolds interval, the friction factors calculated by eq. 5 differ less than 1% from the values calculated by using the relation recently proposed by Behrens et al. (2008).

Once u_{CB_max} is known, the liquid flowrate flowing on the corrugated sheets can be determined by means of the material balance expressed by Equation 3.

At high liquid loads, the maximum liquid hold-up inside the catalytic baskets corresponds to their porosity, ε_{CB} , and the contribution attributable to the corrugated sheets can be easily determined from experimental results by considering the volumetric geometrical parameters as follows:

$$hl_{OC_exp} = hl_{TOT} - hl_{CB_max} \cdot \psi_{CB_V} = hl_{TOT} - \varepsilon_{CB} \cdot \psi_{CB_V} \quad (7)$$

where hl_{TOT} are the values reported in Figure 8, obtained by tomography, RTD or gravimetric measurements.

As reported by Olujić et al. (2007) with experiments on packings of diameter equal to 450 mm and by Aferka et al. (2011) with tomographic experiments on packings of diameter of 100 mm, the liquid hold-up on the corrugated sheets of MellapakPlus 752.Y packings can be well predicted as a function of the liquid load by using the correlation developed by Süss and Spiegel (1992) for conventional Mellapak packings.

To make use of the Süss-Spiegel correlation in the present case, it has been adapted to Katapak-SP geometry according to the following relation:

$$hl_{OC_SS} = c \cdot a_{MP}^{0.83} \cdot u_{OC}^x \cdot \Psi_{OC_V} \quad (8)$$

The empirical constants in the Equation 8 depend on the liquid velocity. According to Süss and Spiegel (1992), $c = 0.0169$ and $x = 0.37$ for $u_{OC} < 40 \text{ m}^3/\text{m}^2/\text{h}$ while $c = 0.0075$ and $x = 0.59$ for $u_{OC} \geq 40 \text{ m}^3/\text{m}^2/\text{h}$. Figure 9 compares the hl_{OC_SS} predicted values and the hl_{OC_exp} experimental results, as a function of the liquid load in the high flowrates range. The agreement is very good, both for Katapak-SP 11 and Katapak-SP 12, and validates the suitability of the adapted correlation for the prediction of liquid hold-up on the corrugated sheets, i.e. in the separation zone of the catalytic structured packing.

Figure 9.

4.2 Low liquid loads

At low liquid load, assumptions on the hydrodynamic inside the catalytic baskets can not be set *a priori*. Nevertheless, tomography provides information on local liquid hold-up outside the catalytic baskets, i.e. hl_{OC} . These values can be used to back-determine the liquid flowrates on the corrugated sheets by means of the adapted correlation of Süss and Spiegel (Eq. 8), where Ψ_{OC_S} and local u_{OC} have to be used now. The local flowrates on the corrugated sheets have been determined only in the sections without wall wipers. Figure 10a shows the profile of the superficial liquid velocity in the

open channel, u_{OC} , along the column height for Katapak-SP 11 measurements. By means of the material balance (Eq. 3), the liquid flowrate inside the catalytic baskets, $Q_{L,CB}$, has been calculated for each section and the resulting liquid splitting factor, defined as the ratio between $Q_{L,CB}$ and Q_L , is reported in Figure 10b.

From the analysis of the profiles, we can argue that at low and medium liquid flowrates, capillary effects draw the liquid into the catalytic baskets at the top, where they are not completely filled with liquid. This locally reduces u_{OC} and increases the splitting factor. In the lower part of the packing, baskets are filled with liquid, hence the excess liquid flows outside and an increment in u_{OC} is observed as well as the corresponding reduction of the splitting factor.

The averaged values of splitting factor obtained by tomography and by sample collection (described in Section 2.3) are practically the same. They both equal 0.90 independently on the liquid load.

Figure 10.

For Katapak-SP 12, collecting experiments have been carried out by Ratheesh and Kannan (2004) on a column of nominal diameter equal to 100 mm and catalytic baskets filled by resin spheres. The authors proposed a correlation for the liquid distribution between catalytic baskets and corrugated sheets. The minimum admissible liquid velocity in the range of validity of the correlation is above $13 \text{ m}^3/\text{m}^2/\text{h}$. We decided therefore to compare the tomographic experimental splitting factor obtained at $14 \text{ m}^3/\text{m}^2/\text{h}$ with the value obtained by the above mentioned correlation based on collecting experiments.

Following the same approach described for Katapak-SP 11, the liquid flowrate on corrugated sheets has been calculated by means of the Suess-Spiegel correlation on the basis of the local h_{lOC} experimental data obtained by tomography. The average splitting factor derived from local values equals to 0.689, while, for the same liquid load, the correlation proposed by Ratheesh and Kannan

(2004) provides a value of 0.685. The agreement is remarkable, thus confirming the soundness of the used approach also for Katapak-SP 12 results.

The importance of this analysis on liquid distribution is twofold. On one side, the liquid flowing in the separation zone interacts with the gas and determines gas pressure drops and separation performances. On the other side, the liquid flowing inside the catalytic baskets affects the corresponding liquid hold-up and hence the saturation of the particle bed and determines the reactive performances.

As shown above, catalytic baskets hold-up is accessible by tomographic measurements. We therefore used some correlations available in the literature for its evaluation to further verify the methodology. In particular we referred to the works of Van Hasselt et al. (1999) and Hoffmann et al. (2004). In particular, the correlations take into account two contributions: a minimum hold-up due to capillary forces acting at the lowest end of the baskets, and corresponding to the capillary liquid height, and a dynamic hold-up contribution which is a function of the liquid load. For the sake of clarity, we write in the following the used correlations with the nomenclature proposed in this paper. We also considered as additional contribution the liquid hold-up corresponding to the wire gauze envelope wet by the liquid capillary hold-up. This last term can be expressed as the ratio of the liquid volume on the catalytic baskets volume:

$$hl_{wg} = \frac{h_{cap}}{h_{CB}} \cdot \frac{\varepsilon_{wg} \cdot \psi_{wg-V}}{\psi_{CB-V}} \quad (9)$$

Van Hasselt et al. (1999) proposed to calculate the liquid hold-up as a function of the bed saturation with liquid, i.e. the volume of liquid contained in a unit void volume of the particle bed. At the bottom of the bed, below the capillary liquid height, the saturation is attained, while above the authors adopted a relation originally proposed by Benkrid et al. (1997) for the dynamic hold-up in single-phase trickle flow beds, as a function of the liquid Reynolds and Galileo numbers:

$$hl_{CB} = \beta \cdot \varepsilon_{PB} + hl_{wg} = \frac{[h_{cap} + (h_{CB} - h_{cap}) \cdot \beta_{TB}]}{h_{CB}} \cdot \varepsilon_{PB} + hl_{wg} \quad (10)$$

The capillary height, h_{cap} , is a function of the liquid properties and of the packed characteristics:

$$h_{cap} = \frac{6}{dp} \cdot \frac{1 - \varepsilon_{PB}}{\varepsilon_{PB}} \cdot \frac{\sigma_L}{\rho_L \cdot g} \cdot \cos(\theta) \quad (11)$$

where the contact angle between liquid and solid particles (water and glass particles in the present case) is taken equal to 56° , as suggested by the authors.

The saturation in the upper part of the bed, β_{TB} , is expressed by the following relation:

$$\beta_{TB} = C \cdot \frac{(a_{LS} \cdot dp)^{0.4}}{\varepsilon_{PB}} \cdot \text{Re}_L^{0.6} \cdot \text{Ga}_L^{-0.4} \quad (12)$$

where the constant C is related to the tortuosity of the particles bed and has a value of about 2.

The liquid solid interface can be estimated as follows:

$$a_{LS} = \frac{a_{PB}}{V_{PB}} = \frac{6}{dp} (1 - \varepsilon_{PB}) \quad (13)$$

While the Reynolds number and the Galileo number in the catalytic baskets are defined by the following relations:

$$\text{Re}_L = \frac{\rho_L \cdot dp \cdot u_{CB}}{\mu_L} \quad (14)$$

$$\text{Ga}_L = \frac{\rho_L^2 \cdot dp^3 \cdot g}{\mu_L^2} \quad (15)$$

Hoffmann et al. (2004), in their study on Multipak packing, proposed a correlation for hl_{CB} as a function of the catalytic basket porosity and the maximum liquid flowrate determined by the Equation 4:

$$hl_{CB} = \varepsilon_{CB} \cdot \left(1 - 0.5 \cdot \left(1 - \frac{u_{CB}}{u_{CB_max}} \right)^2 \right) + hl_{wg} \quad (16)$$

Figure 11 shows the catalytic baskets hold-up as a function of the liquid load for Katapak-SP 11. Global values obtained by averaging local hold-up data (see Figure 6b) relative to cross sections over the corresponding volume are compared with hl_{CB} values calculated by means of Eq.10 and Eq.16.

Figure 11.

The results predicted according to the model suggested by Van Hasselt et al. (1999) are in very good agreement with the tomographic data. The correlation of Hoffmann et al. (2004) gives slightly over-predicted values. This can be explained considering that this correlation was developed and tested on Multipak, where the corrugated sheets are made of wire gauze and capillary effects, encouraging the liquid to flow inside the catalytic baskets, can be supposed to be more important.

Conclusions

The present work has described new qualitative and quantitative data on liquid hold-up and liquid distribution inside the catalytic structured packings Katapak-SP 11 and Katapak-SP 12. These results have been obtained at local scale along the column height by means of a high energy X-ray tomograph and the contributions inside the catalytic baskets and in the open channels occupied by the corrugated sheets have been distinguished.

Concerning the axial variation of the liquid hold-up inside the catalytic baskets as a function of the liquid load, the images visualized the progressive increment of the liquid height at the bottom of the spheres beds and the filling of the catalytic baskets at high liquid loads. This experimental evidence confirmed the existence of a change in the flow regime, as already observed in studies carried out on different catalytic packings. The catalytic bed wetting is complete only at high liquid loads.

The liquid hold-up profiles in the open channels showed a liquid accumulation in the sections corresponding to the wall wipers, at moderate and high liquid loads. If a countercurrent vapour phase is fed, the liquid hold-up in these sections may be additionally affected thus leading to the flooding occurrence. This outcome is particularly important small to pilot scale packings because wall effects are more pronounced than at industrial scale.

Volumetric data on total liquid hold-up have been obtained by averaging over the packed bed height the local values given by the sum of the two contributions. These values have been compared with global liquid hold-up data derived from RTD measurements and total hold-up data obtained by adding static and dynamic contributions measured with traditional draining technique. The observed good agreement confirmed the soundness of the tomographic image processing and the consistency of the different techniques.

Besides new experimental data and invaluable local information gathered with X-ray tomography, the main contribution of this study was the determination of the liquid flow distribution and superficial velocity in the packing zones, i.e. open channels and catalytic baskets, from the local liquid hold-up values. The methodology was validated by comparison with experimental data

obtained by collecting separately the liquid flowing out of the two zones at the bottom of the packed bed.

The use of a well established correlation to relate the liquid hold-up and the liquid superficial velocity in the packing separation zones was validated at high liquid load, being the hydrodynamic parameters related to filled catalytic baskets easily determined. At low liquid loads, the same correlation allowed the estimation of liquid superficial velocity in the separation zones from the knowledge of local hl_{OC} from tomography. This way, a splitting factor of the liquid flowrate between separation and reaction zones was determined. Correlations developed for single-phase trickle beds were adapted to the packing geometry and used to predict hl_{CB} as a function of the liquid superficial velocity in the catalytic baskets. The results were successfully compared with data obtained by tomography.

A similar procedure may be applied to any type of modular packing, which consists in determining the splitting factor between the different zones by x-ray tomography and then, in applying, to each zone of the packing, an hydrodynamic model adapted to its geometry.

In order to assess the applicability of the proposed correlation to industrial conditions, some additional tomographic measurements are planned to be performed. Fluid distribution in packings containing catalyst particles instead of glass beads will be tested. The influence of liquid phase properties will be evaluated by using more viscous liquids as well as liquids with lower surface tension.

Notation

a	specific geometric area (m^2/m^3)
a_{LS}	specific liquid-solid interfacial area of the particles bed (m^2/m^3)
A_C	area of the column section (m^2)
A_j	area of the section occupied by the j^{th} element , with $j^{\text{th}}=\text{CB, OC}$ (m^2)
c	constant in Equation 8
C	constant in Equation 12
D	nominal packing diameter (m)
d_p	glass particle diameter (m)
g	gravitational acceleration (m/s^2)
Ga	Galileo number (-)
h	height (m)
h_{CB}	particles bed height (m)
H	nominal packing height (m)
H_{max}	effective packing height (m)
hl	liquid hold-up (-)
L_{max}	effective packing diameter (m)
Q	volumetric flowrate (m^3/s)
Re	Reynolds number (-)
s_j	thickness of the j^{th} element, with $j=\text{CB, MP, wg}$ (m)
u	liquid velocity (m/s)
x	constant in Equation 8
z	column axial coordinate (m)

Greek letters

β	bed saturation ($\text{m}^3_{\text{liq}}/\text{m}^3_{\text{void}}$)
---------	-----------------------------------------------------------------------

ρ	density (kg/m ³)
ε	packing void fraction (-)
ε_{CB}	catalytic basket void fraction (-)
ε_{PB}	void fraction of the spheres bed inside the catalytic baskets (-)
Ψ	friction factor (-)
ψ_{j_V}	volumetric fraction occupied by the j^{th} element, with $j=CB, OC, WW, wg$ (-)
ψ_{j_S}	superficial fraction occupied by the j^{th} element, with $j=CB, OC, WW, wg$ (-)
μ	viscosity (Pa·s)
σ	surface tension (N/m)
θ	contact angle between liquid and solid spheres (°)

Subscripts

cap	capillary
CB	catalytic basket section
exp	experimental
L	liquid phase
max	maximum
MP	Mellapak Plus sheets
OC	open channel section
SS	Suess-Spiegel
TB	trickle bed
TOT	total
wg	wire gauze
WW	wall wiper circular section

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Figure captions

- Figure 1. Top view of Katapak-SP 11 (a) and side view of Katapak-SP 12 (b). Schematic representation of Katapak-SP 11 with some dimensions (see Table 1) (c). Positioning of the packings in a column (d).
- Figure 2. X-ray CT device (a) and radiogram of the column (b).
- Figure 3. Schematic representation of the experimental arrangement for estimating flow distribution between reaction and separation zones.
- Figure 4. Detail of the column radiogram (a) and tomographic images at (b) $z = 370$ mm, (c) $z = 450$ mm, (d) $z = 490$ mm, (e) $z = 570$ mm, (f) $z = 600$ mm, taken on Katapak-SP 11 packings irrigated with a liquid load of $5 \text{ m}^3/\text{m}^2/\text{h}$. Binary solid pixels are reported in gray and binary liquid pixels in blue.
- Figure 5. Tomographic images of Katapak-SP 11 packing taken at $z = 370$ mm at liquid loads of $5 \text{ m}^3/\text{m}^2/\text{h}$ (a), $10 \text{ m}^3/\text{m}^2/\text{h}$ (b), $14 \text{ m}^3/\text{m}^2/\text{h}$ (c), $19 \text{ m}^3/\text{m}^2/\text{h}$ (d), $25.5 \text{ m}^3/\text{m}^2/\text{h}$ (e). Binary solid pixels are reported in gray and binary liquid pixels in blue.
- Figure 6. Axial profiles of the superficial fraction occupied by catalytic baskets (a), liquid hold-up in catalytic baskets (b) and open channel (c) for Katapak-SP 11. Liquid hold-ups are relative to measurements at liquid loads of $5 \text{ m}^3/\text{m}^2/\text{h}$ (\blacklozenge), $14 \text{ m}^3/\text{m}^2/\text{h}$ (\blacksquare) and $25.5 \text{ m}^3/\text{m}^2/\text{h}$ (\bullet).
- Figure 7. Axial profiles of total liquid hold-up for Katapak-SP 11 packings (a) and Katapak-SP 12 packings (b) measured at liquid loads of $5 \text{ m}^3/\text{m}^2/\text{h}$ (\blacklozenge), $14 \text{ m}^3/\text{m}^2/\text{h}$ (\blacksquare) and $25.5 \text{ m}^3/\text{m}^2/\text{h}$ (\bullet).

Figure 8. Dependence on the liquid load of the dynamic liquid hold-up (\square) and of the volumetric total liquid hold-up obtained by tomography and by conventional measurements for Katapak-SP 11 (a) and Katapak-SP 12 (b) packings.

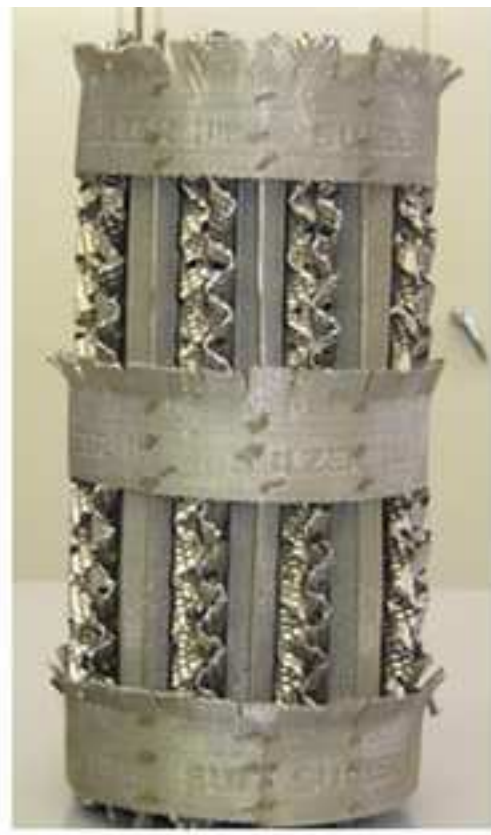
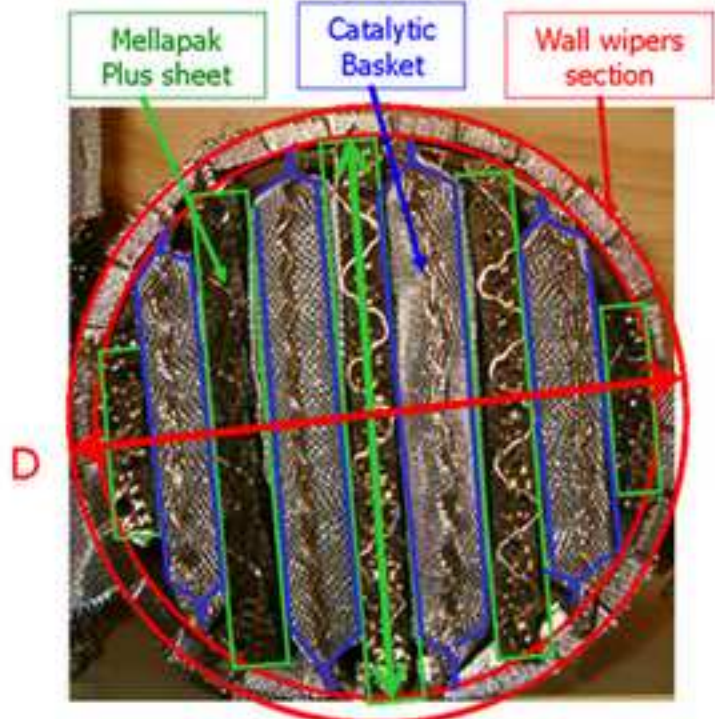
Figure 9. Liquid hold-up in the open channel for Katapak-SP 11 and Katapak-SP 12 as a function of liquid load: comparison between values calculated by the correlation of Suess and Spiegel (1992) (lines) and the experimental data (points).

Figure 10. Axial profile of the liquid superficial velocity in the open channel section (a) and of the splitting factor (b) at liquid loads of $5 \text{ m}^3/\text{m}^2/\text{h}$ (\blacklozenge), $10 \text{ m}^3/\text{m}^2/\text{h}$ (\blacksquare) and $14 \text{ m}^3/\text{m}^2/\text{h}$ (\bullet).

Figure 11. Comparison between catalytic baskets liquid hold-up obtained by tomographic experiments and predicted by correlations.

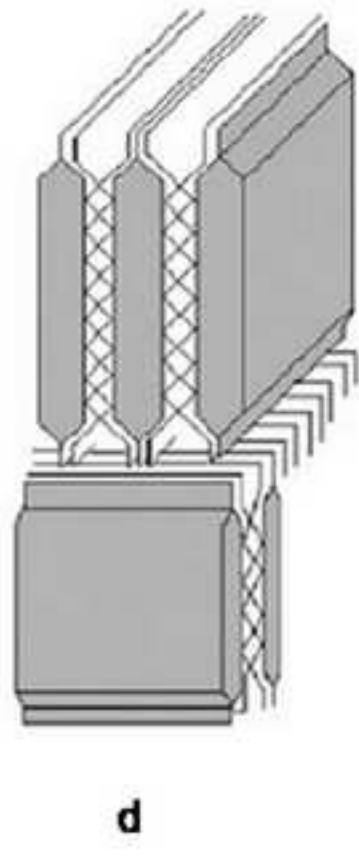
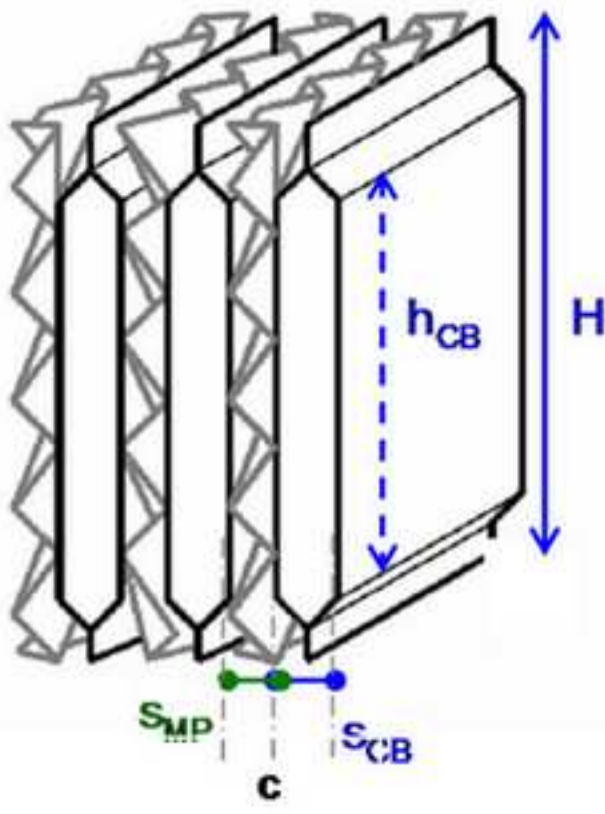
Tables

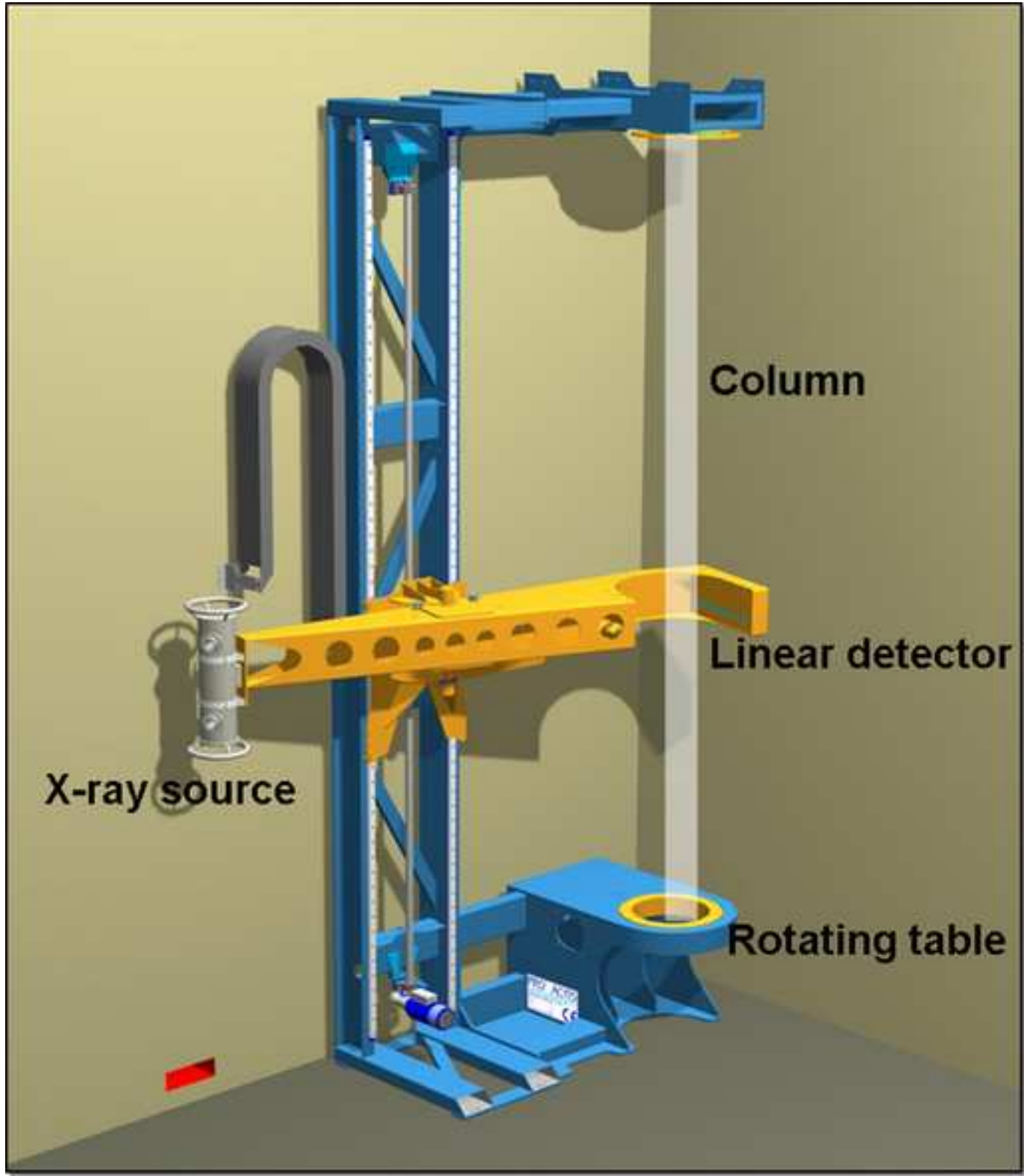
Table 1. Parameters which characterise the geometry of the packings under study.



a L_{max}

b





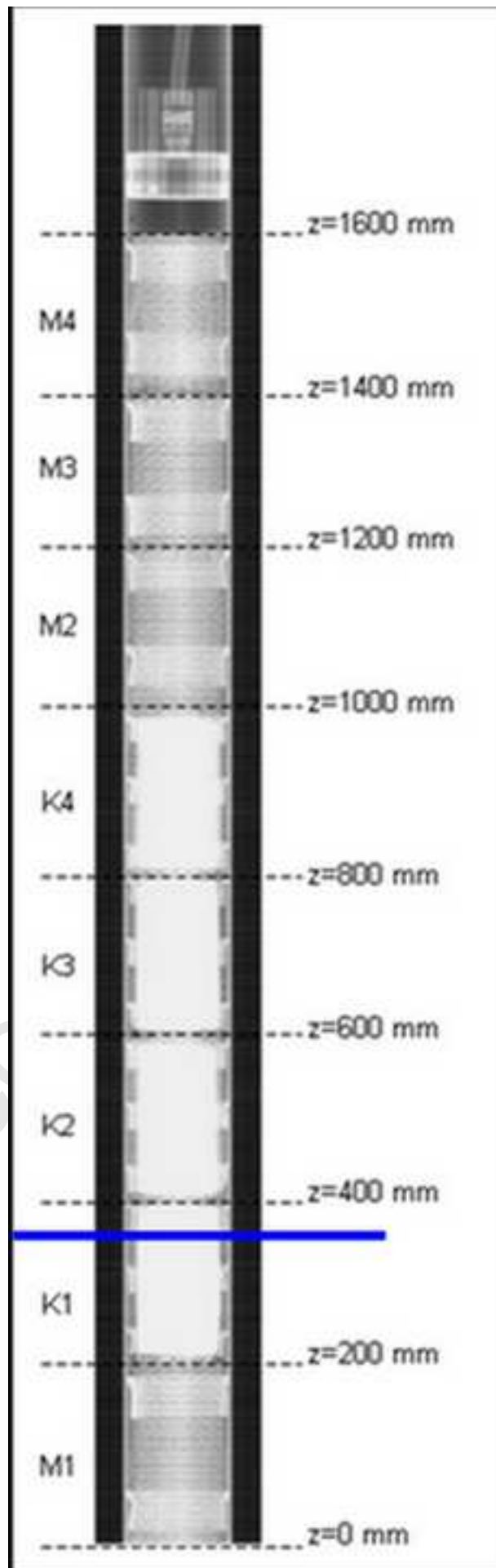


Figure 3
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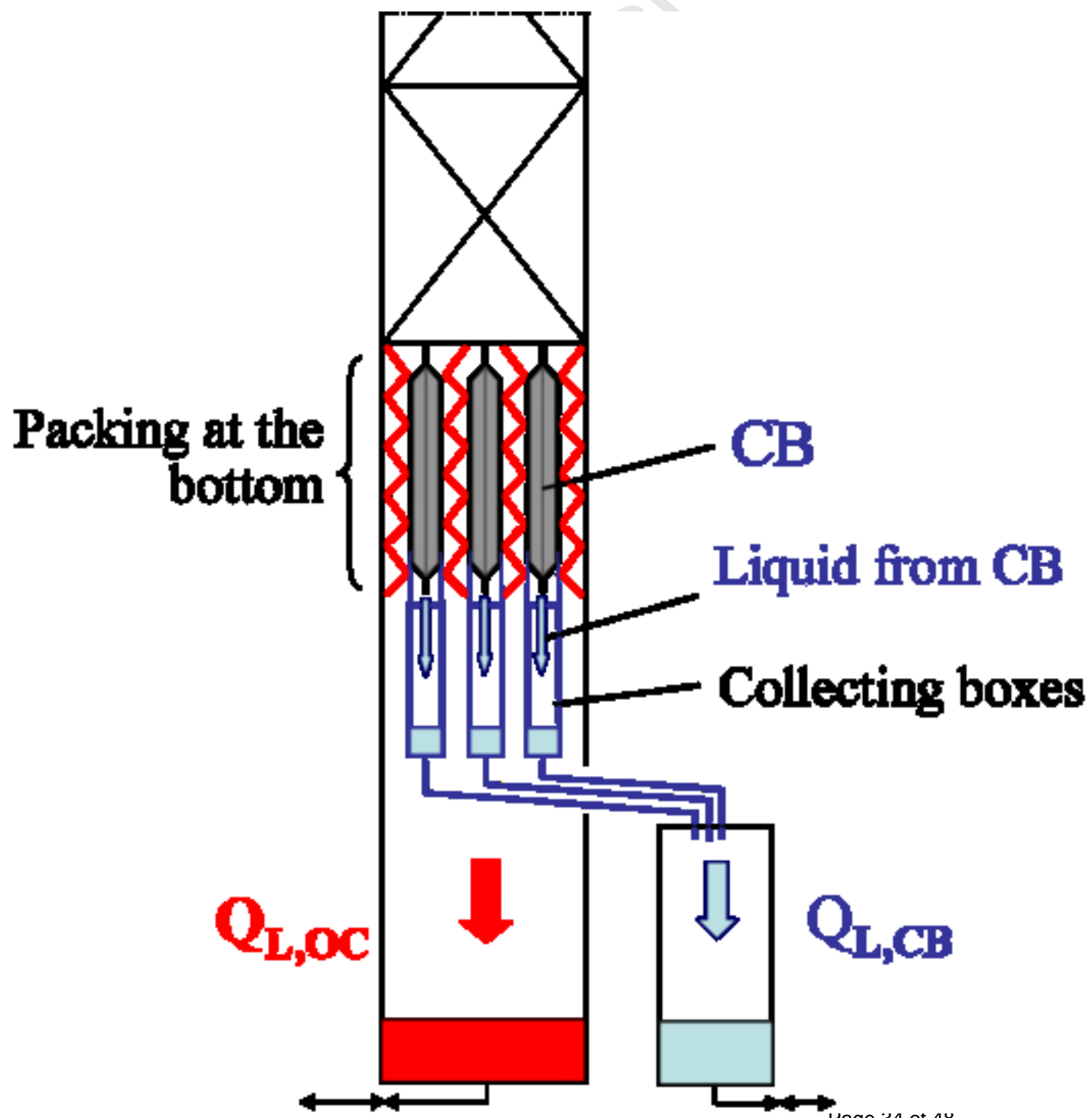


Figure 4
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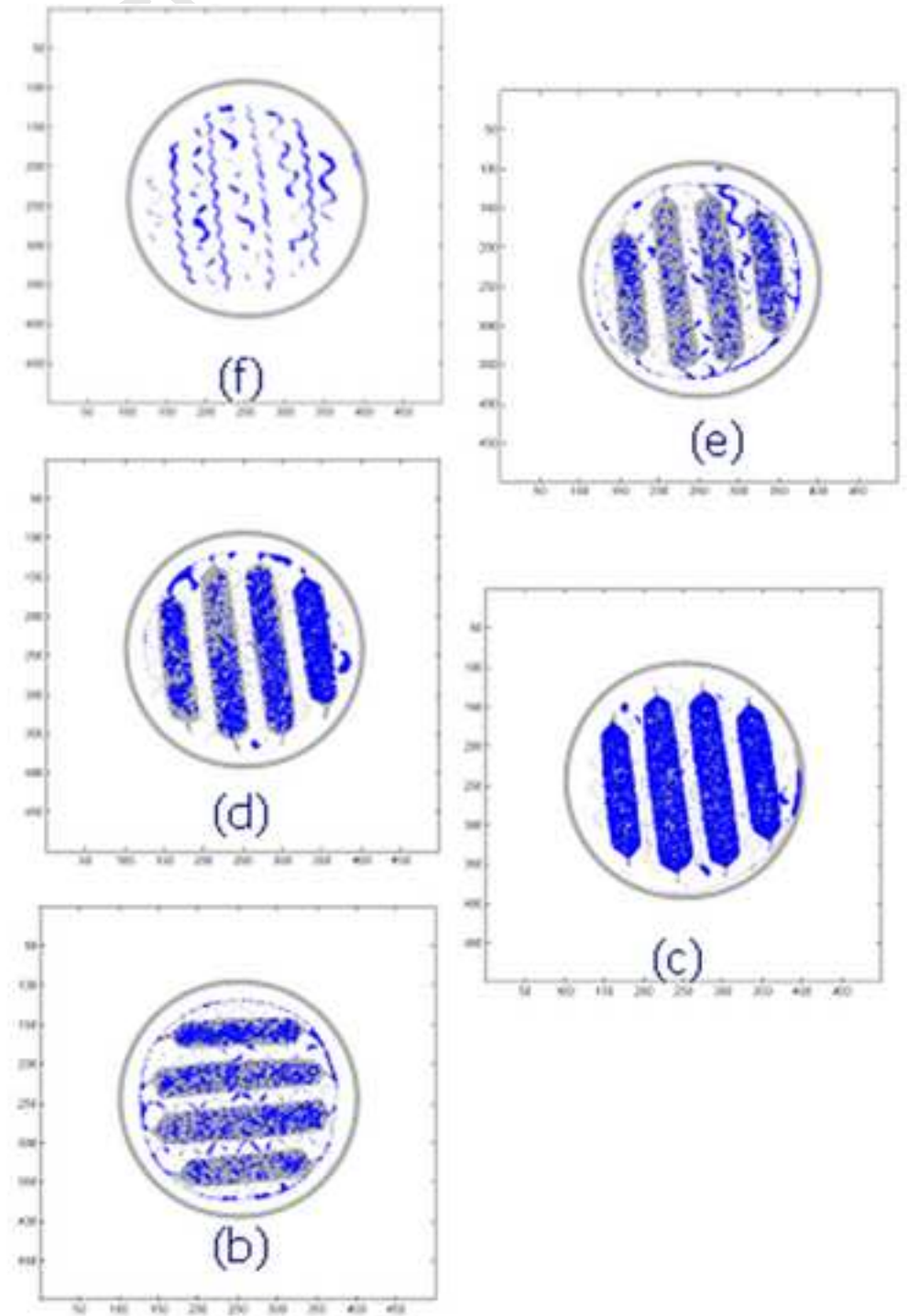
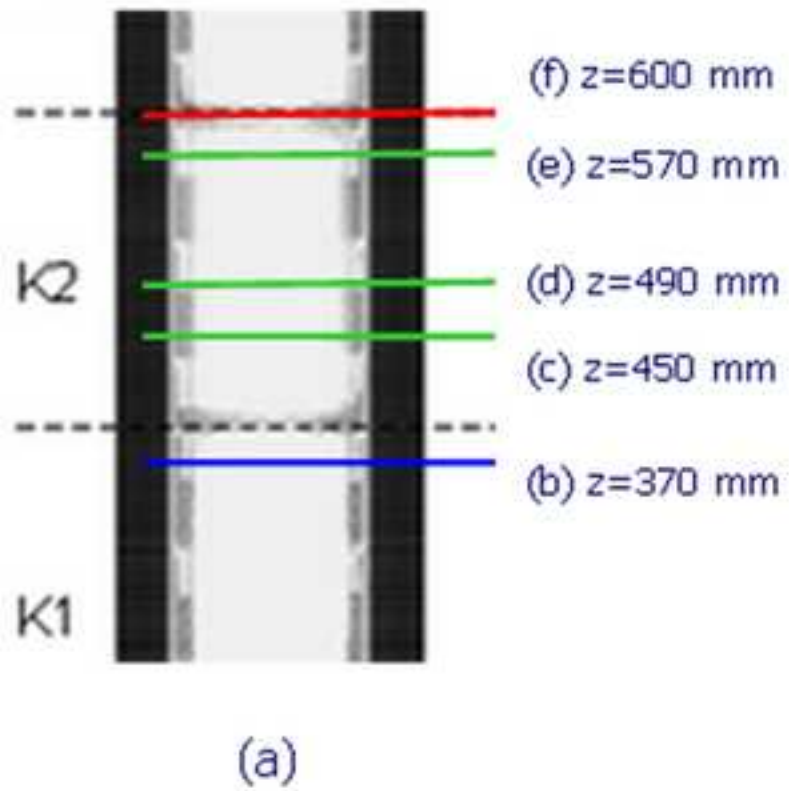
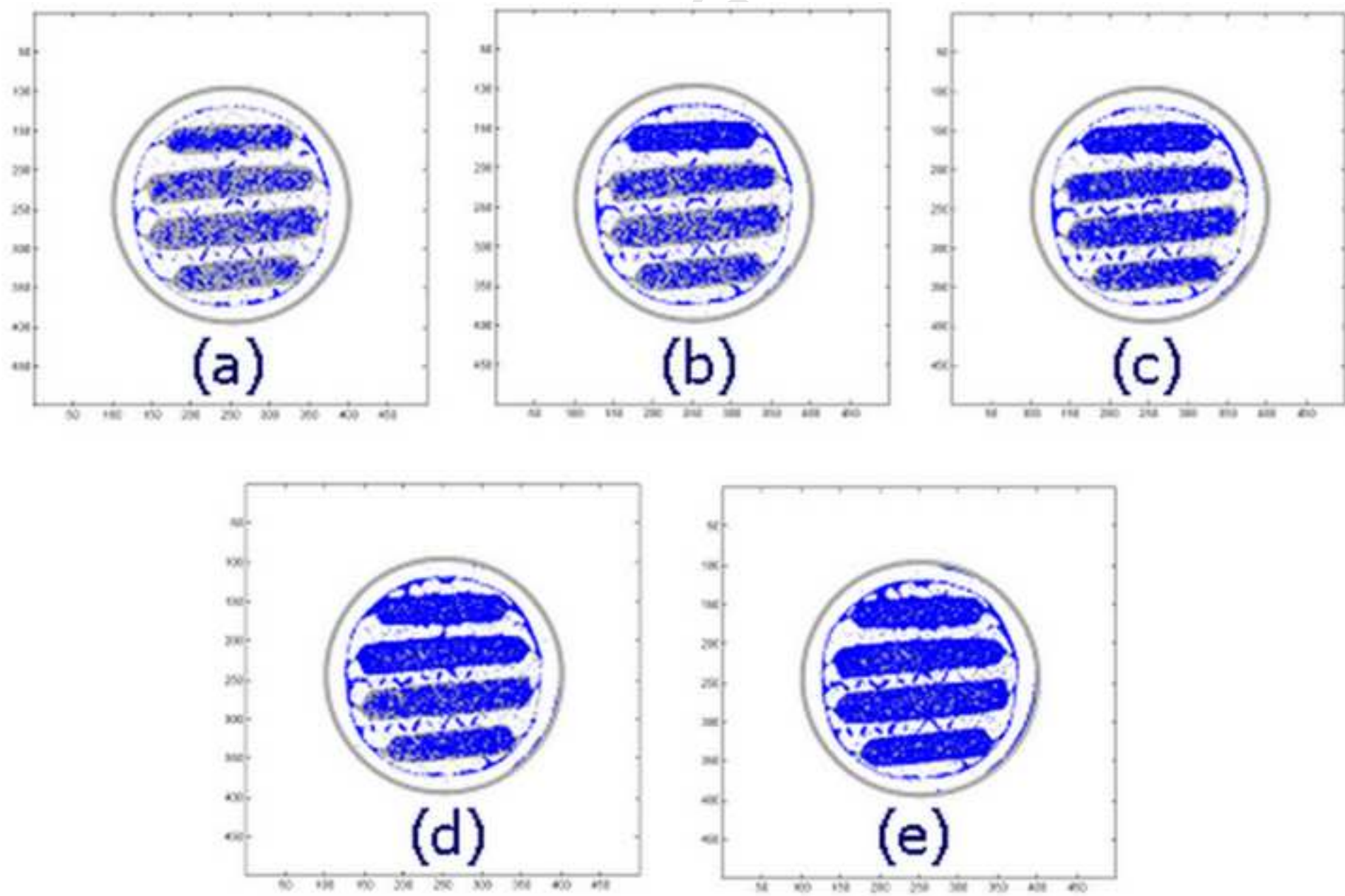
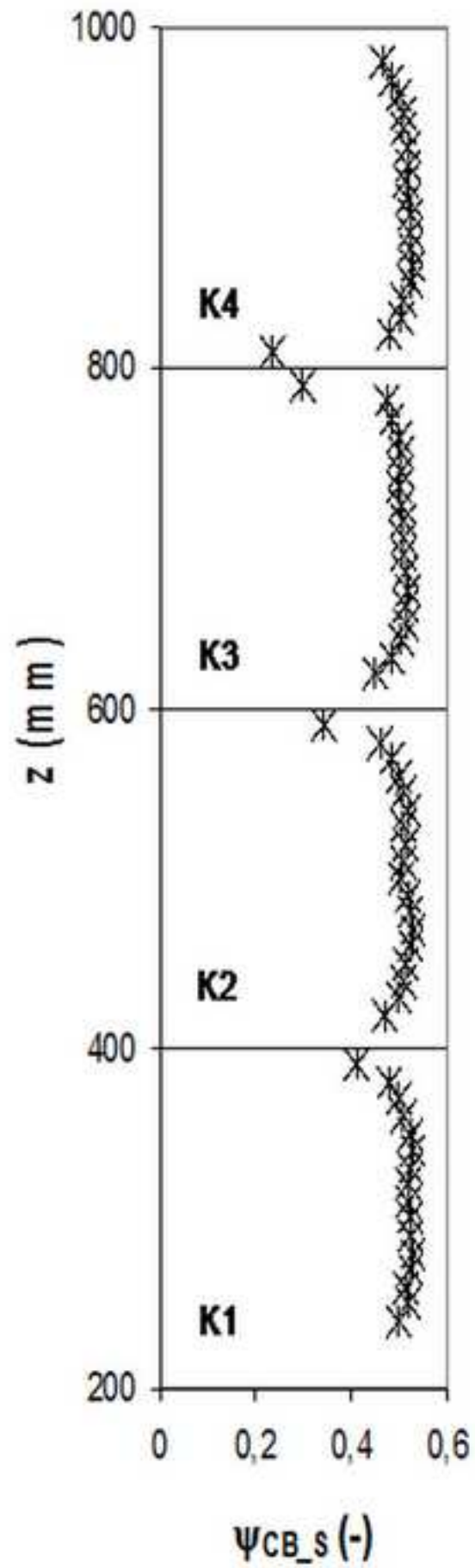
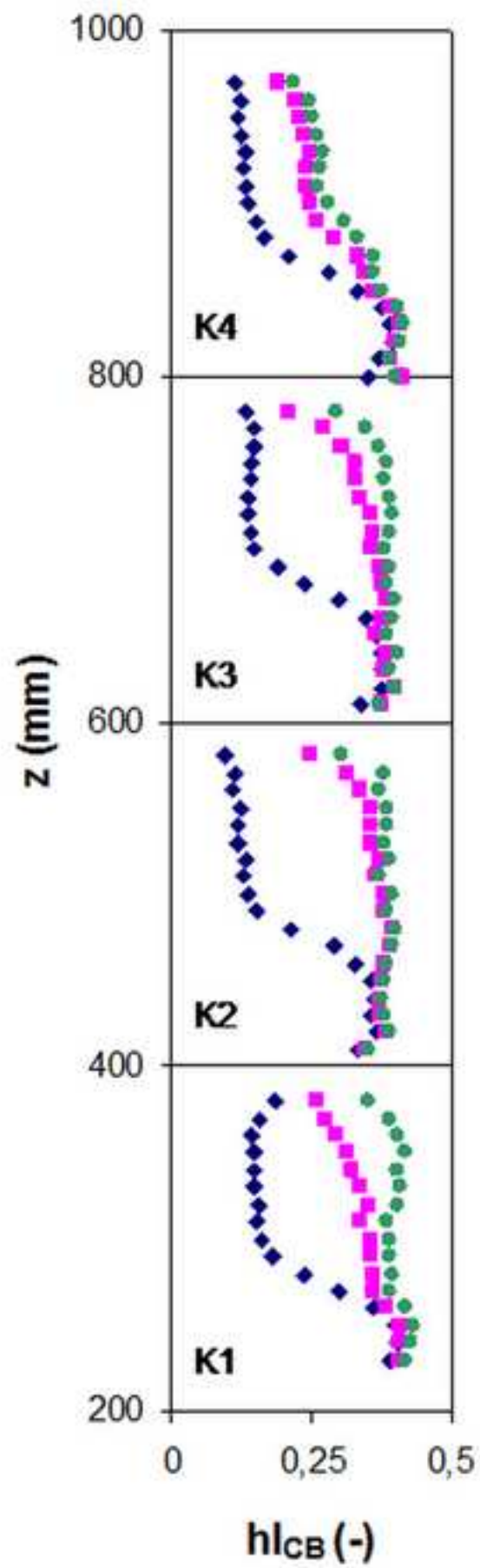
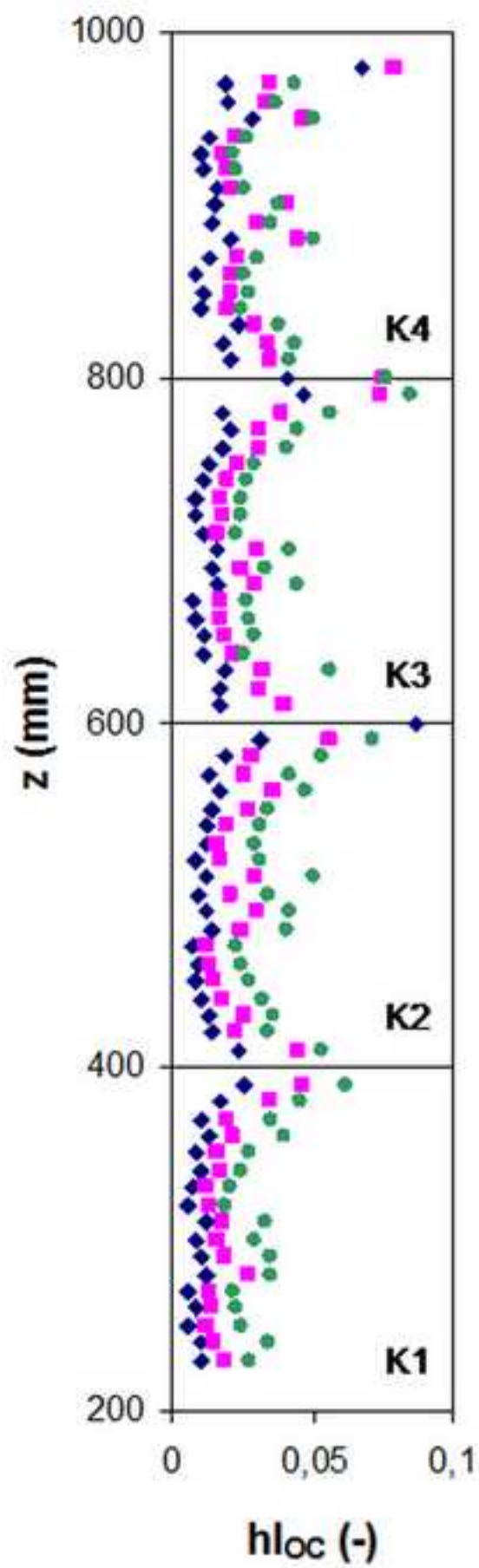


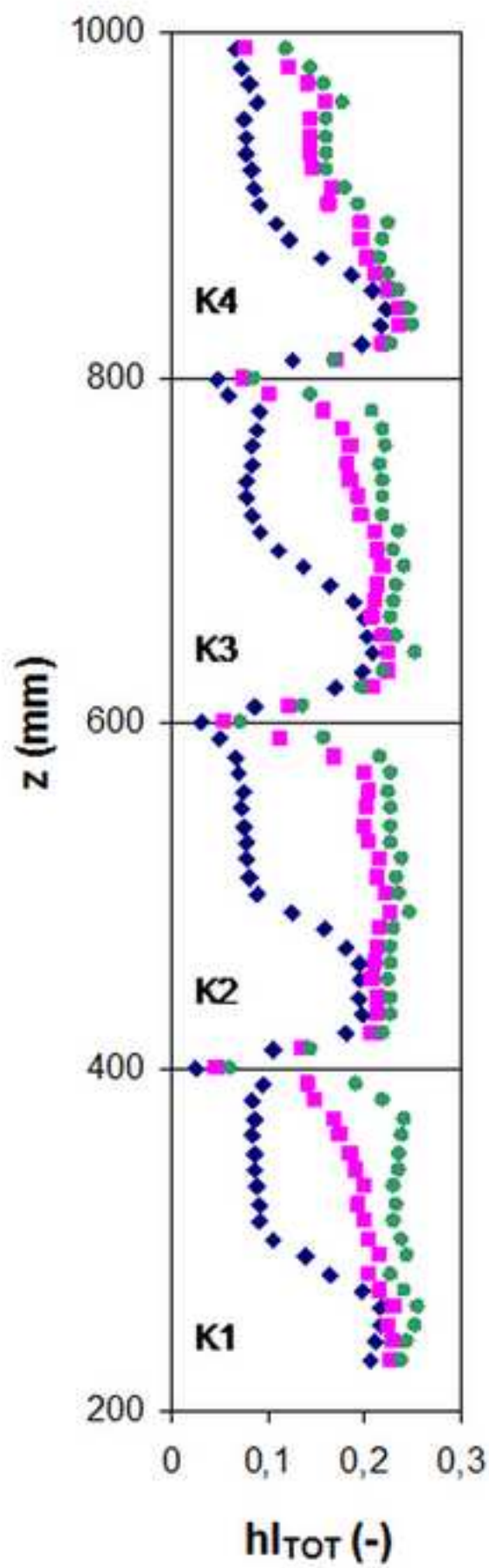
Figure 5
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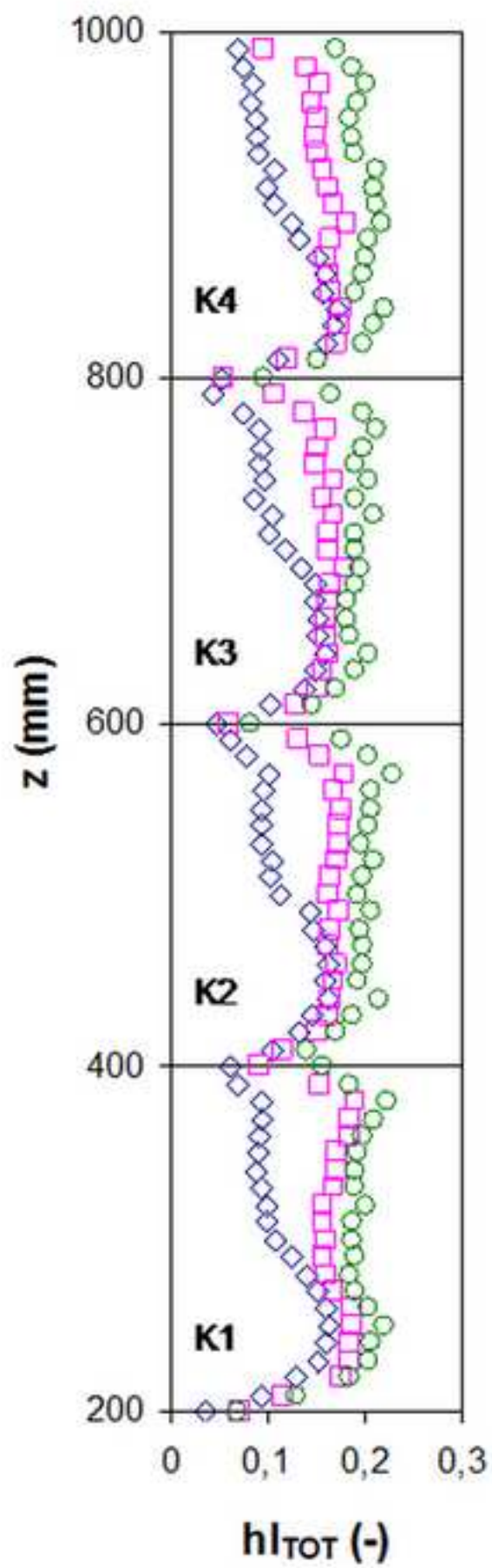


Figure 8a
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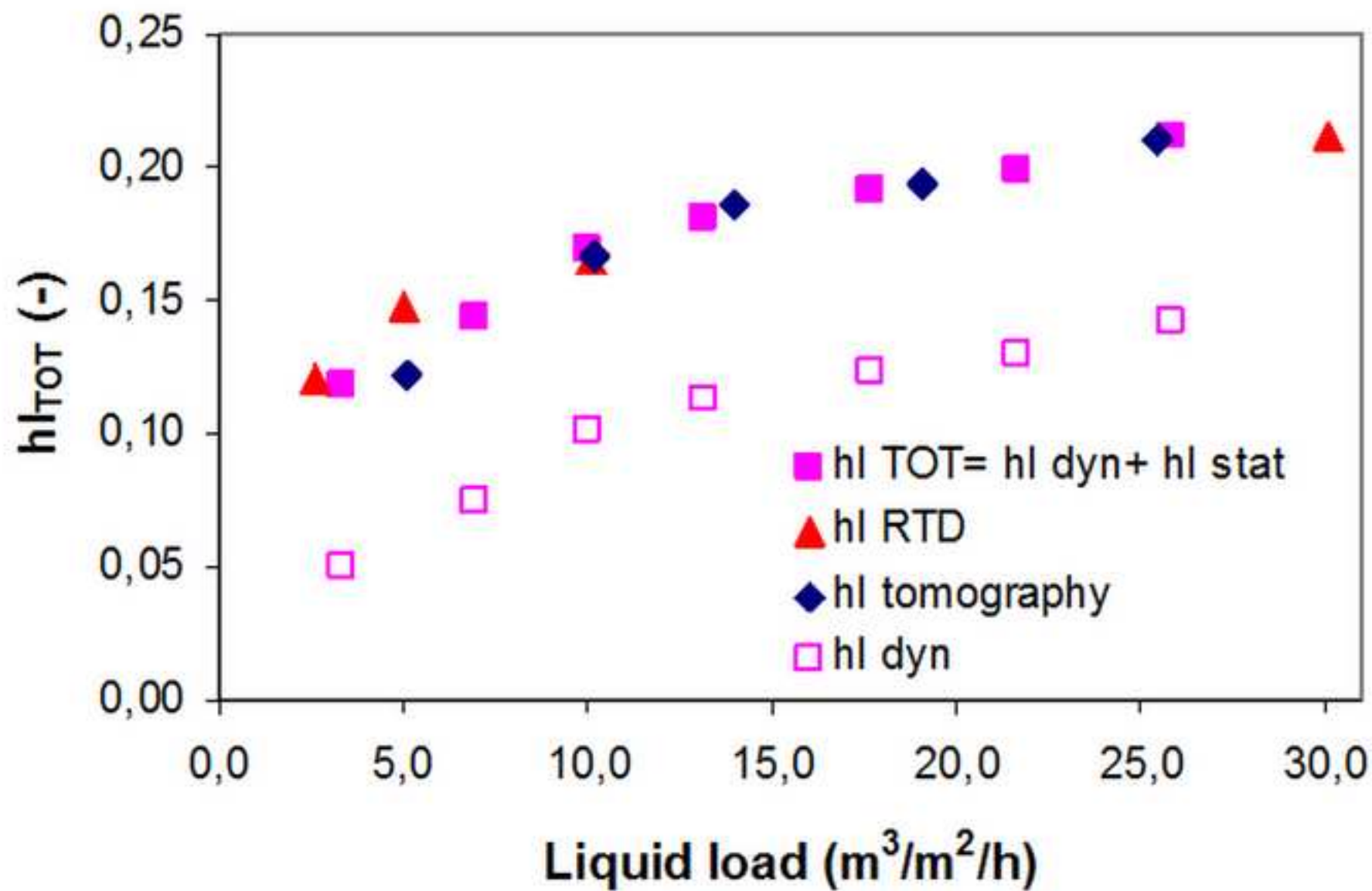


Figure 8b
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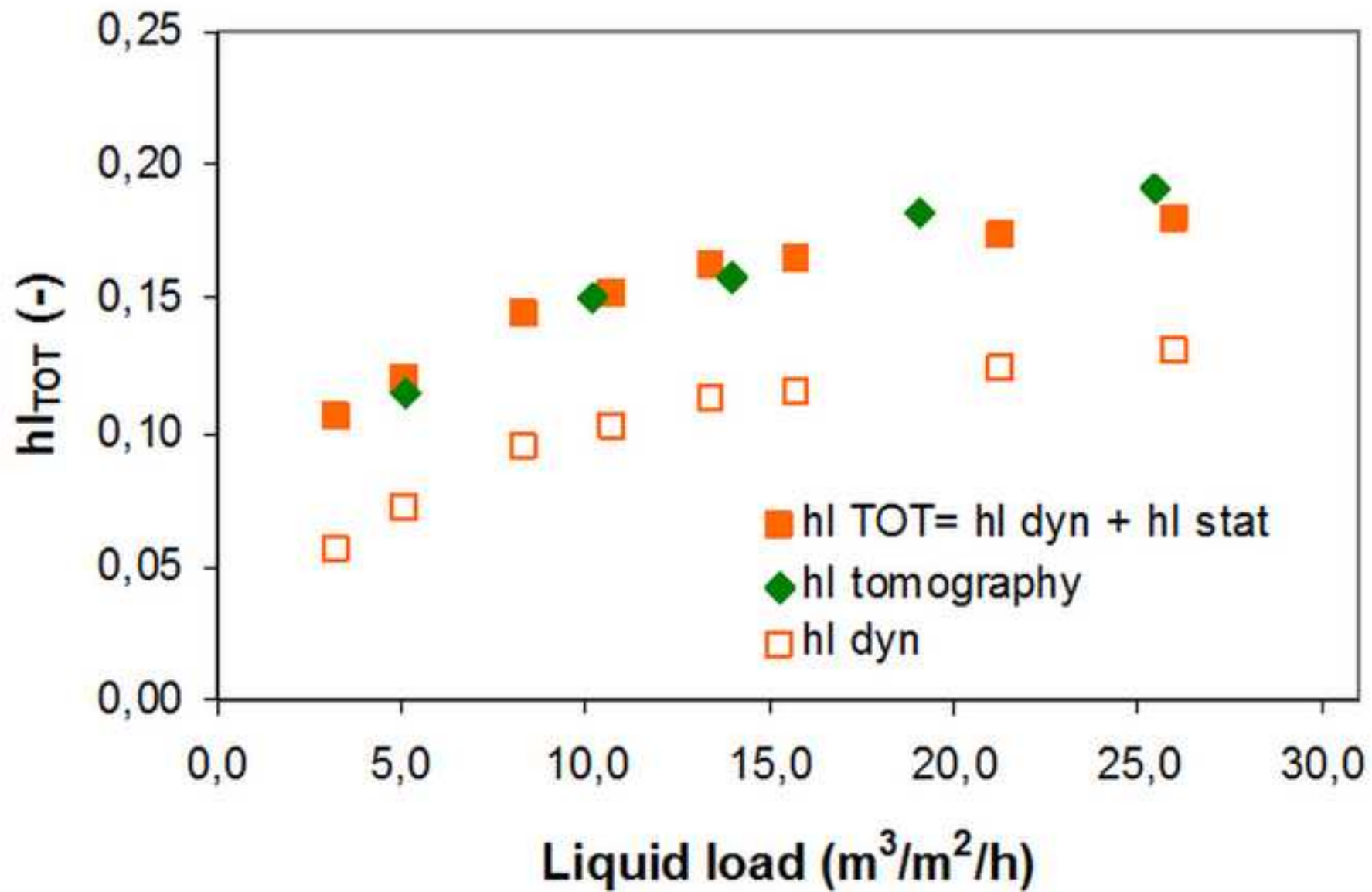
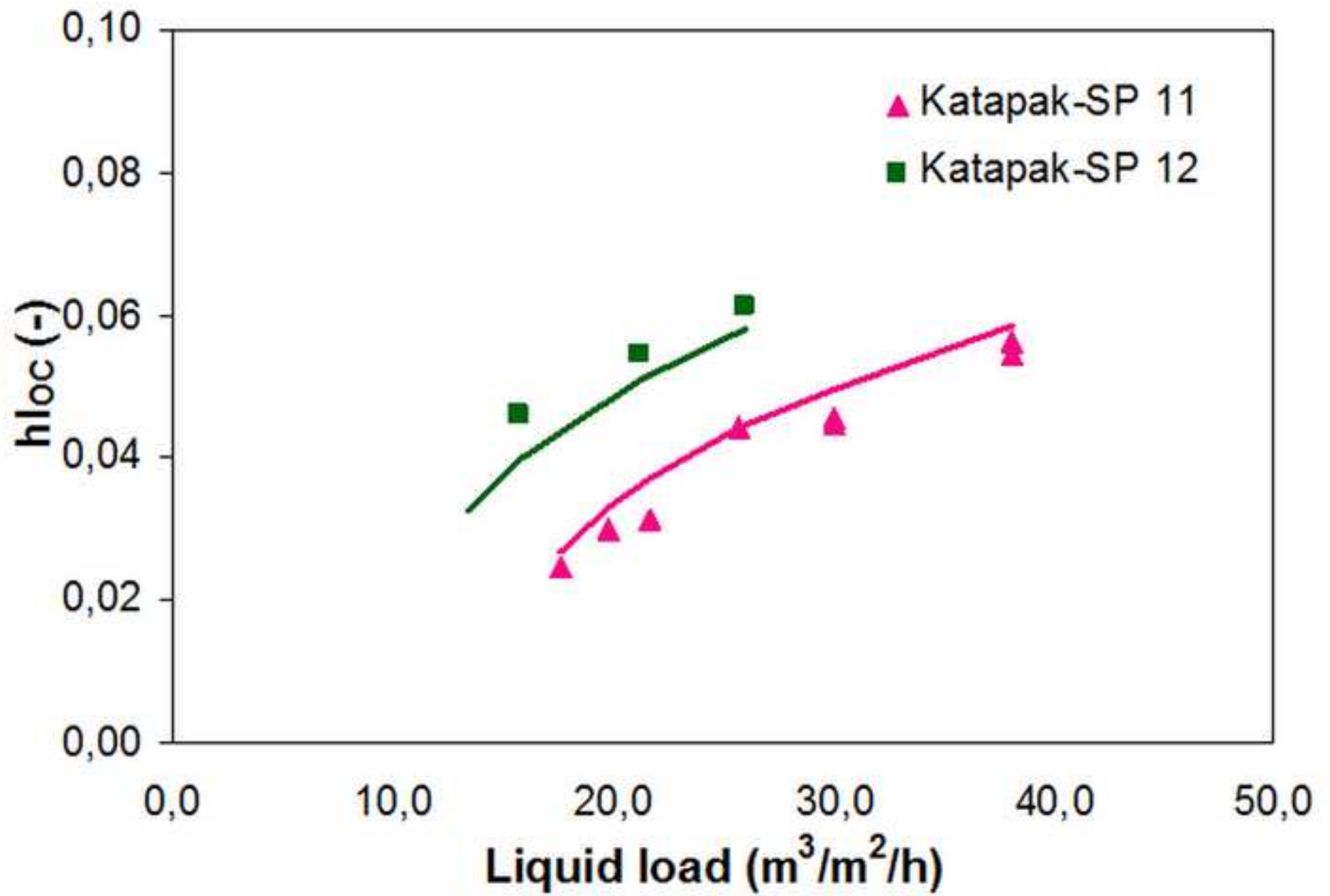
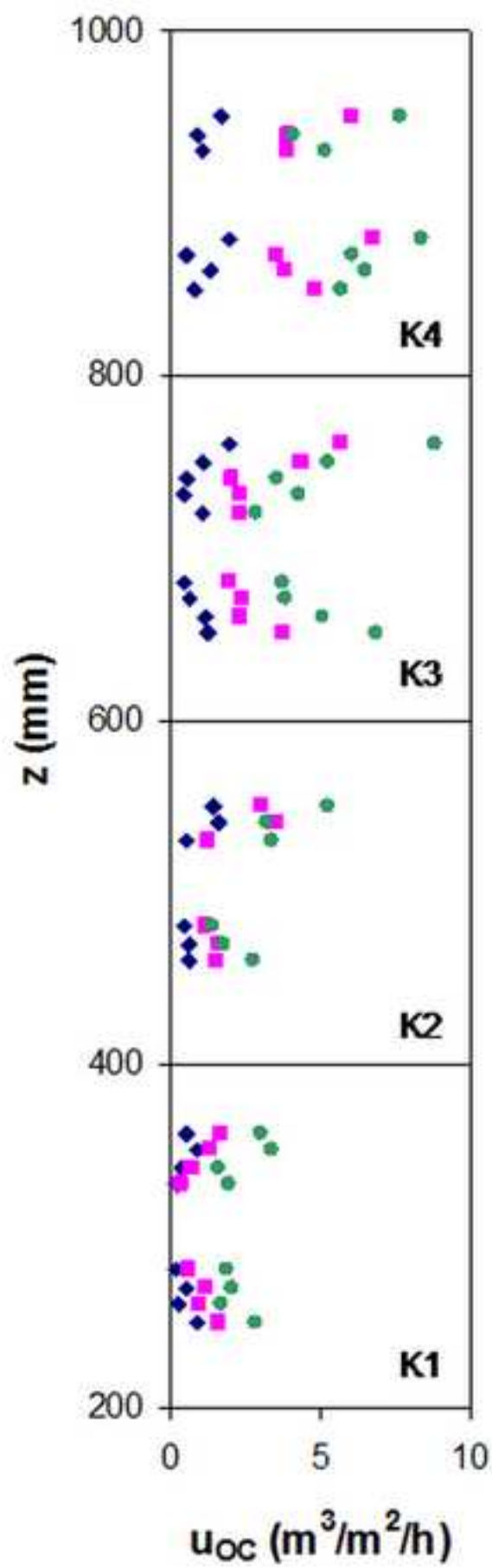


Figure 9
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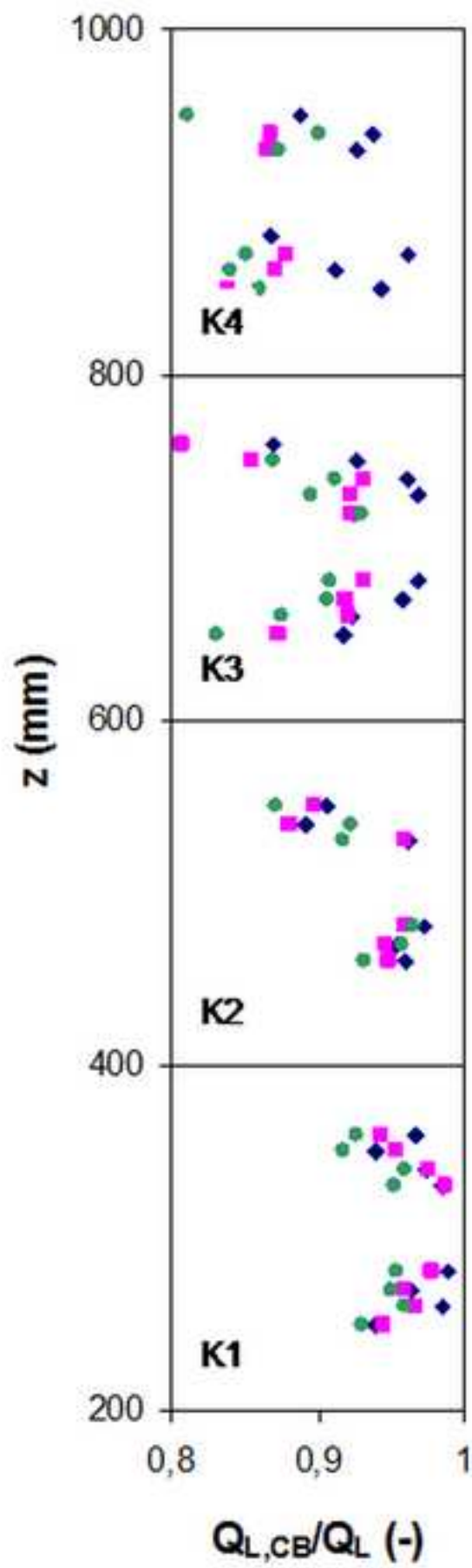


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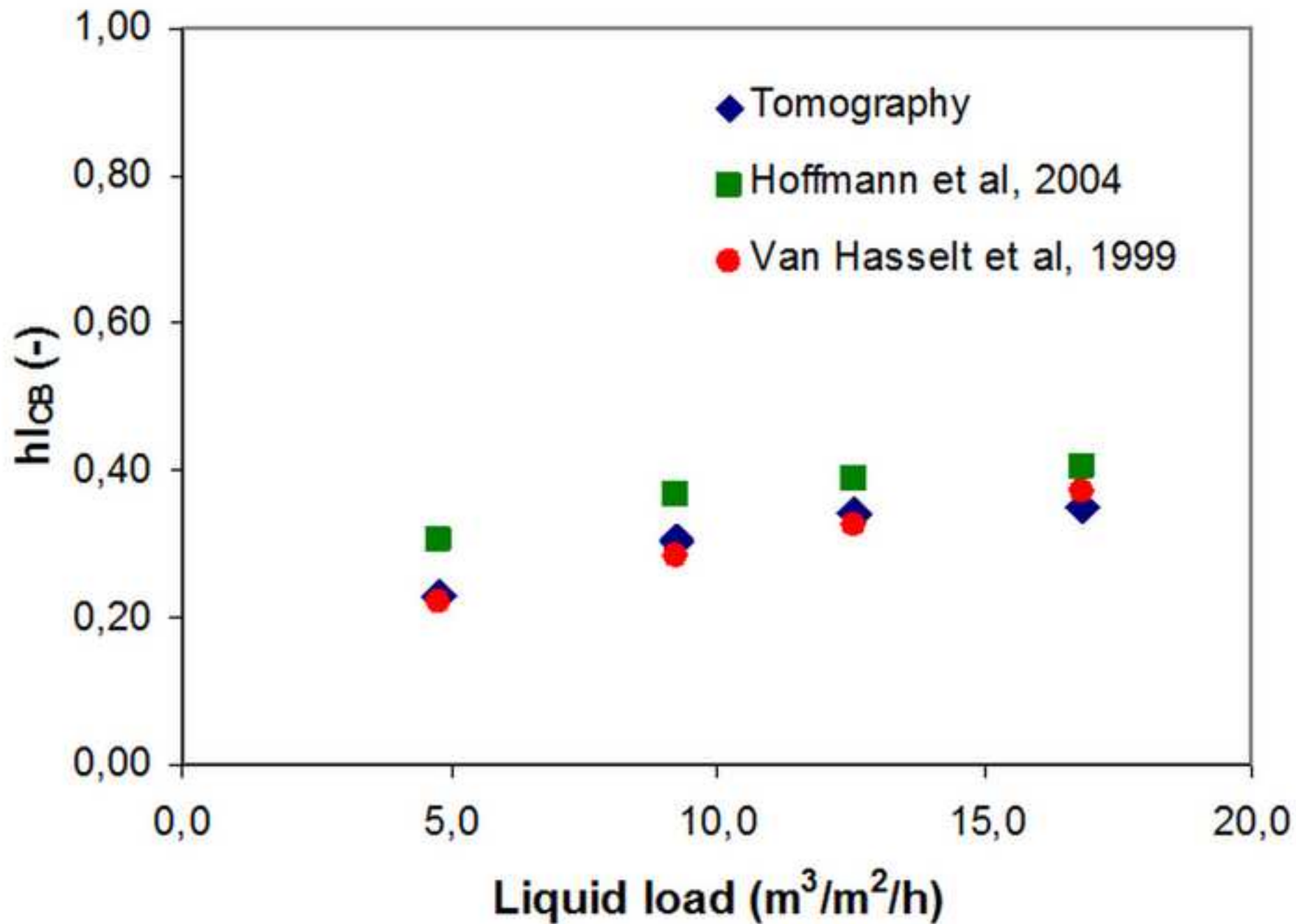


Table 1: Parameters which characterise the geometry of the packings under study.

		Katapak-SP 11	Katapak-SP 12
D (mm)	nominal diameter	100	100
L _{max} (mm)	effective measured diameter	90	93
H (mm)	nominal height	200	200
H _{max} (mm)	effective measured height	197	197
ϵ , -	packing porosity	0.74 (0.743 [*])	0.82 (0.80 [*] -0.82 ^{**})
a, m ² /m ³	specific geometric area	210 (219 [*])	282 (283 [*])
s _{CB} (mm)	catalytic baskets thickness	13.3-14.2	13
h _{CB} (mm)	particles bed height	181	181
dp (mm)	glass particles diameter	1	1
ϵ_{CB} , -	catalytic basket porosity	0.399	0.399
ϵ_{PB} , -	particles bed porosity	0.385	0.385
Ψ_{CB_V} (-)	CB volumetric fraction	0.418	0.297 (0.24 ^{**})
Ψ_{CB_S} (-)	CB superficial fraction	0.465	0.315
s _{wg} (mm)	wire gauze thickness	0.5	0.5
ϵ_{wg} , -	wire gauze porosity	0.7	0.7
Ψ_{wg_V} (-)	wire gauze volumetric fraction	0.015	0.011
Ψ_{WW} (-)	WW volumetric fraction	0.19	0.135
s _{MP} (mm)	Mellapak Plus sheet thickness	0.1	0.1
Ψ_{OC_V} (-)	OC volumetric fraction	0.392	0.567
Ψ_{OC_S} (-)	OC superficial fraction	0.342	0.547

^{*}From Viva et al. (2010); ^{**}From Ratheesh and Kannan (2004)