Gamma scanning evaluation for random packed columns

Pablo A.S. Vásquez, Fabio E. Costa, Paulo R. Rela, Wilson A. P. Calvo,
Galo A.C. LeRoux and Margarida Mizue Hamada

Abstract - In the present study, the liquid (water) holdup distribution was measured in a packed column (7.62 cm diameter) filled with 0.9 cm glass Rashig rings using noninvasive gamma ray technique. Three different radioactive sources: 11,1 MBq ^{60}Co (1133 keV), 74 GBq ^{131}I (380 keV), and 740 GBq ^{99m}Tc (140 keV) with a CsI(Tl) detector coupled to a photodiode were used to scan the column. The primary objective is to detect spatial patterns and statistical information on liquid distribution in packed distillation columns. Vertical scans, at different positions of the packed bed, were made for different liquid and gas flow rates. A mathematical relation based in the transmission radioactive by materials was used to calculate the spatial variation of liquid holdup along the column. It was found that the liquid holdup distribution was not uniform and that the liquid distributor design had a significant effect on the holdup distribution. The system proved to be very useful in monitoring the condition of the process of packed column showing liquid distributions. The better results were found using a ^{99m}Tc source. Flooded sections in the column can easily be identified using this technique.

I. Introduction

Distillation, absorption and liquid–liquid extraction are among typical applications of mass transfer equipments. Packed columns are used extensively in separation processes involving gas and liquid due to their low pressure drops, high capacities and high efficiencies. Compared with tray columns, the design and scale-up procedures for packed columns are less reliable due to the lack of understanding of flow hydrodynamics and mass transfer processes [1]. It has long been recognized that liquid holdup is one of the most important parameters that affect flow distributions, pressure drops, and separation efficiencies in packed columns There exist a few studies on the measurement of liquid holdup based on the whole column. However, the liquid holdup varies locally due to the inhomogeneous structure of the bed [2–11]. Knowledge of such local information of liquid holdup is essential for any rigorous analysis of fluid dynamics and mass transfer efficiencies. Liquid holdup is defined as the volume fraction of the bed occupied by the liquid at a given operating condition. The total liquid holdup consists of static holdup and operating holdup [1,4, 5]. Many researchers found differences between static holdup and operating holdup [4]. The static holdup is defined as the liquid held in the contacting points between packings and on the packing surface which does not drain when the liquid supply to the column is discontinued. The operating holdup represents the liquid which will drain from the packing. There have been two major approaches to measure liquid holdup in the literature: draining method [2–4] and tracer method [5–11]. Elgin and Weiss [2] measured liquid holdup for four different types of packings (4.03-cm. clay Raschig rings, 0.635-cm. porcelain Berl Saddles, 1.27-cm. clay Berl Saddles and 1.27-cm. clay Balls) in a 7.62-cm. glass column by draining method. They found that liquid holdup was almost independent of gas rate and varied linearly with liquid rate below loading point. Using the same method, Jesser and Elgin [3] investigated the effect of liquid physical properties on liquid holdup for different sized packings without gas flow. Shulman et al. [4] measured total, static and operating holdup for ceramic Berl Saddles and Raschig rings, and they used their holdup data to successfully explain the significant differences observed when gas-phase mass transfer rates were measured by absorption and vaporization methods, respectively. The draining method involves the interruption of flow hydrodynamics due to the need to simultaneously cut off the liquid inlet and the outlet to the column. Van Swaaij et al. [6] proposed to use impulse tracer technique to measure holdup and residence time distribution in the liquid phase. They showed that the liquid holdup measured by the draining method and tracer method is equivalent and the entire liquid holdup is accessible to the tracer. However, it has been argued that the response to an impulse input may not include static holdup due to the fact that the short-lived tracer does not stay long enough in the packing to cause interaction with the static holdup [9–11]. Hence several researchers have used step decrease tracer approach to study static and operating holdup [9–11]. It was found that the static holdup determined by the draining method is greater than that determined by the tracer method. The draining method or tracer method can only give the average holdup for a whole packed column; it cannot offer any information on how the liquid is distributed in packings. Thus the effect of liquid distributor design could not be studied. In reality, the liquid holdup can vary with spatial position and this information is very important for a better understanding of flow hydrodynamics and mass

P.A.S. Vásquez, F.E. Costa, P.P Rela, W.A.P. Calvo and M.M. Hamada are with the IPEN/CNEN-SP, Travessa R, 400, Cidade Universitária, 05508-900, São Paulo, Brazil. (mmhamada@net.ipen.br)
G.A.C. LeRoux is with the Escola Politécnica da Universidade de São Paulo – Engenharia Química, São Paulo, Brazil.

transport in packed columns. Recently Toye et al. [12] have used the X-ray computed tomography to measure local liquid holdup in a packed bed filled with Cascade Mini-Ring 1A packing elements. From the reconstructed images, they can vividly demonstrate spatial flow variations. Another technique to measure local conditions is gamma ray tomography. This radiation scanning technique has been successfully used in the trouble shooting of distillation columns [13–19]. It allows engineers to study tray or packing hydraulics inside of a column at any set of online conditions without the need to interrupt column operation.

II. Material and Methods

The experimental setup, as shown in Fig. 1, consists of a 7.62 cm diameter cylindrical column filled with 0.95 cm glass Rashig rings, a liquid feed pump, a liquid distributor, a gas feed system and a gamma ray emitting and detecting system. The holdup measurements were taken at different packing depths. The gamma ray scanner had a CsI(Tl) detector and ^{60}Co, ^{99m}Tc, and ^{131}I sources. A schematic diagram of gamma ray attenuation measurement is shown in Fig. 2.

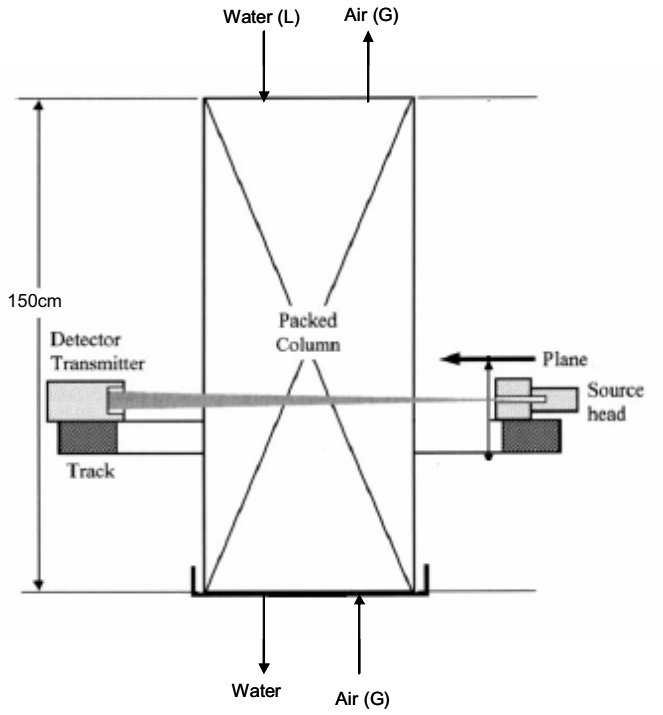


Figure 1. Experimental Set-up.

The gamma scanning measurements were carried along the column with tree different liquid flow rates 4, 10 and 20 gal/h with a fixed gas (air) flow of 2 ft^3/min. At each plane and a fixed liquid flow rate, the source-detector system was moved around the track through 3 equally spaced, premarked positions. The measurements were first made for the dry column to obtain the base line data for each of the scanning points. The same measurements were later made at the same positions with liquid circulating through the column. The average liquid holdup along each direction was then calculated based on these measurements as outlined in the next section.

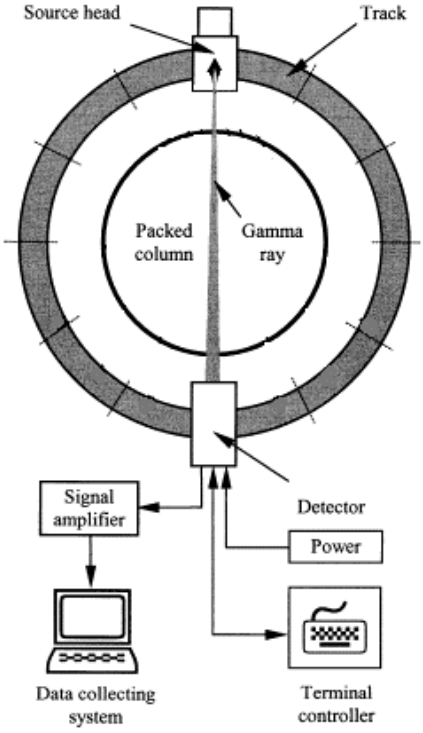


Figure 2. Scan system.

Determination of liquid holdup

If a gamma ray passes through a medium, its intensity will reduce from *Io* to *I* due to the absorption of radiation flux by the medium. The relationship between *I* and *Io* is given by Beer–Lambert's law:

$$\frac{I}{Io} = \exp\left(-\int \mu dL\right) \qquad (1)$$

where *Io* is the incident radiation intensity. *I* refers to the intensity measured after the gamma ray passes through attenuating medium of length *L*. and μ is the attenuation coefficient of the medium. From the measured *I* and *Io*, the attenuation *A* can be calculated as:

$$A = -\ln\left(\frac{I}{Io}\right) = \int \mu dL \qquad (2)$$

Depending on the constituting materials of the attenuating medium, the attenuation *A* can vary spatially across the column cross section. When the gamma ray passes through a

column filled with glass Rashig rings (dry column), the measured attenuation can be expressed in terms of the attenuation coefficient as:

$$\ln\left(\frac{I_{ref}}{Io}\right) = -\mu_{air}L_{air} - \mu_{rings}L_{rings} - \mu_{wall}L_{wall} \qquad (3)$$

where L_{air}, L_{rings} and L_{wall} are the total path lengths of gamma ray passing through gas, packing elements and wall, respectively. When there is a liquid phase circulating through the same column (wet column), the attenuation can be calculated as:

$$\ln\left(\frac{I_{hl}}{Io}\right) = -\mu_{air}(L_{air} - L_{water}) - \mu_{water}L_{water} - \mu_{rings}L_{rings} - \mu_{wall}L_{wall} \qquad (4)$$

where L_{water} is the total path length of gamma ray passing through water phase in the medium, which is related to the liquid holdup. Subtraction of Eq. (3) from Eq. (4), we get

$$\ln\left(\frac{I_{hl}}{I_{ref}}\right) = -(\mu_{water} - \mu_{air})L_{water} \qquad (5)$$

In order to get L_{water}, attenuation coefficients μ_{water} and μ_{air} or their difference must be determined first. This can be done through base line shootings. A base line shooting, with air as the only medium inside the column (empty column), results in

$$\ln\left(\frac{I_{air}}{Io}\right) = -\mu_{air}L_{water} - \mu_{wall}L_{wall} \qquad (6)$$

A base line shooting, with water as the only medium *inside* the column, results:

$$\ln\left(\frac{I_{water}}{I_{air}}\right) = -\mu_{air}(L_{air} - L_{water,b}) - \mu_{water}L_{water,b} - \mu_{wall}L_{wall} \qquad (7)$$

The first term in Eq. (7) represents attenuation through air that is outside of the column and in the air space between the source and the detector. From Eqs. (6) and (7), it follows

$$\ln\left(\frac{I_{water}}{I_{air}}\right) = -(\mu_{water} - \mu_{air})L_{water,b} \qquad (8)$$

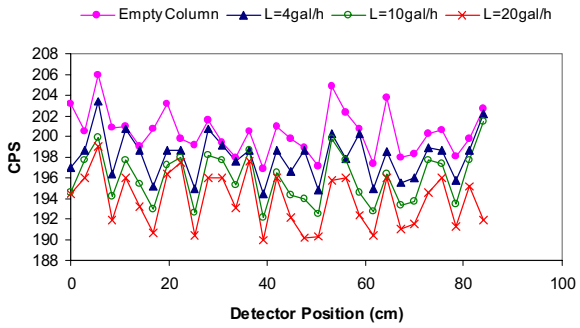
with I_{air}, I_{water}, and $L_{water,b}$ known, μwater–μair can be easily determined from Eq. (8). L_{water} can then be determined from Eq. (5). The chord-averaged liquid holdup along the shooting path '*i*' can be calculated by

$$\overline{h_{li}} = \frac{L_{water,i}}{L_{T,i}} \qquad (9)$$

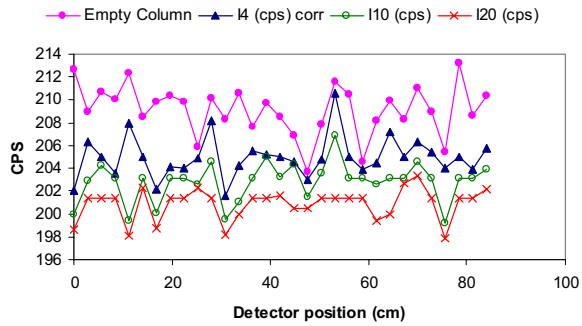
where L_T is the total length of a chord along the shooting path '*i*'. It can be determined from the geometrical considerations. To measure the attenuation along a certain chord, the radiation source and detector are aligned on the opposite sides of the column, as shown in Fig. 2. In this arrangement, the medium is the composite material of the column wall, solid packing elements, air, and water if there is liquid circulating through the column. At a fixed radiation source head and detector position, the radiation intensity transmitting through the medium was recorded by the detector.

III. Results and Discussion

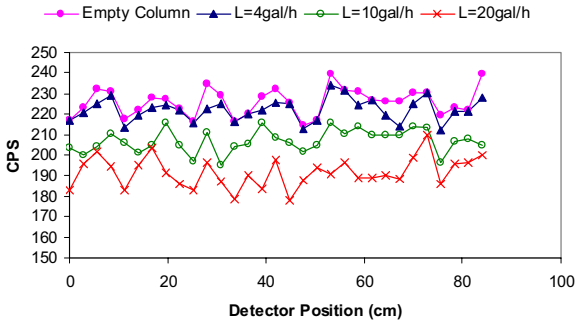
The results from the scans of the column with the CsI(Tl) detector and the sources (^{60}Co, ^{131}I and ^{99m}Tc) are shown in Fig.3. The Fig. 3 (a), (b) and (c) shows the average radiation intensity transmitted for different liquid flow rates: *L*= 4 gal/h (0.93 kg m^{-2} s^{-1}); L=10 gal/h (2,31 kg m^{-2} s^{-1}); and *L*=20 gal/h (4,61 kg m^{-2} s^{-1}). The data were analyzed using F–test to compare the standard deviation and T-test to compare the means with p=0.05 significance. The standard deviation error was less to 0.4%. For each scan lines of head and detector positions, the attenuation was first recorded and then the chord-averaged liquid holdup was calculated according to Eq. (9).



(a)



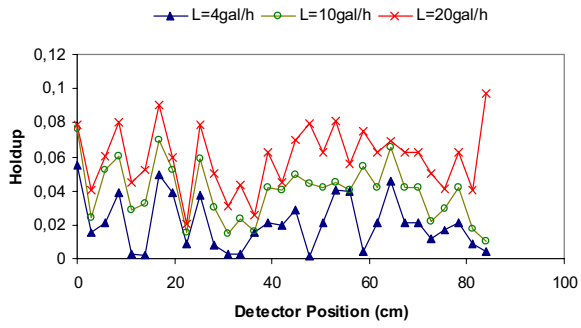
(b)



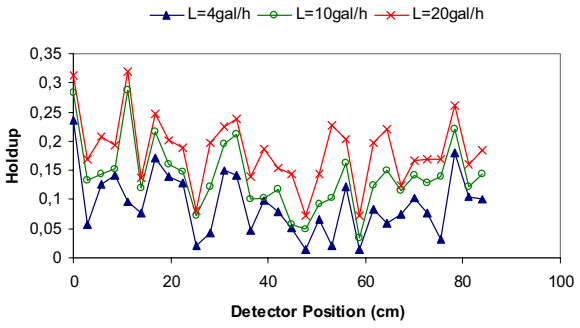
(c)

Fig. 3 Transmission radiation intensity for: (a) ^{60}Co, (b) ^{131}I and (c) ^{99m}Tc.

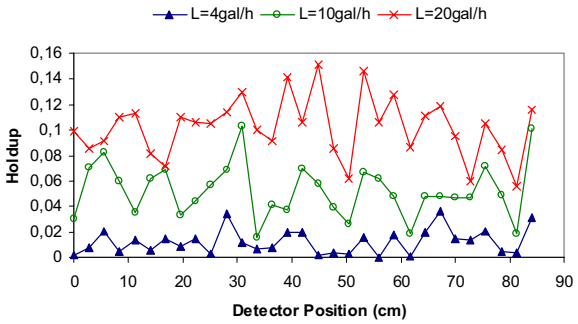
Fig. 4 shows the comparison of line-averaged holdup for 11.1 MBq ^{60}Co (1133 keV), 74 GBq ^{131}I (380 keV), and 740 GBq ^{99m}Tc (140 keV) sources. It is clear that the liquid holdup distribution tends to be more uniform at a higher liquid flow rate, with a lower variance, improving for the ^{99m}Tc source. It can be concluded that the higher liquid flow rate tends to increase the liquid radial spreading and smooth out the local flow irregularities. Liquid holdup has been experimentally shown to increase with increasing liquid flow rate [1, 11], and this has been correctly captured by the gamma ray scanning, as can be seen from Fig. 4.



(a)



(b)



(c)

Fig. 4 Comparison of scan line averaged liquid holdup for different flow rates for: (a) ^{60}Co, (b) ^{131}I and (c) ^{99m}Tc.

Table I shows the average holdup results compared with the literature values. In this specific case, the better values were obtained using ^{99m}Tc source, because it had low energy, compared with the others radioisotopes and this propriety was more adequate to study the used system due to the small dimensions of the column and the material to be of glass.

TABLE I
COMPARISON OF LIQIUD HOLDUP BY THIS STUDY WITH LITERATURE VALUES

Sources	Holdup		
	L=4gal/h	L=10gal/h	L=20gal/h
^{60}Co	0,021	0,039	0,059
^{131}I	0,092	0,140	0,184
^{99m}Tc	0,013	0,052	0,102
Literature value [11]	0,01	0,058	0,123

IV. CONCLUSION

The liquid holdup and its distribution in a packed column filled with glass Rashig rings have been studied as functions of operating conditions at using gamma ray scanning. A radiation transmission mathematic relation has been used to calculate the spatial variation of liquid holdup trough the column cross section. It was found that liquid holdup distribution in randomly packed columns is not uniform and strongly depends on liquid distributor design and the regime flow. Therefore they can be used as the basic tools for the design and scale-up of the packed columns.

V. REFERENCES

[1] H. Z. Kister, *Distillation Design*, McGraw-Hill, New York, 1992.
[2] J.C. Elgin, F.B. "Weiss, Liquid holdup and flooding in packed towers" *Ind. Eng. Chem.*, vol. 31, pp. 435–445, 1939.
[3] B.W. Jesser, J.C. "Elgin, Studies of liquid holdup in packed towers Part I Relations of operating holdup on various packing materials to liquor velocity and physical properties" *Trans. AIChE*, vol. 39, pp. 277–298, 1943.
[4] H.L. Shulman, C.F. Ullrich, N. Wells. "Performance of packed columns I. Total, static, and operating holdups" *AIChE J.*, vol.1, pp. 247–253, 1955.
[5] C.J. Hoogendoorn, J. Lips "Axial mixing of liquid in gas–liquid

flow through packed beds" *Can. J. Chem. Eng.,* June, pp. 125–131F, 1965.
[6] W.P.M. van Swaaij, J.C. Charpenti er, J. Villermaux "Residence time distribution in the liquid phase of trickle flow in packed columns" *Chem. Eng. Sci.* vol. 24, pp. 1083–1095, 1969.
[7] M.M. Farid, D.J. Gunn "Dispersion in trickle and two-phase flow in packed columns" *Chem. Eng. Sci*. vol. 34, pp. 579–592, 1979.
[8] V.E. Sater, O. Levenspiel "Two-phase flow in packed beds" *I&EC Fundam*. vol. 5, pp. 86–92, 1966.
[9] A. Bennett, F. Goodridge "Hydrodynamic and mass transfer studies in packed absorption columns Part I: Axial liquid dispersion" *Trans. IchemE* vol. 48, pp. T232–240, 1970.
[10] C.N. Schubert, J.R. Lindner, R.M. Kelly "Experimental methods for measuring static liquid holdup in packed columns" *AIChE J.* vol. 32, pp. 1920–1923, 1986.
[11] K.B. Kushalkar, V.G. Pangarkar "Liquid-holdup and dispersion in packed columns" *Chem. Eng. Sci.* vol. 45, pp. 759–763, 1990.
[12] D. Toye, P. Marchot, M. Crine, A.-M. Pelsser, G. L'Homme "Local measurements of void fraction and liquid holdup in packed columns using x-ray computed tomography" *Chem. Eng. Process*. Vol. 37, pp. 511–520, 1998.
[13] M.J. Fulham, V.G. Hulbert "Gamma scanning of large towers" *Chem. Eng. Prog*. vol. 71, pp. 73–77, 1975.
[14] W.A.N. Severance "Advances in radiation scanning of distillation columns" *Chem. Eng. Prog*. vol. 77, pp. 38–41, 1981.
[15] W.A.N. Severance "Differential radiation scanning improves the visibility of liquid distribution" Chem. *Eng. Prog.* vol. 81, pp. 48–51, 1985.
[16] M.E. Harrison "Gamma scan evaluation for distillation column debottlenecking" *Chem. Eng. Prog*. vol. 86, pp. 37–44, 1990.
[17] J.D. Bowman "Use column scanning for predictive maintenance" *Chem. Eng. Prog.* vol. 87, pp. 25–31, 1991.
[18] J.D. Bowman "Troubleshoot packed towers with radioisotopes" *Chem. Eng. Prog*. vol. 89, pp. 34–41, 1993.
[19] M. Perez, R. Duarte, L. Pless "Temperature and gamma scans identify problems in crude tower" *Oil Gas J.* vol. 97, pp. 44–51, 1999.