

## X-Ray CT Study of the Influence of Liquid Viscosity on Fluid Phase Distribution in Modular Catalytic Packings

S. Aferka\*, <sup>1</sup>A. Viva, <sup>1</sup>E. Brunazzi, P. Marchot, M. Crine, D. Toye

University of Liège, Laboratory of Chemical Engineering  
Sart Tilman, B6, B4000 Liège, Belgium  
s.aferka@ulg.ac.be

<sup>1</sup>University of Pisa, Department of Chemical Engineering  
Via Diotisalvi 2, 56126 Pisa, Italy

In the frame of the present study, the influence of liquid viscosity on liquid holdup distribution in a packed column equipped with the modular catalytic packing Katapak-SP 11 is measured. Water and an aqueous solution of glycerine, the viscosity of which equals 10 cP, are used as feed liquids. The estimation of liquid holdup is of great interest due to its strong influence on pressure drop, on solid wetting and on heat and mass transfer coefficients. The experimental technique used is high energy X-ray tomography, which is a unique technique to visualize the local liquid distribution inside the complex structure of packings and to quantify the liquid holdup at different scales.

### 1. Introduction

Modular catalytic packings are used in an increasing number of industrial applications where reaction and separation (i.e. distillation, absorption, extraction...) can be efficiently integrated in single equipment. Katapak-SP is the last generation modular catalytic packing manufactured by Sulzer Chemtech. The solid catalyst particles are maintained in baskets made of metallic gauze envelopes which are separated by corrugated sheets of MellapakPlus type.

Among the few experimental studies reported in the literature dedicated to the analysis of hydrodynamics in these modular catalytic packings, most have been carried out with water as working liquid (Behrens et al. 2008, Goetze et al 2001, Viva and Brunazzi 2009). But it is well known that industrial liquid fluids have usually a different viscosity than water and there is thus a lack of experimental data available to fully characterize hydrodynamics in modular catalytic packings.

X-ray tomography has been shown to be an efficient non-intrusive tool to see inside and to adequately image the liquid and gas flow distribution in columns filled with metal packings (Green et al., 2007; Aferka et al., 2007).

The present paper aims at studying the influence of liquid viscosity on liquid holdup distribution measured in a packed column equipped with Katapak-SP11 packing elements by means of X-ray tomography. An experimental campaign has been therefore carried out by using a glycerine solution with a viscosity of 10 cP as working liquid.

Results have been compared to those obtained with water in previous studies by the same technique (Aferka et al. 2010; Viva et al. 2011a) and to those evaluated with a more classical draining method.

## 2. Experimental Set-up

The column used in this study is 4 m high and has an inner diameter equal to 0.1 m. It is made of transparent PVC. The packed bed (1.6 m high) is constituted by the superposition of, from the bottom to the top, one MellapakPlus 752.Y element, four Katapak-SP11 elements and three MellapakPlus 752.Y used to get an initial uniform liquid distribution. These packings are manufactured by Sulzer Chemtech, CH. They are made of stainless steel (Figure 1). Baskets in the Katapak-SP11 elements are filled with 1 mm glass spheres. Both types of packings are 0.10 m diameter and 0.2 m high. The geometric characteristics of both types of packing elements are presented in Table 1. Water and an aqueous solution of glycerine (60% wt and 10 cP) are used as working fluids. The liquid superficial velocity ranges between 5 and 25.5 m<sup>3</sup>/m<sup>2</sup>/h for water and 4 and 22.8 m<sup>3</sup>/m<sup>2</sup>/h for glycerine solution. A multiple point source distributor (approx. 4000 drip points/m<sup>2</sup>) is used to feed the liquid at the top of the column.

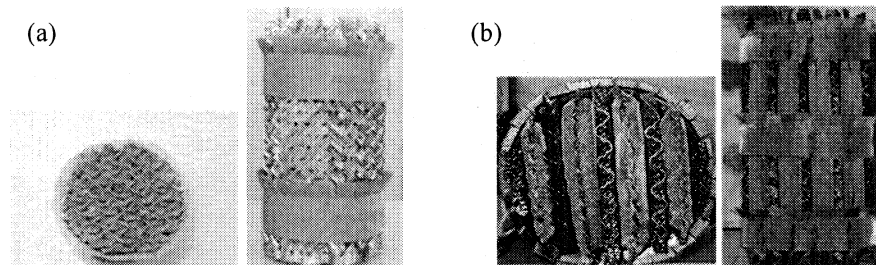


Figure 1: Top and side view of MellapakPlus 752.Y (a) and Katapak-SP11 (b)

Table 1: Packings geometrical parameters

	MellapakPlus 752.Y	Katapak-SP11
Height of packing element (mm)	200	200
Diameter of packing element (mm)	100	100
Void fraction (%)	97.5	76.7
Nominal specific area (m <sup>2</sup> /m <sup>3</sup> )	510	203

The X-ray CT facility is a high energy (420 kV) X-ray tomograph equipped with a fan beam X-ray source and with a 1280 photodiode linear detector which are both fixed on an arm able to translate vertically along the scanned object height (Figure 2). 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 3.8 m

may be analyzed with a spatial resolution equal to 0.37mm. 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 (70 in the present work) have to be reconstructed to get information on phase distributions relative to the whole bed.

Figure 3 shows a radiographic image (radiogram) of the whole packed bed obtained with the X-ray CT used in radiographic mode. On this radiogram, one may see four MellapakPlus 752.Y (M1, M2, M3, M4) and four Katapak-SP11 (K1, K2, K3, K4). Above, the liquid distributor is visible.

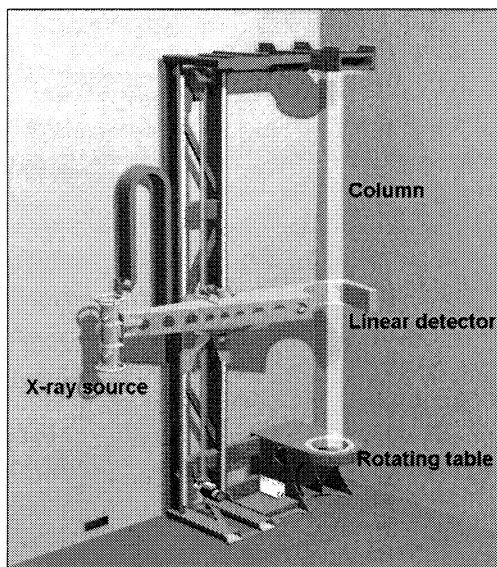


Figure 2: X-ray tomograph.

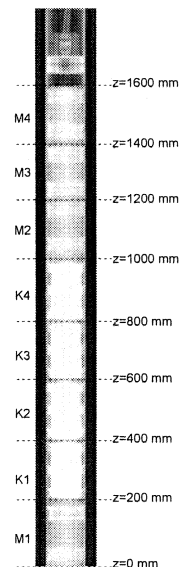


Figure 3: Radiogram of the packed column.

### 3. Results and Discussions

#### 3.1 Qualitative information

Figure 4 presents images of irrigated packing corresponding to cross sections situated at the same height in the column irrigated by the glycerine solution and by water,

respectively. On both images, the liquid superficial velocity,  $UL$ , equals  $10.2 \text{ m}^3/\text{m}^2/\text{h}$  and there is no gas flow. To obtain these images, we proceed as follows. First, dry packing images are recorded, reconstructed and thresholded, leading to the gray part of the images. Then, liquid distributions in the same cross sections are reconstructed and thresholded, leading to the blue part of the images. Finally, gray and blue parts are superimposed. Qualitatively, tomographic images show that, even at low liquid loads, catalytic baskets are completely filled by the more viscous solution, while they are partially filled by water in the same operating conditions.

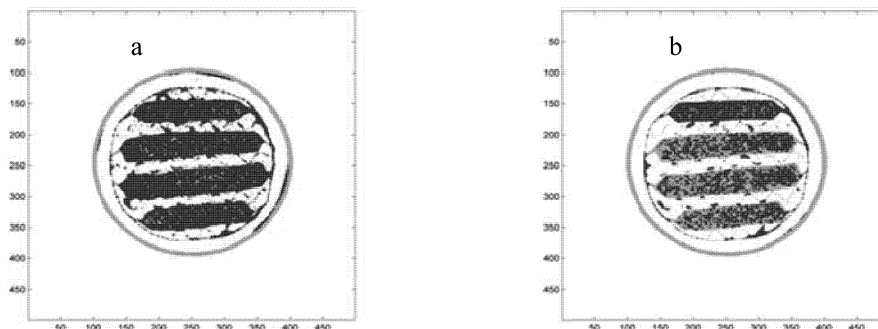


Figure 4: Cross section of Katapak-SP11 situated at a height of 420 mm from the bottom irrigated with the glycerine solution (a) and with water (b), for  $UL = 10.2 \text{ m}^3/\text{m}^2/\text{h}$ .

### 3.2 Quantitative information

From liquid distribution images similar to those presented on Figure 4, liquid holdup values may be computed (Aferka et al., 2010; Viva et al. 2011b). Figure 5 plots the total liquid hold up in different cross sections located at different distances from the bottom of the packed column, as a function of liquid velocity, without gas for the glycerine solution and for water. Liquid is not uniformly distributed along the packed bed height. As expected, the total liquid hold up increases with liquid velocity. Axial profile of liquid holdup is significantly improved (flattened) if liquid viscosity is increased.

For the determination of static liquid hold up, the column is first totally filled up with liquid, and then drained for 24 hours. Figure 5c plots the static hold up obtained by X-ray tomography at various heights for the four Katapak-SP11 elements with the glycerine solution and with water. One may observe that, at the bottom of each packing element, the static liquid hold up is higher, which corresponds to the liquid retained in baskets by the capillary forces (Aferka et al., 2007). As expected, axial profiles of static liquid holdup also show that static liquid holdup is increased if a more viscous liquid is used.

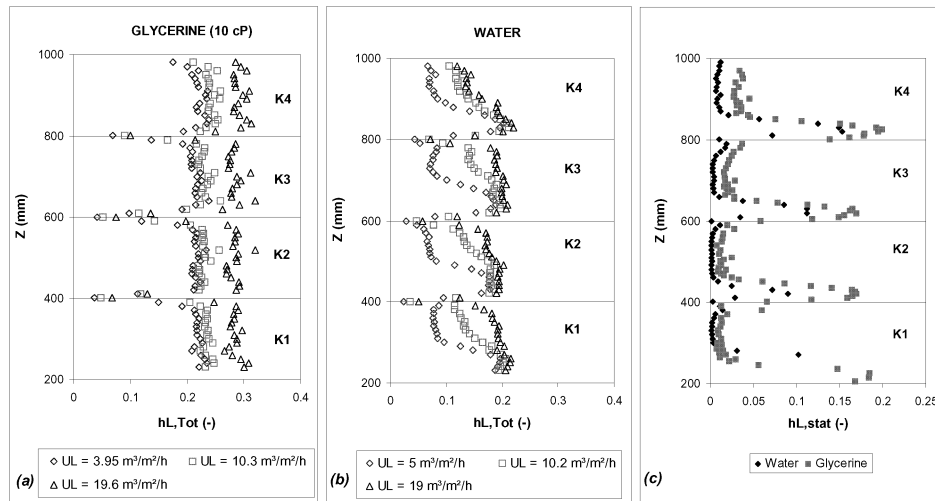


Figure 5: Axial profile of total liquid hold up (a and b) and of static hold-up (c) along the column height with water and glycerine.

### 3.3 Comparison with other experimental data

By averaging hold-up values obtained in different cross sections over the corresponding volume, one can determine global, bed scale, hold-up values which can then be compared to those obtained using global measurement method. As shown in Figure 6, a very good agreement is observed between global liquid holdup values measured by tomography and values obtained with a more classical draining technique (Viva and Brunazzi, 2009) both for water and for glycerine solution.

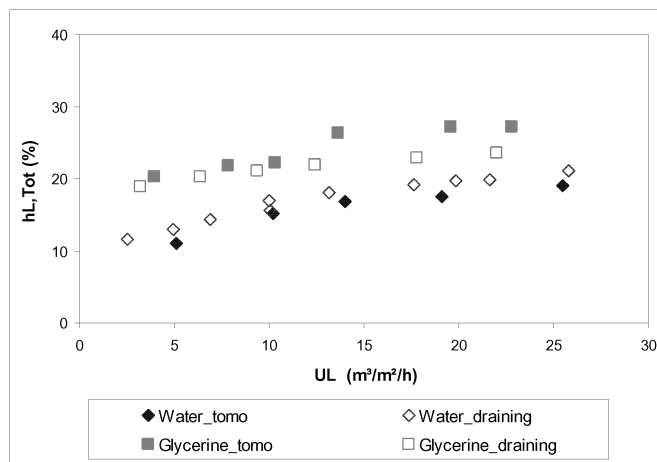


Figure 6: Comparison between experimental values of liquid hold-up obtained by X-ray tomography and by the draining technique.

#### 4. Conclusion

Local results obtained by X-ray tomography enable to get a better understanding of the fluid dynamic behaviour in modular catalytic packings when used with more viscous liquids. The analysis of liquid distribution images and of axial profiles of liquid holdup allows quantifying the positive influence of an increase of liquid viscosity on packing wetting. This information is crucial to support on-going efforts in the development of fundamental hydrodynamic models.

#### References

- Aferka S., Crine M., Saroha A.K., Toye D., Marchot P., 2007, In situ measurements of the static liquid holdup in Katapak-SP12 packed column using X-ray tomography, *Chemical Engineering Science*, 62, 6076-6080.
- Aferka S., Viva A., Brunazzi E., Marchot P., Crine M., Toye D., 2010, Liquid load determination in a reactive distillation packing by X-ray tomography, *Can. J. Chem. Eng.* 88, 611-617.
- Behrens M., Olujić Ž., Jansens P.J., 2008, Liquid hold-up in catalyst-containing pockets of a modular catalytic structured packing, *Chem. Eng. Technol.* 31, 1639-1637.
- Green C.W., Farone J., Briley J.K., Eldridge R.B., Ketcham A.R., Nigtingale B., 2007, Novel application of X-ray computed tomography: Determination of gas/liquid contact area and liquid holdup in structured packing, *Ind. Eng. Chem. Res.*, 46, 5734-5753.
- Toye D., Crine M., Marchot P., 2005, Imaging of liquid distribution in reactive distillation packings with a new high energy X-ray tomograph, *Meas. Sci. Technol.*, 16, 2213-2220.
- Viva A., Brunazzi E., 2009, The influence of modular structure on the hydrodynamics of catalytic structured packings for reactive separation processes, *Chem. Eng. Trans.*, 17, 1519-1524.
- Viva A., Aferka S., Toye D., Marchot P., Crine M., Brunazzi E., 2011a, Determination of liquid hold-up and flow distribution inside modular catalytic structured packings, *Chem. Eng. Res. Des.*, doi:10.1016/j.cherd.2011.02.2009.
- Viva A., Aferka S., Brunazzi E., Marchot P., Crine M., Toye D., 2011b, Processing of X-ray tomographic images: a procedure adapted for the analysis of phase distribution in MellapakPlus 752.Y and Katapak-SP packings, *Flow Meas. Inst.*, doi:10.1016/j.flowmeasinst.2011.03.008.