X-ray tomography: a tool for revealing local distribution of liquid in structured packed columns

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Contents

- Introduction
- Experimental setup
- Results
  - Global hydrodynamic quantities
  - Influence of viscosity
  - Flow morphology
- Conclusions
Structured packed columns

Packed column applications
- Gas-liquid absorption ⇔ CO₂ capture
- Solvent stripping ⇔ regeneration
- Distillation

High performance structured packings
- High void fraction
  - low pressure drop ⇔ high capacity
- High specific surface area
  - high mass transfer properties

Liquid flow hydrodynamics
- Liquid holdup
- Gas – liquid interfacial surface area
- Liquid flow morphology
  (films, rivulets…)

Reliable predictive models
⇔ Characterisation of the fluid distribution
down to a very local scale

⇒ X-ray tomography
**ULG X-ray tomograph**

**Operating modes**
1. tomographic mode
2. radiographic mode

**X-ray source**
- Voltage: 30 - 420 kV
- Current: 2 - 8 mA
- Focal spot: 0.8 mm
- Collimator: Pb
- Fan beam geometry
- Angle: 40° - Thickness: 1 mm

**Linear detector**
- 1280 high energy photodiodes
- Pitch: 0.4 mm – H: 0.6 mm

**Rotating table**
- 360° object rotation
- (time = 45 s)
- Max diam: 0.45 m
- Max. height: 3.8 m

**Packed column**

Radiography of the column (transparent PVC)

- Liquid distributor
- Pall rings
  - \( H_{\text{Pall}} = 800 \text{ mm} \)
- 4 MellapakPlus 752 elements rotated by 90°
  - \( H_{\text{Mellapak}} = 800 \text{ mm} \)

\[ \text{Liquid:} \quad \begin{align*}
\mu & = 1 \text{ mPa.s} \\
\mu & = 10 - 20 \text{ mPa.s} \\
\end{align*} \]

\[ \text{Flowrate:} \quad \dot{u}_L = 0 - 25 \text{ m³/m².h} \]

**Operating conditions**

**Liquid**
- Water
- Glycerine solutions

**Gas**
- Air

**Packed column dimensions**
- \( D_{\text{bed}} = 100 \text{ mm} \)
- \( H_{\text{bed}} = 1600 \text{ mm} \)

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Structured packing

**MellapakPlus 752.Y geometrical properties**
*(Sulzer Chemtech)*

- Packing element height: 0.2 m
- Packing element diameter: 0.10 m (0.09 m)
- Specific surface area: 510 m²/m³
- Void fraction: 97.5%
- Corrugation angle: 41°
- Corrugation base: 9.85 mm
- Corrugation height: 6.50 mm

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Image of dry packing image

Mellapak 752Y

(1) Column wall
(2) Corrugated sheet
(3) Wall wiper
(4) Hole

Aferka et al., 2010, Chem. Eng. Sci., 65, 511–516
Methodology

Water
\[ u_L = 23 \text{ m}^3\text{m}^{-2}\text{h}^{-1} \]
No gas flow

Grey = solid (binary image)
Blue = liquid (binary image)

Images of liquid distribution

Liquid distribution in cross sections

Mellapak 752Y
Water
\[ u_L = 23 \text{ m}^3\text{h}^{-1} \]
No gas flow
Uniform liquid distribution
Film flow predominates
At contact points between sheets, rivulets and/or flooded channels

Image processing

Objective = quantitative analysis

1. Normalisation (partial volume effect)
   - convert grayscale values into liquid holdup values
   - divide each pixel value by the value of a pixel completely filled by liquid

\[
\text{liquid holdup} = \frac{\text{pixel value}}{\text{value of a pixel completely filled by liquid}}
\]

→ Distribution of liquid holdup

2. Counting of interfacial pixels (liquid – gas)
   → Distribution of gas-liquid interfacial area

Viva et al., 2011, Flow Meas Instrum, 22, 279-290

Method validation: liquid holdup

Comparison to experimental data
(global values averaged on 70 sections)

Very different methods:
- quick closing valves + weighing
- Tomo + geometrical

Viva, 2008, PhD thesis, University of Pisa
**Method validation : G-L interfacial area**

**Comparison to experimental data**

(global values averaged on 70 sections)

Very different methods:
- chemical
- geometrical


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**Influence of liquid viscosity**

Mellapak 752Y

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H$</td>
<td>300 mm</td>
</tr>
<tr>
<td>$u_L$</td>
<td>17 m$^3$m$^{-2}$h$^{-1}$</td>
</tr>
<tr>
<td>$u_G$</td>
<td>0</td>
</tr>
</tbody>
</table>

Water

- $\mu = 1$ mPa.s
- Uniform liquid distribution
- Thicker liquid films
- Larger number of flooded channels between sheets

Aqueous solution of glycerine

- $\mu = 10$ mPa.s

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On liquid holdup

Each point = one operating condition
= averaged value over the whole packed bed (70 sections)


On G-L interfacial area

Each point = one operating condition
= averaged value over the whole packed bed (70 sections)
**On axial profiles**

**Liquid hold-up**

![Graph showing liquid hold-up with different liquid viscosities (1 mPas, 10 mPas, 20 mPas) across different elevation levels (M1, M2, M3, M4).]

\[ u_L = 17 \text{ m}^3/\text{m}^2\text{h} \]

70 cross-section images for each operating condition

**Gas-liquid interfacial surface area**

![Graph showing gas-liquid interfacial surface area with different liquid viscosities across different elevation levels (M1, M2, M3, M4).]

\[ u_L = 17 \text{ m}^3/\text{m}^2\text{h} \]

**Liquid flow morphology**

**Flow structures**

- **Films**: Ideal morphology
- **Contact point flow**: Liquid mixing
- **Flooded channels**: No transfer

![Diagram illustrating liquid distribution and binary image with flow structures marked.]
Flow structure classification

= Iterative method

Step 1 = Separation and labelling of each flow structure

Step 2 = Classification based on the size and shape

SIZE

Minimum and maximum Feret diameters ($F_{\text{min}}$ and $F_{\text{max}}$) computed on all flow structures

$F_{\text{min}}$ and $F_{\text{max}}$ = dimensions of the minimal enclosing parallelogram


Flow structure classification

Step 1 = Separation and labelling of flow structures

Limited spatial resolution of the ULG X-ray CT

Distinct flow structures may be adjacent

« seen » as a single structure

Criterion for structure splitting

$$\frac{S_{\text{Flow_struct}}}{S_{\text{Feret \_rect}}} < 0.5$$

Elimination of pixels with the smallest number of neighbors
( erased pixels not lost = arbitrarily added to the main flow pattern = film flow)

Flow structure classification

Step 2 = Classification based on the size and shape

**Flooding channels**
Thicknes $\geq$ distance between packing sheets

$F_{\text{min}} > 9 \text{ mm}$

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Flow structure classification

**Step 2 = Classification based on the size and shape**

**Contact point flow**

« Round » shape

$\frac{F_{\text{max}}}{F_{\text{min}}} < 2$

and

$F_{\text{min}} < 9 \text{ mm}$
**Flow structure classification**

**Step 2** = Classification based on the size and shape

**Film flow**

- Elongated thin shape
- \( \frac{F_{\text{max}}}{F_{\text{min}}} > 2 \)
- and
- \( F_{\text{min}} < 9 \text{ mm} \)
- + all remaining pixels (including erased pixels from structure separation)

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**Flow morphology: results**

- Flooding channels
- Contact points
- Films

\[ \mu = 1 \text{ mPa.s} \]
\[ u_L = 17 \text{ m}^3/\text{m}^2\text{h} \]

\[ \mu = 10 \text{ mPa.s} \]
\[ u_L = 17 \text{ m}^3/\text{m}^2\text{h} \]

\[ \mu = 20 \text{ mPa/s} \]
\[ u_L = 17 \text{ m}^3/\text{m}^2\text{h} \]
**Flow morphology**

![Graph showing flow morphology](image)

Films

Contact points

Flooded channels


**Influence of liquid flowrate**

![Graph showing influence of liquid flowrate](image)

M1

M2

M3

M4

Floated channels

Contact points

Films

\( \mu = 1 \text{mPa.s} \)
Influence of liquid viscosity

\[ u_L = 17 \text{ m/h} \]

Flooded channels  Contact points  Films

Hydrodynamic Analogy model

G-L interfacial surface area
\[ \Leftrightarrow \text{Wetting efficiency} \]
\[ \Rightarrow \text{fraction of irrigated and non-irrigated channels} \]

Liquid holdup
\[ \Rightarrow \text{total amount of liquid} \]

Liquid flow morphology
\[ \Rightarrow \text{Flooded channels fraction} \]
\[ \Rightarrow \text{Contact point flow fraction} \]
\[ (\Leftrightarrow \text{mixing length : } z_L) \]

\[ z_C = \text{gas mixing length (packing geometry)} \]

Shilkin et al., 2006, AIChE J., 52, 3055-3066

\[ z_L = \text{dist. between contact points} \]
\[ \text{contact point flow fraction} \]
**Model validation: mass transfer**

- F-factor = \( u_G \rho_G^{0.5} = 0.63 \text{ Pa}^{0.5} \)
- CO\(_2\) desorption from water

\[
k_L a = \frac{u_L}{H_{pack}} \ln \left( \frac{x_{top} - x^*_{top}}{x_{bot} - x^*_{bot}} \right)
\]

**Conclusions**

**Tomographic measurements**
Quantitative assessment of the influence of **liquid load** and **liquid viscosity** on
- G-L interfacial surface area
- Liquid holdup
- Flow morphology (films, rivulets, flooded)

**Global values**
**Spatial distributions**

**Mass-transfer model**
- Based on an Hydrodynamic Analogy
- Validated by mass transfer experiments
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