

New Calibration Technique for X-ray Absorption Studies in Single and Multiphase Flows in Packed Bed

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An X-ray technique has been developed to determine the concentration and liquid holdup in a packed bed. To quantify these parameters in single and multiphase flow systems, at local level, a calibration method/graph has been developed. This graph has also been used to obtain the local concentration value of a liquid in presence/absence of another miscible liquid in a porous media. The same graph has been used to quantify the local liquid holdups (static and dynamic) in a packed bed. The same technique can be used for other radiological methods such as gamma rays and neutron radiography. Results obtained from this technique are in agreement with the published data.

KEY WORDS: X-ray visualization; local concentration; local liquid holdups; porous media; calibration graph; trickle bed.

1. Introduction

Flow visualization in porous media is very important to understand the internal state of the packed bed fundamentally and to improve the existing numerical models so that they can predict more accurately.^{1,2)} X-ray radiography and fluoroscopy have been used extensively^{3–5)} to study/visualize the fluid flow, liquid holdups, bed porosity and dispersions phenomena in the packed beds besides some other techniques such as nuclear magnetic resonance,^{6,7)} gamma rays⁸⁾ and neutron attenuation.⁹⁾ However, there are inherent problems associated with the use of X-rays such as polychromatic nature of X-rays from the medical and industrial units, accurate measurement of mass absorption coefficient, and point source emission of X-rays.

As such in many X-rays studies the above problems either have been neglected or have been simplified or both.^{3–5,8)} This approach can contribute towards inaccuracy in the results obtained from these studies. However, it is permissible to neglect the above effects if one is interested in the qualitative analysis.^{3,10)} For the quantitative analysis, one has to take care of all these points. Few of the researchers^{4,5,8,11)} have considered some of these points but their solution was problem specific which may not be applicable to other systems. Recently, Tidwell *et al.*^{12,13)} have obtained quantitatively the relative concentration of a solution at a particular point with respect to the saturation concentration at that point in a porous rock sample from the stills images of X-ray. However, data interpretation from the relative concentration would be difficult. Besides this, there are several problems associated to stills X-ray image from image taking to development to image capturing which may change the results significantly. In fact, many of these problems have been discussed by them. Tidwell *et al.*

have used a simple theory to obtain the concentration with respect to saturated concentration in the porous media. However, this technique may not be applicable if there are different miscible fluids present together in a system. They did not carry out the liquid holdup study and the polychromatic nature of X-ray was also not considered.

In this article, we are presenting a method which will not only take care of polychromatic nature of the X-ray and thus the mass absorption coefficient to get the local concentration of a fluid in a packed bed but also would be able to provide the local concentration in presence of another miscible fluid. It will be shown later that the same method can be applied to measure the local and global liquid holdups in a packed bed. In this article, first we present a simple theory of X-rays absorption and associated difficulties with it. Calibration procedure and graph are described in the later sections followed by the application of the graph in measuring the local concentration/liquid holdups in a packed bed.

2. X-ray Absorption

When a monochromatic X-ray beam passes through a substance, it absorbs a fraction of incident beam and is expressed by Beer–Lambert's law.^{14–16)}

$$I = I_0 e^{-(\mu/\rho)\rho x} \dots \dots \dots (1)$$

Equation (1) represent a relation between the incident (I_0) and transmitted intensity (I) of a monochromatic X-ray beam through a homogenous substance of thickness (path length) x . μ/ρ is the mass absorption coefficient of the material. It depends on the substance considered and the wavelength of X-rays. ρ is the density of the material. The mass absorption coefficient is a function of the wavelength and atomic number of the material.

If a substance contains more than one element, its mass absorption coefficient could be considered as the weighted average of the mass absorption coefficients of its constituent elements.¹⁴⁻¹⁶ Hence, for a monochromatic X-ray beam through a composite substance, Eq. (1) would take the following form:

$$I=I_0 e^{-[w_1^*(\mu/\rho)_1+w_2^*(\mu/\rho)_2+w_3^*(\mu/\rho)_3+\dots+w_n^*(\mu/\rho)_n] \rho x} \dots\dots\dots(2)$$

Where the subscripts 1, 2, 3, ..., n indicate the chemical species present in the sample, w₁ is the weight fraction and (μ/ρ)₁ is the mass absorption coefficient of the chemical specie 1 and so on.

All the industrial and medical radiography units emit polychromatic X-rays, and then the variation in the mass absorption coefficient with the wavelength has to be taken into consideration. Equation (3) shows a relation between incident (I₀) and transmitted intensity (I) of a polychromatic X-ray beam through a homogenous substance (comprises of one element only) of thickness x.

$$I=I_0 e^{-((\mu/\rho)_{\lambda_1}+(\mu/\rho)_{\lambda_2}+\dots+(\mu/\rho)_{\lambda_n}) \rho x} \dots\dots\dots(3)$$

λ₁, λ₂, λ₃, ..., λ_n represents the wavelength distribution of polychromatic X-rays and (μ/ρ)_{λ₁}, (μ/ρ)_{λ₂}, (μ/ρ)_{λ₃}, ..., (μ/ρ)_{λ_n} are the corresponding mass absorption coefficients.

Usually, to enhance the visualization of liquid, a contrast agent, called the dopant, is used. Hence, there will be more than one chemical species in the flow system. Therefore, the problem of X-ray absorption becomes more complex and can be represented by a general formula¹⁴) as given below.

$$I=I_0 e^{-[(w_1^*(\mu/\rho)_1+w_2^*(\mu/\rho)_2+w_3^*(\mu/\rho)_3+\dots+w_n^*(\mu/\rho)_n) \lambda_1+(w_1^*(\mu/\rho)_1+w_2^*(\mu/\rho)_2+w_3^*(\mu/\rho)_3+\dots+w_n^*(\mu/\rho)_n) \lambda_2+\dots+(w_1^*(\mu/\rho)_1+w_2^*(\mu/\rho)_2+w_3^*(\mu/\rho)_3+\dots+w_n^*(\mu/\rho)_n) \rho x} \dots\dots\dots(4)$$

The X-rays generated in industrial and medical radiography units consist of a mixture of different wavelength, and the variation of intensity with wavelength is found to depend on the X-ray tube voltage. When using polychromatic X-rays for flow visualization applications, attempts to calculate mass absorption coefficient (of each species), which relates transmitted and incident intensity to concentration is not only very complicated but also less accurate. The main uncertainty is to acquire the wavelength distribution and corresponding X-ray intensities. Published mass absorption

coefficients are not yet accurate enough at most wavelengths.¹⁴⁾ Moreover, the mass absorption coefficient of many of the chemical species is not yet known. Therefore, a simple method is needed which can take care of the above mentioned problems in a reasonable way and can be used in visualization study to get the quantitative information. To overcome these problems a calibration technique has been developed which is described below.

3. Calibration for Concentration/Holdup and Related Studies

X-ray absorption imaging produces two-dimensional images that reveal the interior of an object. These images are a point array with each point assigned a gray-level between 0 and 255 according to the transmitted X-ray intensity. The gray scale intensity data from these images are related to X-ray attenuation by the specimen. A major requirement in such studies is to relate the X-ray intensity variations captured in the images to changes occurring in the test section. This idea has been exploited in calibration.

Let us consider an example of trickle bed reactor in which the objective is to find out the liquid distribution inside the test media. In X-ray visualization studies, a liquid with the dopant, which enhances the flow visualization, is used. **Figures 1(a)** and **1(b)** show the images of a portion of a trickle bed under dry and fluid flow conditions. Dark regions in Fig. 1(b) are due to the presence of dopant, barium chloride. Even though the concentration of the liquid trapped in the test sample under both (dynamic and static) conditions is same as its original concentration (steady state), the volume trapped or path length differs. This leads to different intensity distribution in the X-ray images of the test media as shown in Fig. 1. A calibration graph has been developed which captures this intensity variation with the X-ray path length. This X-ray path length data could be related to the liquid volume trapped and thus the concentration.

3.1. Theory

Consider the X-ray image of an object. This object is superimposed with some other material under the same operating conditions (object position is same in both the cases) and a composite X-ray image is taken. In principle, the composite X-ray image can be split into two, image of the

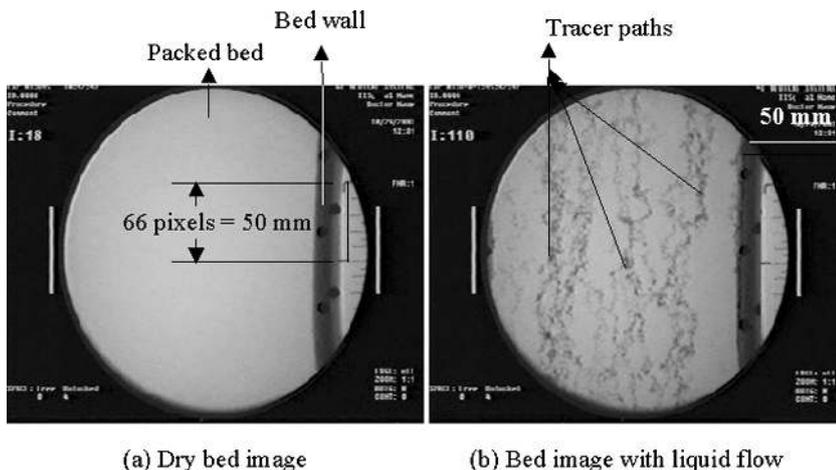


Fig. 1. Images of a portion of packed bed under different conditions.

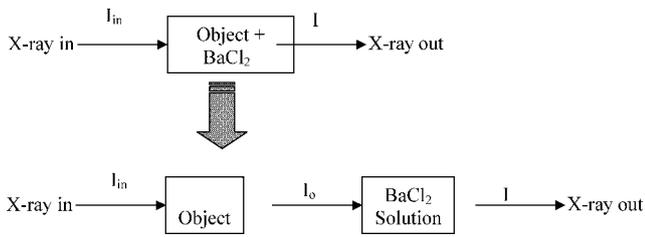


Fig. 2. Concepts used in preparation of calibration.

object and that of the material. This concept has been used in preparing the calibration graph. **Figure 2** shows that X-ray image of any test media containing a solution (BaCl_2) could be visualized as the sum of the X-ray image of the object and the solution in series. As such the theory developed here is on the same line as described by Haw *et al.*¹⁰⁾ for gamma rays absorption with some modification.

For an aqueous barium chloride solution containing more than one chemical (such as water and barium chloride) species, Beer-Lamberts law could be written as^{14,15,16)}:

$$I = I_0 e^{-(\mu/\rho)\rho x} \dots\dots\dots(1)$$

Where,

$$\frac{\mu}{\rho} = \left(W_{\text{water}} \left(\frac{\mu}{\rho} \right)_{\text{water}} + W_{\text{BaCl}_2} \left(\frac{\mu}{\rho} \right)_{\text{BaCl}_2} \right) \lambda_i, \quad (i=1,2,\dots,n) \dots\dots\dots(5)$$

To overcome the unavailability of wavelength distribution, we have used the same operating conditions for calibration and concentration experiments and it was assumed that this distribution would remain same as long as the operating conditions are same.

Let us consider a packed bed made of some particulate material (beads) through which liquid is flowing. If one scans this system in various conditions using X-ray then one can write the following expressions. Assuming negligible absorption of X-rays by air then for empty column Beer Lambert's law will give

$$\ln(I_1/I_0) = -\mu_1 2x_1 \quad (\text{for empty column}) \dots\dots\dots(6)$$

Where, I_1 and I_0 are the incident and transmitted intensity of X-ray beam respectively. μ_1 is the absorption coefficient of the column's material and x_1 is the thickness of the column material.

Similarly, one can write

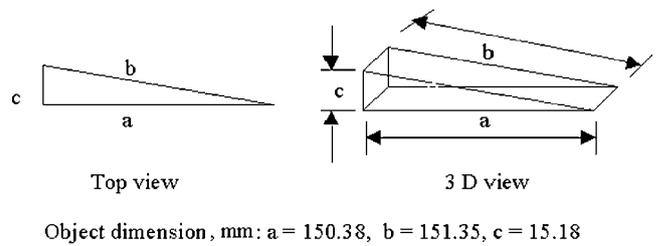
$$\ln(I_1/I_0) = -\mu_2 x_2 \quad (\text{for beads}) \dots\dots\dots(7)$$

and,

$$\ln(I_3/I_2) = -\mu_3 x_3 \quad (\text{for liquid}) \dots\dots\dots(8)$$

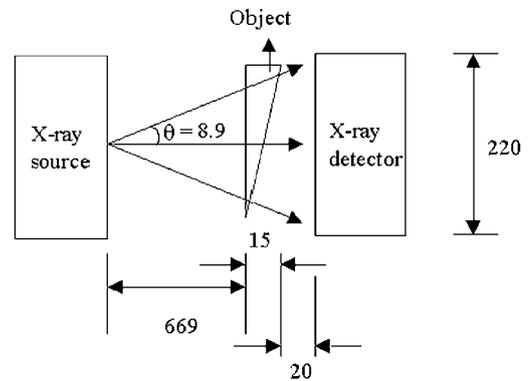
Where, μ_2 and μ_3 are absorption coefficient of the beads and liquid respectively. And x_2 and x_3 are the thickness of the beads and liquid respectively. Note that absorption coefficient is different than mass absorption coefficient. Using the above equations, I_1 and I_2 can be eliminated and the resulting equation can be written as

$$I_3 = I_0 e^{-(\mu_1 2x_1 + \mu_2 x_2 + \mu_3 x_3)} \dots\dots\dots(9)$$



Object dimension, mm: a = 150.38, b = 151.35, c = 15.18

(a)



All dimensions are in mm and θ is in degrees

(b)

Fig. 3. (a) Object used for calibration. (b) Experimental arrangement for calibration.

Or,

$$I_3 = I'_0 e^{-(\mu_3 x_3)} \dots\dots\dots(10)$$

Where,

$$I'_0 = I_0 e^{-(\mu_1 2x_1 + \mu_2 x_2)} \dots\dots\dots(11)$$

Because, x_1 and x_2 are constant throughout the experiments. Therefore, from the X-ray image of empty column filled with beads, one can get the gray scale values which in principle would be constant in all the experiments. So I'_0 in terms of gray level is known. Therefore, if liquid is flowing through the packing then one has to know I_3 for various path length x_3 (thickness) of the liquid. So a plot between $\ln I'_0 - \ln I_3$ against path length (x_3) will give a straight line which passes through the origin. The above concept has been applied in preparing the calibration graph which is discussed below.

3.2. Object Used for Calibration

A triangular box, made of 10 mm thick Perspex sheet was used for the calibration purpose as shown in **Fig. 3(a)**. Note that the same material was used to fabricate the actual experimental setup for the local concentration measurement. The internal dimensions of calibration box were ($a=150.38$ mm, $b=151.35$ mm, $c=15.18$ mm) measured using digital Vernier caliper. The calibration graph was prepared at the same operating current and voltage as those were used in concentration measurement experiments. Many precautions such as accurate measurement of the dimensions of object, and placing of the object at the same position between the X-ray source and detector each time, were taken to get the correct calibration graph.

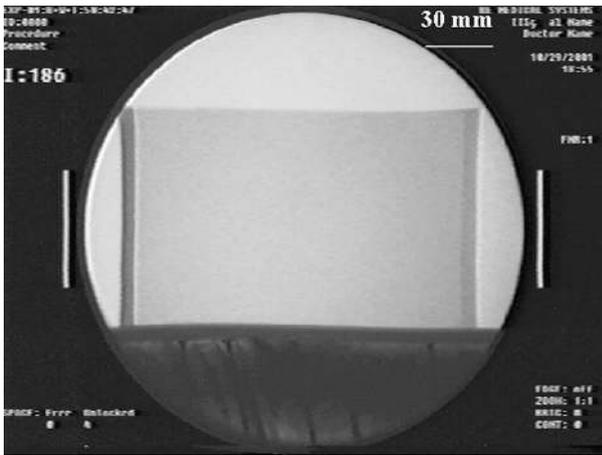


Fig. 4. X-ray absorption image of blank object.

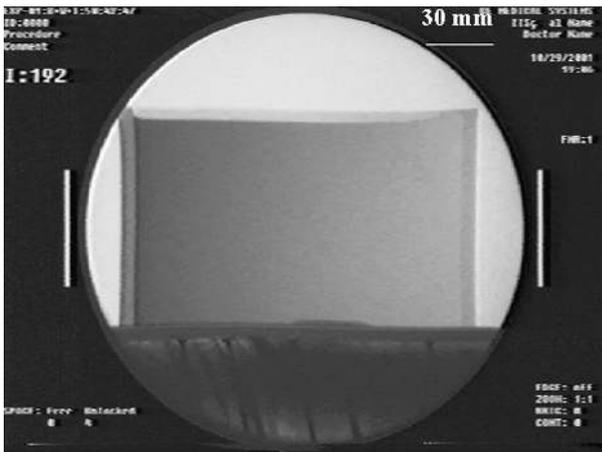


Fig. 5. X-ray image of object filled with aqueous barium chloride solution.

3.3. Procedure for Calibration

Operating conditions of the X-ray unit were set to desired values, at which actual experiments have been done. The object was mounted on a flat fixed table. The position of the X-ray source and detector was adjusted so as to get the full view of the object, as shown schematically in Fig. 3(b). X-ray emission was switched on and an image of the object without any liquid was taken. The object in this position was filled with barium chloride solution of the same concentration as the one used in the packed bed for concentration experiments. X-ray source was switched on so as to get the image of the object filled with barium chloride solution. X-ray images of blank object and object filled with barium chloride solutions are shown in Figs. 4 and 5 respectively. All the X-ray images were recorded on a video cassette using a video cassette recorder and then converted into snapshots/frames using Dazzle hardware and Dazzle-Movie-Star software. Gray scale intensity data of an image (in the form of arrays) were obtained using IMREAD function of MATLAB software. IMREAD function reads the gray scale intensity corresponding to the pixels in the image into an array. If one subtracts Fig. 4 from Fig. 5 then one will get the gray scale values of liquid only. Let us say that I_0 and I are the gray scale intensity arrays corresponding to the blank object image and object image filled with barium chloride respectively. Then $\ln(I_0/I)$, can be plotted

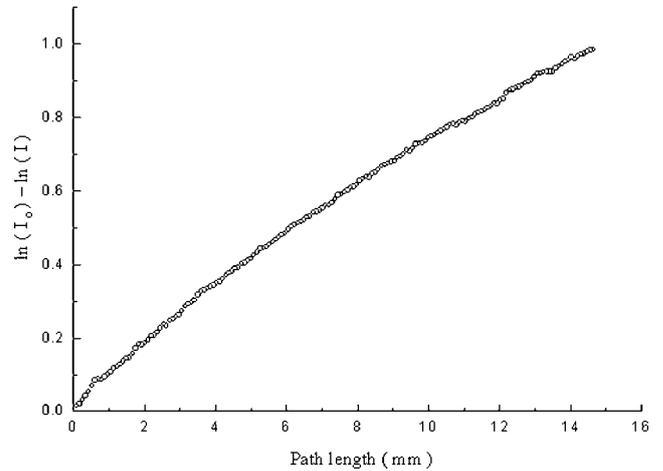


Fig. 6. Calibration graph used for holdup measurement.

against path length (or liquid thickness) as shown in Fig. 6. Path length data are obtained from the dimensions of the object. Figure 6 shows a straight line passing through the origin. The slope of the curve gives the value of composite absorption coefficient. Nature of the graph is in the same line as predicted by theory, discussed above.

The parameters, which can change the nature of the calibration, are the concentration of the solution, and current & voltage at which X-rays are emitted. A fresh calibration graph has been prepared for each of the concentration experiments though the above mentioned operating parameters have been kept constant. It was found that if all the parameters, mentioned above, are kept constant during all the experiments then there is no need to prepare separate calibration graph for each experiment. X-ray exposure-time experiments on test sample filled with $BaCl_2$ solution showed a minimum exposure time of 2 s. In all the experiments, the exposure time used was more than 2 s. Total time taken by X-ray to scan the whole bed in fluoroscopy mode by continuous emission of X-ray was approximately 15 min.

The above calibration graph is applicable to estimate the local concentration and liquid holdups only for one liquid *i.e.*, single phase flow in porous media. However, if there are two miscible liquids present simultaneously in a porous media (*i.e.* more than one phase are present) then one has to use a different calibration graph. In the present system, we have aqueous barium chloride solution which is dispersing in the water and miscible. For this system, to create a calibration graph, following approach has been adopted.

Same calibration box was used as described before. An empty X-ray image (Fig. 4) of the box was taken as before without any liquid. Under the same conditions the object is filled with de-ionized distilled water and the X-ray emission was switched on to take the image of the object with just water. Object was thoroughly cleaned and dried after each image was taken and was placed exactly at the same position each time. The empty box was filled with barium chloride solution of concentration 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 14, 16 g in 100 mL of de-ionized distilled water. For each concentration one X-ray image was taken. Few X-ray images of the object are shown in Fig. 7.

The calibration graph obtained from these images is shown in Fig. 8. The use of this graph to obtain the concen-

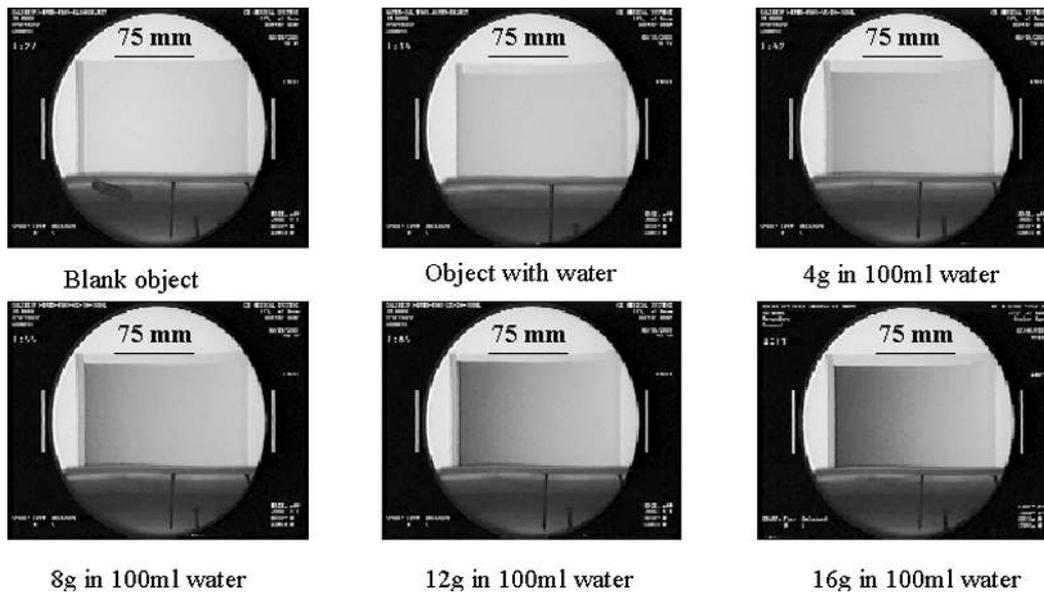


Fig. 7. X-ray images of the object filled with different concentration of barium chloride solution.

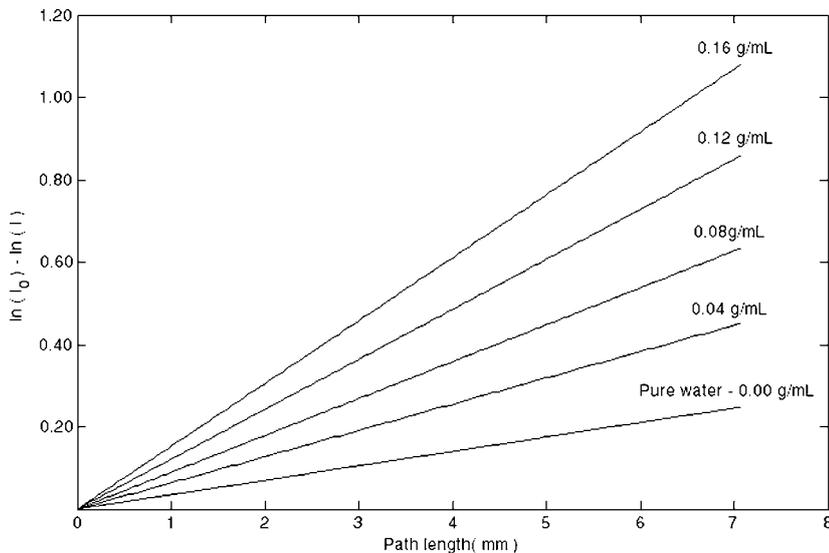


Fig. 8. Calibration graph used for tracer concentration measurement.

tration data is discussed in Sec. 5.

4. Concentration Experiments

Tracer concentration measurement has been a part of dispersion, holdup studies in packed beds.¹⁷⁻²¹⁾ Many other fluid flow and mass transfer research also involve the determination of concentration distribution. Use of conductivity probes is one of the techniques, which has been used by few researchers.^{19,20)} The probes are invasive, *i.e.*, they disturb the flow system, and the exact concentration cannot be obtained. Location of the probe also matters; many times the probe may not have access to the regions of tracer flow, as the flow in the packed bed is very much localized due to channeling, stagnant zones and other irregularities. Hence the measured concentration data are prone to inaccuracy. Also, one cannot have as many probes as one wants. The dimensions of the probes used as well as that of the system under investigation limits number of probes, hence the number of data sampling points. However, X-ray method does not have the above mentioned drawbacks and is more

versatile. Therefore, use of X-ray method to quantify the concentration in a packed bed has been demonstrated here along with liquid holdup. The experimental procedure for the concentration measurement, using this method, is described below.

4.1. Experimental Procedure

A Perspex rig of dimensions 700×170×60 mm filled with expanded polystyrene beads has been used to demonstrate the application of the calibration. A schematic diagram of the setup is shown in Fig. 9. An X-ray image of the dry bed was taken. Water was allowed to flow through seven rotameters (at a flow rate of 0.07 lpm through each rotameter) at the top of the bed. Flow through the rotameters, which were uniform distance apart, was monitored regularly. The setup was left in this position for about 30 min to reach the steady state. Bed was imaged under this condition. A step input of the tracer (aqueous barium chloride solution of known concentration, 0.16 g/mL) was introduced from the center inlet at the top of the bed. X-ray image of the bed was imaged again after the steady state

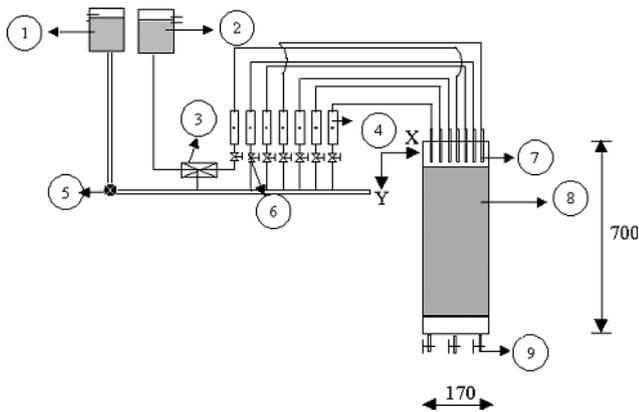


Fig. 9. Experimental arrangement for concentration/holdup measurement.

1. Constant head reservoir for barium chloride/water.
2. Constant head barium chloride tank for tracer dynamics studies.
3. Three way valve.
4. Rotameters.
5. Main liquid flow control valve.
6. Flow controller valves.
7. Liquid inlet.
8. Perspex rig.
9. Liquid drain.

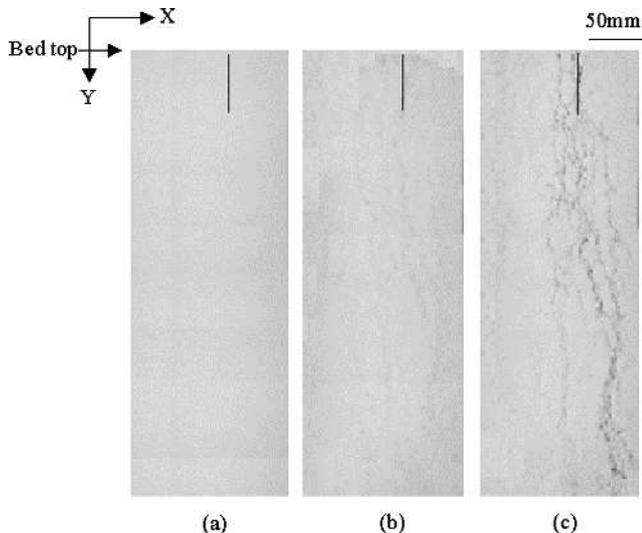


Fig. 10. X-ray images of the bed, bed+water, bed+water+tracer.

was reached. More details of the experiment is given elsewhere.²²⁾

As the tracer flows down, it will mix with water from other inlets, and hence its concentration will also vary. For example, the barium chloride solution used in the experiments was obtained by dissolving 16 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ into 100 mL water. Therefore, one would have the variation in barium chloride concentration in the bed ranging from 0 to 16 g in 100 mL of water. The calibration graph (Fig. 8), developed in Sec. 3, captures this feature. Use of the calibration graph to obtain the concentration is explained below.

5. Use of Calibration Graph

5.1. For Concentration

To illustrate the use of Fig. 8, consider a pixel located at (18, 127) along a line shown in **Fig. 10**. This figure shows the X-ray images of the bed in three conditions *i.e.* dry bed (Fig. 10(a)), bed with water (Fig. 10(b)) and bed with water and barium chloride solution (Fig. 10(c)). The line marked

in these images refer to the same portion of the bed. Each image in Fig. 10 was read using IMREAD function of MATLAB. From the gray level intensity matrix, intensity data corresponding to the pixel (18, 127) *i.e.*, $I_{\text{beads}}(18, 127)$, $I_{\text{water}}(18, 127)$, and $I_{\text{tracer}}(18, 127)$ were found. The bottom most curve, in Fig. 8, was used to get the path length data of water, knowing $(\ln(I_{\text{beads}}) - \ln(I_{\text{water}}))$. For the pixel located at (18, 127), $(\ln(I_{\text{beads}}) - \ln(I_{\text{water}})) = 0.0607$ and the corresponding path length data from the bottom most curve of Fig. 8, is 6.2686 mm. This path length data/value along with the value of tracer concentration intensity, $(\ln(I_{\text{beads}}) - \ln(I_{\text{tracer}})) = 0.2877$, at the same point (18, 127) was used to find out the tracer concentration using Fig. 8. The tracer concentration is found to be 0.15059 g/mL.

In this method, for the concentration calculation, it has been assumed tacitly that the path length for X-ray in the steady state flow of water through the bed is same as that for flow of tracer. To understand this one has to go into experimental details. There are seven rotameters, which monitor the flow of water through the packed bed. When a steady state is reached, the fluid through the center rotameter was changed from water to barium chloride solution using a three-way valve and maintaining the same flow rate as water. The barium chloride solution flowing through the system will follow the same path as its predecessor (water). Therefore, it could be assumed that the path length for X-rays at a particular point for the former case is same as that for a corresponding point in the later case. To verify this assumption, few experiments were done. Water was supplied through one rotameter into the packed bed filled with expanded polystyrene beads through only one inlet. After leaving the system for 30 min to reach steady state, flow was changed to barium chloride solution. Using continuous emission of X-rays in fluoroscopy mode, it was found that barium chloride took only those paths that were established by water which justifies the assumption.

5.2. For Liquid Holdups

The calibration thus developed can be used to obtain holdup of any region in the packed bed. Let consider Fig. 1, where the gray scale intensity array corresponding to dry bed image and in presence of liquid flow be denoted by I_0 and I respectively. Number of arrays in both the images is same, (as the image size is same). From these arrays one can select array elements corresponding to the flow region of interest for further analysis. Consider a pixel located at (214, 99). This pixel falls well within the packed bed region/domain. The liquid holdup calculation for this pixel has been illustrated below.

Figures 11(a) and **11(b)** show the idealization of this pixel. Figure 11 shows the packed region of a bed, which excludes bed wall thickness, and T ($=60$ mm) is the thickness of this packed region. $I_0(214, 99)$ and $I(214, 99)$ respectively correspond to the gray scale intensity of this pixel in the dry bed and in presence of liquid flow images. In Fig. 11(b), X-rays have to penetrate through all the flow lines and thus more X-rays absorption. Therefore, $I(214, 99)$ is less than $I_0(214, 99)$. Sum of the thickness of all the flow lines would be the total X-ray path length due to liquid.

As the slope of the calibration graph ($=0.0728 \text{ mm}^{-1}$) is

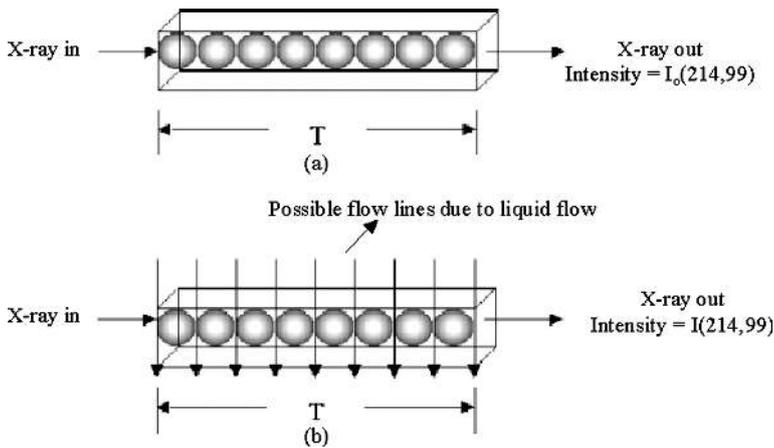


Fig. 11. Idealization of a pixel located at a position (214, 99) in a packed bed.

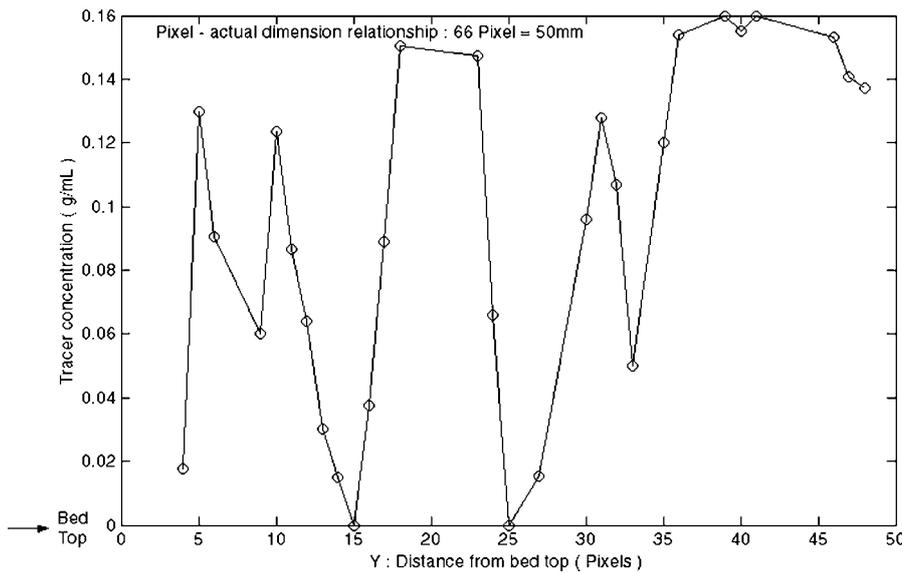


Fig. 12. Tracer concentration data obtained from the images of Fig. 10.

known from Fig. 6, the path length of X-rays through pixel located at (214, 99) can be calculated as follows (using Eq. (10)).

$$\begin{aligned} \text{Path length, } x &= \frac{[\ln(I_0(214, 99)) - \ln(I(214, 99))]}{\text{Slope of the calibration graph, } \mu} \\ &= \frac{[\{\ln(182) - \ln(73)\}]}{0.0728} = 12.5481 \text{ mm} \end{aligned}$$

The liquid holdup within the pixel can be calculated from the following expression.

$$\begin{aligned} \text{Holdup fraction in a pixel} &= \frac{\text{Volume of barium chloride solution in the pixel}}{\text{Volume of the pixel}} \\ &= \frac{1 \text{ pixel} \times 1 \text{ pixel (area of a pixel)} \times 12.5481}{1 \text{ pixel} \times 1 \text{ pixel} \times T} \end{aligned}$$

Where T is the thickness of the bed in mm. As such a pixel has the physical dimensions of 0.757×0.757 mm.

6. Results

6.1. Concentration

For the vertical line shown in Fig. 10, $(\ln(I_{\text{beads}}) - \ln(I_{\text{water}}))$

and $(\ln(I_{\text{beads}}) - \ln(I_{\text{tracer}}))$, were calculated. The local tracer concentration obtained from these data with the aid of Fig. 8 has been plotted in Fig. 12 (concentration data figure). This figure also indicates pixel to actual dimension correlation. It is evident from Fig. 10 that the line passes from no tracer regions to tracer regions of various concentrations; therefore, our concentration data must show this trend which is obvious from Fig. 12. It can be seen from the concentration figure that they vary randomly from 0 to 0.16 g/mL as expected.

6.1.1. Validation of Concentration Data

Two approaches were used to validate the concentration data. In first approach, a region (bed cross section) close to the tracer inlet was selected, where the mixing of tracer with water is minimal. In another approach, a region away from the inlet was chosen where the mixing of water with tracer is high. The overall concentration in both the regions should be same and should be equal to the overall initial concentration. The overall initial concentration of the tracer in the bed can be found by a simple mass balance approach. As we know the total volume of water supply to the bed, the initial tracer flow rate and its concentration, one can calculate overall concentration, which should be same across any cross section of the bed. The average tracer concentration in the bed was found to be 0.0228 g/mL. However, in two-di-

mensional X-ray image one gets the averaged cross sectional data at a given pixel. Therefore, two small regions, which are located at distances 3.2 and 9.3 cm from the top surface of the bed, were considered as shown in Fig. 13. Both the

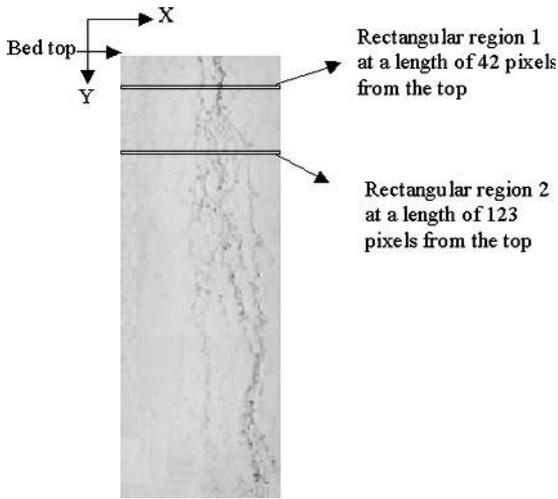


Fig. 13. X-ray image used in concentration data validation.

regions have 3x216 square pixels size (3 pixels along Y direction and 216 pixels along X direction) in order to get the better average values. For regions 1 and 2, the average tracer concentration was found to be 0.0269 and 0.0267 g/mL respectively. These values are very close to the overall average concentration of the bed and thus validating the X-ray technique proposed here.

6.2. Validation of Liquid Holdup

A typical map of the total local liquid holdup is shown in Fig. 14. This image (Fig. 14(a)) was taken at 0.49 lpm (total) flow rate of barium chloride solution in a packed bed of expanded polystyrene beads. The total liquid holdup was found 3.72% of the total bed volume. The total holdup obtained for this flow rate, from published correlations²³⁻²⁵⁾ was found to be in the range 1.21 to 7.62%. Holdup obtained from the conventional drainage method²²⁾ was 3.99%. Thus, the total liquid holdup obtained by X-ray method is well within the range.

One can see many localized regions in Fig. 14(b) where the liquid holdup value is very high. About 390 pixels in the flow domain were found to have a very high total liquid

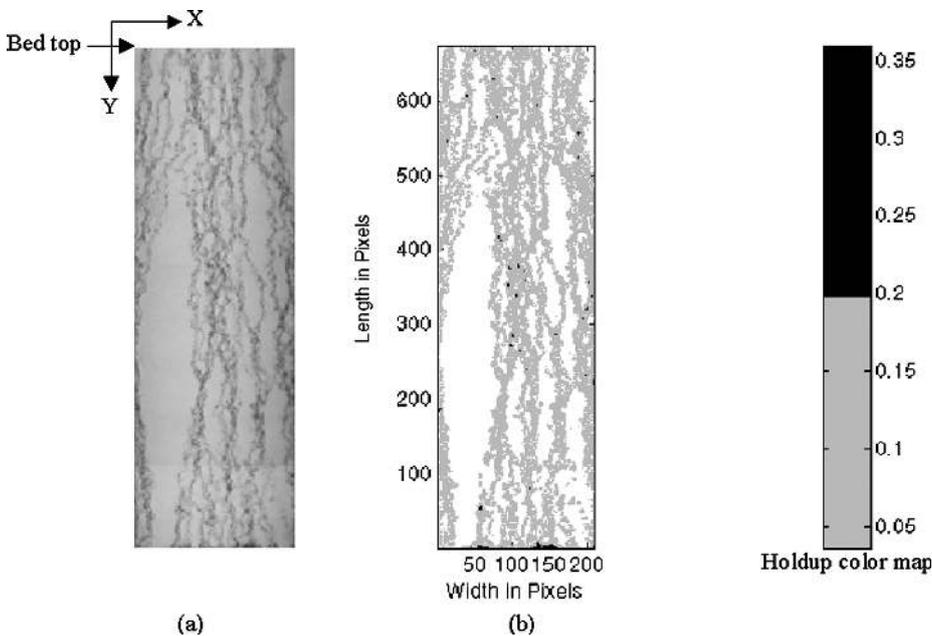


Fig. 14. X-ray image of the bed at 0.49 lpm flow rate under flowing conditions.

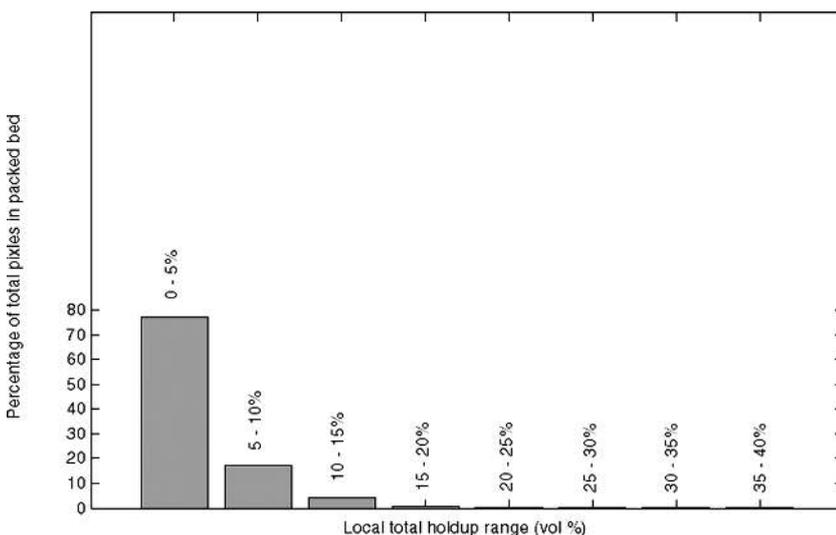


Fig. 15. Bar graph of total local liquid holdup distribution in a packed bed.

holdup of greater than 20%, the maximum being 39.57%. In calculating these holdup values, volume of single pixel has been considered. **Figure 15** is bar chart showing percentage pixels that fall in various liquid holdup ranges. 77.25% pixels of the packed bed were found to have holdup in the range 0–5% and 0.0317% of the pixels had holdup in the range 30–40%.

6.3. Limitations and Source of Errors

Even for blank X-ray images, gray scale intensity profile was found to vary from center to periphery. As the calibration was prepared from a set of images (that of blank object image and of object filled with tracer) by using an operation similar to subtraction, this above-mentioned effect would cancel out. The same technique has been used to obtain the liquid holdup and concentration data from X-ray images. The effect of point source emission (divergence of X-ray beam) on the results was found negligible (relative error was approximately 1%). However, if the sample thickness is more and the distance between the X-ray source and detector is large then this relative error could increase.

One can see from the calibration graph in Fig. 8 that near the origin, curves are too close to distinguished. If the intensity data obtained from X-ray images of the bed are such that, they fall in this region, it becomes too difficult to refer the graph. A higher magnification can then be used to overcome this problem. If one enlarges this region, a lot of small fluctuations in the data can be seen. One should use techniques such as least square fit/data averaging to minimize the errors. In the present study least square fit has been used.

Position of the object between the X-ray source and image intensifier is an important parameter in getting the correct results because it directly affects the image size and thus the intensity.

Also, one should be careful in joining the images. If pixels are not matched properly in joining, then it may lead to unrealistic results. One way to eliminate this error is using the data averaging near the joining edges, such as 3×3-pixel averaging. Then the average value is assigned to the center pixel. This operation is similar to the image filtering²⁶⁾ operation. However, in the present case we have used pixel to pixel matching. Highly concentrated solution (used to increase the image contrast) may not give the perfect straight line at higher path lengths in the calibration, which can introduce the errors. Therefore, either one should use low concentration barium chloride solution or should use some other solution which has high atomic number element present in it such as iodine. Using high atomic number element solution can increase the contrast at small concentration. However, one should make a proper choice of the solution considering the suitability of it such as it should be harmless to equipment, human and environment, economically viable and easy to use.

7. Conclusions

An X-ray technique to quantify the local concentration

and liquid holdups in a packed bed has been developed which takes care of the polychromatic nature of X-rays and thus the mass absorption coefficient. Therefore, the technique is quite versatile and has been used to quantify the local concentration in presence of another miscible liquid. Results obtained from X-ray study have been compared with the total concentration value obtained by simple mass balance in the packed bed. Reasonable agreement was found between the two values. Similarly, the results obtained for total liquid holdup in the packed bed using X-ray method are within the range of the values obtained by other conventional method (drainage) and published correlations. This technique can be used in other visualization techniques, such as neutron radiography and gamma ray tomography.

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