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# Detailed spatio-temporal solids concentration profiling during batch settling of activated sludge using a radiotracer

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**Abstract**

In building and tuning good settling models for secondary clarifiers of wastewater treatment plants, there is a need for measured continuous solids concentration profiles during batch settling. Conventional measuring techniques have difficulties in recording this kind of data, either because they are invasive, or because of the low solids concentration and/or solids density of activated sludge.

This paper investigates a novel non-invasive measurement technique borrowed from nuclear medicine, using a solids radiotracer and gamma cameras, to obtain solids concentration profiles during the batch settling of activated sludge, in a pilot-scale column with a height of 1 m.

The technique does not disturb the settling process, does not alter the settling characteristics, gives profiles every minute and every few millimeters, and is capable of measuring in a range of 0–25 g/l with high accuracy.

Dynamic solids concentration profile measurements were performed for sludges of different wastewater treatment plants, and at different initial concentrations. The results show a quantitative representation of the settling process, and reveal hindered and compression settling.


*Keywords:* Activated sludge; Batch settling; Pilot-scale; Concentration profiles; Radiotracer; Gamma camera

## 1. Introduction

Batch settling experiments are commonly used for hindered settling experiments and process monitoring intended to measure how well sludge can be thickened in a continuous thickening process, as well as to validate thickening models. In activated sludge batch settling usually only the batch settling curve is recorded. As far as known to the authors, the only exceptions are Kinnear (2002), who measured equilibrium solids concentration profiles, and Chu et al. (2003), who measured dynamic solids concentration profiles during the settling of waste activated sludge. In soil mechanics on the other hand, such profiles (Bergström, 1992; Scott, 1968; Dreher, 1997; Shih et al., 1986; Been and Sills, 1981; Wells, 1990; Tiller et al., 1991) are more frequently

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measured. These dynamic profiles provide a lot more information about the settling process than just the recording of the suspension–liquid interface.

It is intended in this work to measure such dynamic solids concentration profiles for activated sludge batch settling with an appropriate technique. Such a technique should fulfill the following requirements:

- settling process may not be disturbed,
- settling characteristics of the sludge may not be altered,
- solids concentrations within a range of 0–25 g/l need to be measured,
- on-line analysis at high spatial and temporal resolution.

Non-destructive techniques, such as $\gamma$-ray (Bergström, 1992; Scott, 1968; Dreher, 1997) and X-ray (Shih et al., 1986; Been and Sills, 1981; Wells, 1990; Tiller et al., 1991) have been applied for the measurement of the solids concentration profiles during batch settling of suspensions other than activated sludge. Those suspensions all have a higher solids concentration and solids density than activated sludge. Some reports mentioned the accuracy of the measurement, i.e. 0.25 (Been and Sills, 1981) and 0.5 vol% (Bergström, 1992), which is too low for activated sludge with a solids concentration of 0.1–0.4 vol% (i.e. for a solids concentration between 3 and 6 g/l and a solids density around 1700–1900 kg/m$^3$, as found in the present case). This makes these techniques unsuitable for activated sludge.

Chu et al. (2003) used CATScan measurements for the monitoring of settling activated sludge but the measurement uncertainty obtained was only 0.1 vol%. The accuracy is again too low for activated sludge.

In wastewater treatment processes, optical devices, ultrasound and dielectric spectrometry are commonly used for the measurement of the solids concentration (Vanrolleghem and Lee, 2003). However, such sensors cannot be used for monitoring the solids concentration profile during batch settling since they are commonly positioned inside the clarifier (Olsson and Nielsen, 1997), which disturbs the settling process.

Since the reported techniques cannot be used, the idea of measuring a characteristic of a substance which adsorbs to the solids arose. Solids tracers have already been used to determine the sludge residence time distribution:

- Lumley and Balmer (1990) used $MnCl_2$ in a secondary clarifier,
- Audic et al. (1993) used radioactive Au-198 in a secondary clarifier,
- Grijspeerdt and Verstraete (1995) used pyrene in a lab-scale secondary clarifier,
- IAEA (2001) used radioactive La-140 in an aeration tank.

The radioactive tracers (Au-198 and La-140) have the advantage over Mn and pyrene that they can be measured on-line. Besides radioactive Au-198 and La-140, other radiotracers such as Br-82, I-131 and Tc-99m have been used as a liquid tracer to investigate wastewater treatment (Borroto et al., 2003; Farooq et al., 2003; Chmielewski et al., 1998; IAEA, 2001). Among those five radiotracers, Tc-99m is the only one that can be produced in a generator. Such a generator consists of Mo-99 (67 h half-life) that decays by $\beta^-$-emission to Tc-99m (6 h half-life). The Tc-99m can be extracted by flushing physiological saline through the generator. This radionuclide is the most commonly used radioisotope in nuclear medicine. This is because of its good half-life (long enough to perform a study and short enough to limit the radioactive dose received by patients), the monochromatic $\gamma$-ray energy it emits, and its existence in oxidation states from +1 to +7 in radiopharmaceuticals. Those radiopharmaceuticals can be anionic, neutral or cationic. A cationic Tc-99m complex could be used to trace the solids of activated sludge since the solids are negatively charged. One such positively charged complex is Tc-99m Sestamibi (Methoxy IsoButyl Isonitril), which could be produced and detected at the Department of Nuclear Medicine of the Ghent University Hospital, since a Mo-99/Tc-99m generator and different gamma cameras are available.

Since Tc-99m Sestamibi behaves similarly to potassium in viable myocardial tissue (Geatti, 1999), it is not so unlikely that activated sludge has a strong affinity towards it just like it has a strong affinity for potassium (Wang et al., 2000). The disadvantage of behaving in the same way as potassium in activated sludge, could be that the settling properties of the activated sludge are changed, as is observed for potassium (Muller et al., 2002; Novak et al., 1998; Murthy and Novak, 1998).

It is therefore investigated whether Tc-99m Sestamibi adsorbs onto the solids of activated sludge, without altering the settling properties and if so, whether the on-line analysis of the Tc-99m Sestamibi can be converted into solids concentration profiles. The influence of radiotracer on the settling properties is determined by comparing the batch settling curve with and without radiotracer, in a way similar to Grijspeerdt and Verstraete (1995). These investigations are discussed in the preliminary experiments. Afterwards, pilot-scale batch settling experiments are discussed.

## 2. Material and methods

### 2.1. Experimental set-up

Since the largest gamma camera that we could use has a field of view of 508 mm by 381 mm, only 0.5 m height of one settling column can be scanned per experiment. To scan over a height of 1 m with the same sludge at the same time, two parallel experiments are done with two gamma cameras and two settling columns: one gamma camera for detection of the lower 0.5 m of one column (1500 XP, single-headed gamma camera) and the other one for the upper 0.5 m of the other column (AXIS, two-headed gamma camera). The lowest 20 mm of the column could not be scanned because of the presence of a metallic aerator which interfered with the gamma radiation. An overlap of 50 mm between the two columns was provided to evaluate the coherence of the two parallel settling tests. A third settling column in the lab was used to verify the influence of the tracer on the settling properties. Thus, the set-up consists of three settling columns and two gamma cameras. The experimental set-up at the Ghent University hospital is shown in Fig. 1.

The batch settling columns are made of plexi and have an inner diameter of 386 mm, which is large enough to avoid wall effects (Matsui et al., 1978; Behn, 1957; Chen et al., 1996). Two columns have a height of 1.6 m, the third one has a height of 1.2 m.

The gamma camera detects the 140 keV photons emitted from Tc-99m during a chosen time interval and produces a two-dimensional projection of the radioactivity of the emitting object, i.e. an image. Images are produced every 30 or 60 s, which results in radiotracer profiles over time. The images have a resolution of $256 \times 256$ pixels (65536). The size of each pixel is $2.33 \times 2.33$ mm. An example of such an image is shown in Fig. 2. With a Co-57 flood source,it is determined which pixels correspond to the scanned object. Such sources are normally used to determine the response uniformity of the gamma camera. For the AXIS-camera, the highest vertical rectangular area was 144 pixels wide and 222 pixels high. For the 1500 XP-camera we obtained a similar region of 123 pixels wide and 215 pixels high. As the solids concentration profile is considered homogeneous in a plane parallel to the bottom of the column, the two-dimensional data of each image are transformed to one-dimensional data by summing the values per pixel in every plane. This results in a vector with 222 elements (counts) for the AXIS-camera, resp. 215 elements for the 1500 XP-camera. The data are subsequently corrected for radioactive decay. The half-life of Tc-99m is approximately 6 h.

Fig. 1. Experimental set-up at the Ghent University Hospital (left: lower section, 1500XP camera; RIGHT: upper section, AXIS camera).

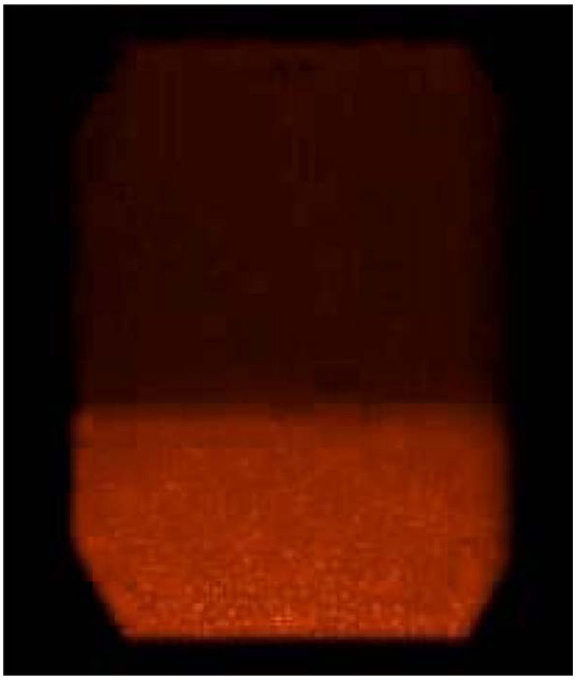

Fig. 2. Example of an image obtained after 6 h settling of sludge with Tc-99 m Sestamibi as radiotracer. The concentration of the tracer is higher when the color is brighter; the brightest color corresponds to the sludge blanket, the darkest to the supernatant.

### 2.2. *Origin of the sludge*

Sludge was taken from two different municipal wastewater treatment plants close to Ghent, namely from Destelbergen and from Deinze and is referred to as Destelbergen sludge and Deinze sludge. The two plants have the same treatment steps, i.e. screening, selector tank, aerobic treatment (with nitrification/denitrification and chemical phosphorous removal) and secondary clarification (and sludge treatment). Sludge was taken from the overflow of the aeration tank and from the effluent and recycle of the secondary clarifier.

### 2.3. *Experimental procedure*

Sludge (a total volume of 360 l) was collected at the wastewater treatment plant 2 days before the actual measurements. All three columns were filled with the sludge and the columns were aerated for 2 days to avoid temperature differences during the batch settling experiment. Just before the experiment, the columns were nivellated to make sure that all three columns have the same suspension height. Samples were taken for analysis of the solids concentration (method 2540 D of Standard Methods, Eaton et al., 1995) and solids density. The solids density was determined according to a soil science method which is under approval for certification (ISO/DTS 17892-3-2003) and is called "Geotechnical Investigation and Testing—Laboratory Testing of Soil—Part 3: Determination of Particle Density–Pycnometer Method". This method is based on liquid displacement. The temperature of the suspension was measured at the beginning and end of each experiment. The two columns at the University Hospital were positioned as closely as possible to the gamma cameras (about 10–20 mm). About 20–40 mCi Tc-99m Sestamibi was injected and the columns were aerated for about 5 min to get a good distribution of the radiotracer over the whole suspension. The experiments were then started and lasted between 5 and 6 h. During the first 2 h, images were taken every 30 s, for the last 3–4 h, images were taken every 60 s. The sludge blanket height was also determined visually in the column in the lab without radiotracer.

## 3. Preliminary experiments: results and discussion

Preliminary experiments were needed to gain more insight in the proposed measurement technique and to answer the following questions

- Does Tc-99m Sestamibi adsorb on the sludge?
- Does Tc-99m Sestamibi change the settling properties?

Preliminary radiotracer experiments were performed at lab-scale, i.e. in 2 l bottles, and with varying radioactivity versus solids concentration ratios. Sludge was collected from the overflow of the aeration tank of the Destelbergen treatment plant. One of the samples was scanned for 1 h, the others at the beginning and after approximately 1–2 h of settling, as summarized in Table 1.

Since the radiating recipient is smaller than the field of view of the gamma camera, the pixels of the image corresponding to the recipient were determined and extracted. Subsequently, the data were converted to one-dimensional vectors by summing all pixels corresponding to the same horizontal plane. The results of the 120 scans of the first experiment (Fig. 3) show that Tc-99m Sestamibi partly adsorbs onto the sludge, the radioactivity in the supernatant stays constant, therefore the adsorption does not change during the experiment, and the radioactivity decreases with height in the sludge blanket (i.e. a radioactivity profile is observed inside the sludge blanket), just like the solids concentration decreases with height.

The partial adsorption and radioactivity profile are also observed in the other experiments of Table 1

Table 1
Experimental conditions in the preliminary experiments: solids concentration of sludge, radioactivity of Tc-99 m Sestamibi and gammascan measurements

| $C_0$ (g/l) | Radioactivity (mCi) | Gammascan |
|---|---|---|
| 5.1 | 2.06 | 120 scans of 30 s (i.e. 1 h) and one scan of 5 min after 1.24 h of settling |
| 5.1 | 0.458 | 10 scans of 30 s during first 5 min of settling and one scan of 5 min after 1.53 h of settling |
| 6.6 | 0.487 | 10 scans of 30 s during first 5 min of settling and one scan of 5 min after 1.8 h of settling |
| 10.1 | 1.83 | 10 scans of 30 s during first 5 min of settling and one scan of 5 min after 2.07 h of settling |
| 10.2 | 0.488 | 10 scans of 30 s during first 5 min of settling and one scan of 5 min after 2.36 h of settling |

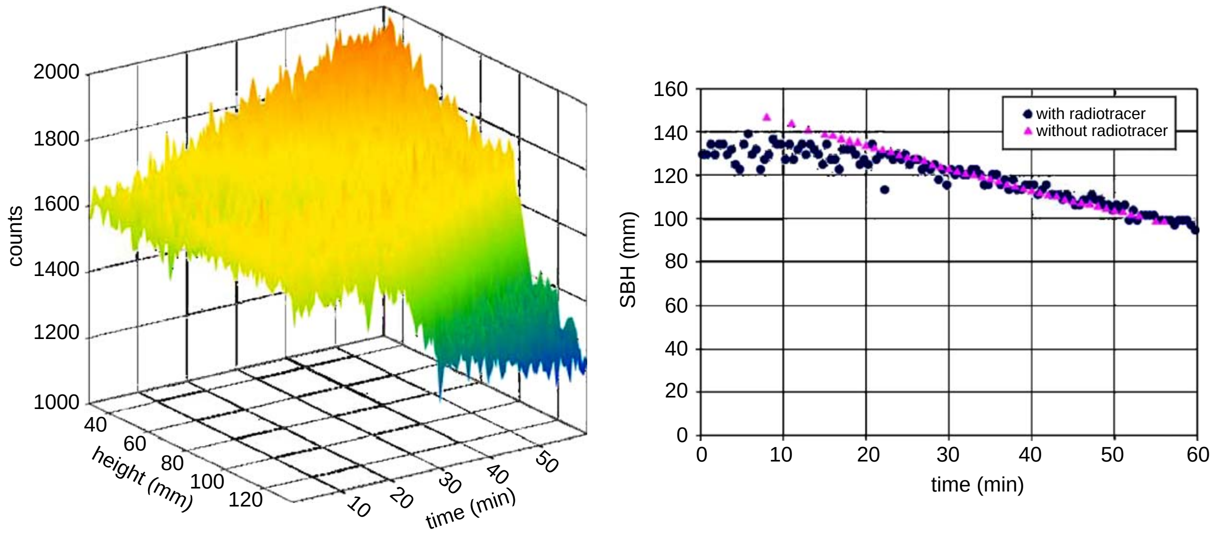


Fig. 3. Time-height profile of the counts of radiating Tc-99 m Sestamibi (2.06 mCi) (left) and its sludge blanket height and measured sludge blanket height (SBH) in sludge without radiotracer (right) during the batch settling of Destelbergen sludge ($C = 5.1$ g/l) (height = 0 mm corresponds to the bottom of the bottle).

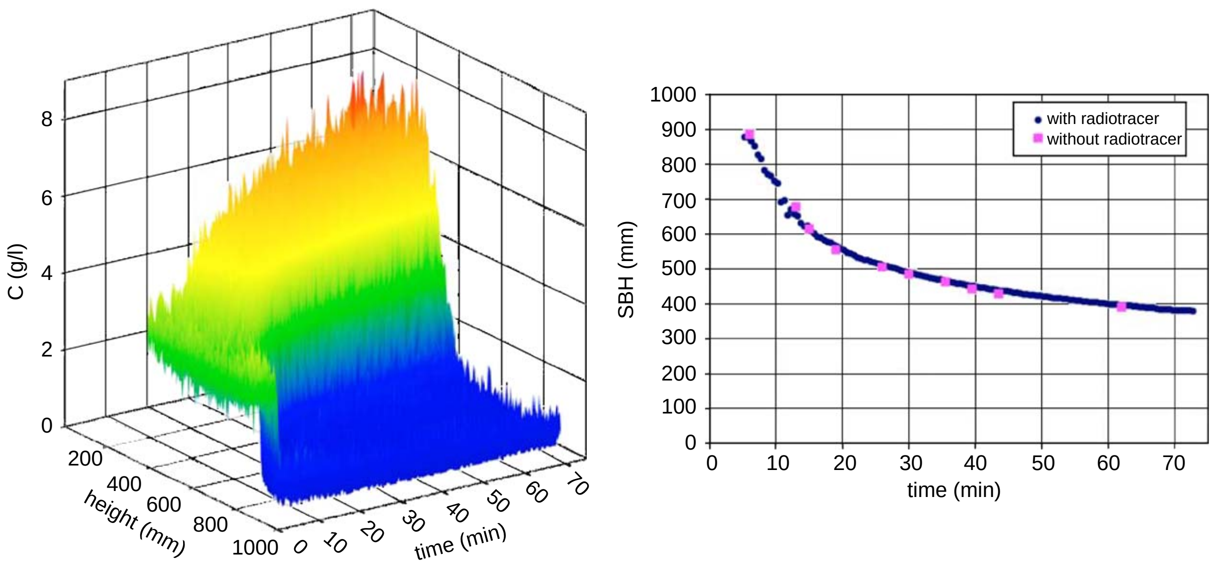


Fig. 4. Calculated solids concentration profile (left) and its sludge blanket height and measured sludge blanket height in sludge without radiotracer (right) during the batch settling of Destelbergen sludge ($C = 3.3$ g/l) (height = 0 mm corresponds to the bottom of the column).

(results not shown). The total radioactivity remained constant with time for all five experiments.

Fig. 3 also shows the evolution of the sludge blanket height (SBH) in a sample with and without Tc-99m Sestamibi. The sludge blanket height in the sample with Tc-99m Sestamibi is determined as the depth where the counts are higher than the counts of the completely mixed sample. The deviation during the first 20 min is due to the fact that the scanned images can only be collected from a certain depth (approximately 30 mm) in the recipient. It can be concluded that the settling properties do not change with the addition of the radiotracer for the Destelbergen sludge. However, it is felt that this should be verified for each experiment.

Knowing that the radioactivity remains constant in the supernatant and that the radioactivity increases with increasing solids concentration, the solids concentration at a certain height $z$ and time $t$, $C(z, t)$, can be calculated with

$$C(z,t) = \frac{\mathrm{Cnts}(z,t) - \mathrm{Cnts}_{\mathrm{l}}}{\mathrm{Cnts}_0 - \mathrm{Cnts}_{\mathrm{l}}}\, C_0$$

in which $\mathrm{Cnts}_0$ are the counts of the completely mixed suspension, $C_0$ is the initial solids concentration, $\mathrm{Cnts}_{\mathrm{l}}$ are the counts of the supernatant and $\mathrm{Cnts}(z,t)$ are the counts at a certain height and time. To illustrate the result of these calculations, a batch settling experiment with Destelbergen sludge ($C = 3.3$ g/l) was performed in the pilot-scale set-up, with radiotracer and with the 1500 XP-camera. The settling properties were verified in the laboratory without radiotracer. Scans were taken over a height of 378–878 mm. Solids concentration profiles as calculated with the above formula are shown in Fig. 4 together with the sludge blanket heights obtained from these profiles and from the laboratory measurements. The high-resolution profile gives a nice representation of the settling process and can be used for a better understanding and modelling of the process. The coefficient of variation for the obtained solids concentration is calculated from the standard deviation $\sigma$ of the measured counts and measured solids concentration

$$\frac{\sigma^2_{C(z,t)}}{C(z,t)^2} = \frac{\sigma^2_{\mathrm{Cnts}(z,t)}}{(\mathrm{Cnts}(z,t) - \mathrm{Cnts}_{\mathrm{l}})^2} + \frac{\sigma^2_{\mathrm{Cnts}_0} + \sigma^2_{\mathrm{Cnts}_{\mathrm{l}}}}{(\mathrm{Cnts}_0 - \mathrm{Cnts}_{\mathrm{l}})^2} + \frac{\sigma^2_{C_0}}{C_0^2}$$

and results in a coefficient of variation of 20% for the initial concentration and 16% for the maximum observed concentration. In comparison, the reported accuracy of 0.1 vol% of the CATScan measurements (Chu et al., 2003) results in a coefficient of variation of 50% for the initial concentration and 7% for the maximum observed concentration. As indicated in Fig. 4 (right), the settling properties once again did not change with the addition of the radiotracer.

In conclusion, the proposed technique, i.e. the use of the radiotracer and measuring the radiation with a gamma camera, can be used to determine a high-resolution solids concentration profile during batch settling with good accuracy.

## 4. Pilot-scale batch settling experiments: results and discussion

Six batch settling experiments were performed: three on Destelbergen sludge and three on Deinze sludge. Table 2 shows the initial concentrations, the according adsorption and calculated coefficient of variation for the initial and maximum observed solids concentrations and solids densities for these experiments. The Deinze sludge clearly had a higher affinity towards Tc-99m Sestamibi than the Destelbergen sludge which resulted in the lower coefficient of variation for Deinze sludge. The coefficient of variation decreases with increasing initial solids concentration because more radiotracer is adsorbed when the initial solids concentration is higher. The total mass of solids in the column, calculated from the solids concentration profile and assuming the same concentration in the lower 20 mm as the concentration measured

Table 2
Origin of sludge, initial solids concentrations, coefficients of variation for the initial and maximum observed solids concentrations and solids density of the six batch settling experiments

| $C_0$ (g/l) | Fraction of radiotracer adsorbed on $C_0$ (%) | Coefficient of variation for $C_0$–max $C$(%) | Solids density (kg/m$^3$) |
|---|---|---|---|
| $2.40 \pm 0.05^\circ$ | 21 | 27–21 | $1762 \pm 19$ |
| $3.23 \pm 0.05^\circ$ | 26 | 20–16 | $1753 \pm 36$ |
| $4.30 \pm 0.02^\circ$ | 34 | 14–11 | $1714 \pm 6$ |
| $3.67 \pm 0.06$* | 74 | 11–9 | $1943 \pm 42$ |
| $6.12 \pm 0.09$* | 83 | 10–8 | $1898 \pm 57$ |
| $7.29 \pm 0.04$* | 85 | 6–4 | $1881 \pm 27$ |

$^\circ$: Destelbergen; *: Deinze.

at the lowest point, does not deviate more than 2% from the total mass calculated from the initial solids concentration and volume of the suspension. This shows that mass balancing can be performed with these radiotracer data.

In all experiments, the settling properties did not change with the addition of the radiotracer, the temperature did not change, the coherence of the two parallel settling tests was excellent and the hindered settling rates decreased with increasing initial solids concentrations, as expected.

The solids concentration profiles during batch settling are shown in Fig. 5 for the Destelbergen sludge and in Fig. 6 for the Deinze sludge. All profiles show the same trend. Initially the solids concentration is uniform. Subsequent profiles show the accumulation of sludge at the base as a result of settling, as well as the decrease of the sludge blanket height. The concentrations at the base are continuously increasing and higher concentrations move towards the sludge blanket height. When concentrations in excess of the original one reach the sludge blanket height, the sludge blanket height decreases more slowly. The solids concentration at the base increases faster in the beginning and more slowly towards the end for the Deinze sludge in comparison with the Destelbergen sludge. This indicates that the

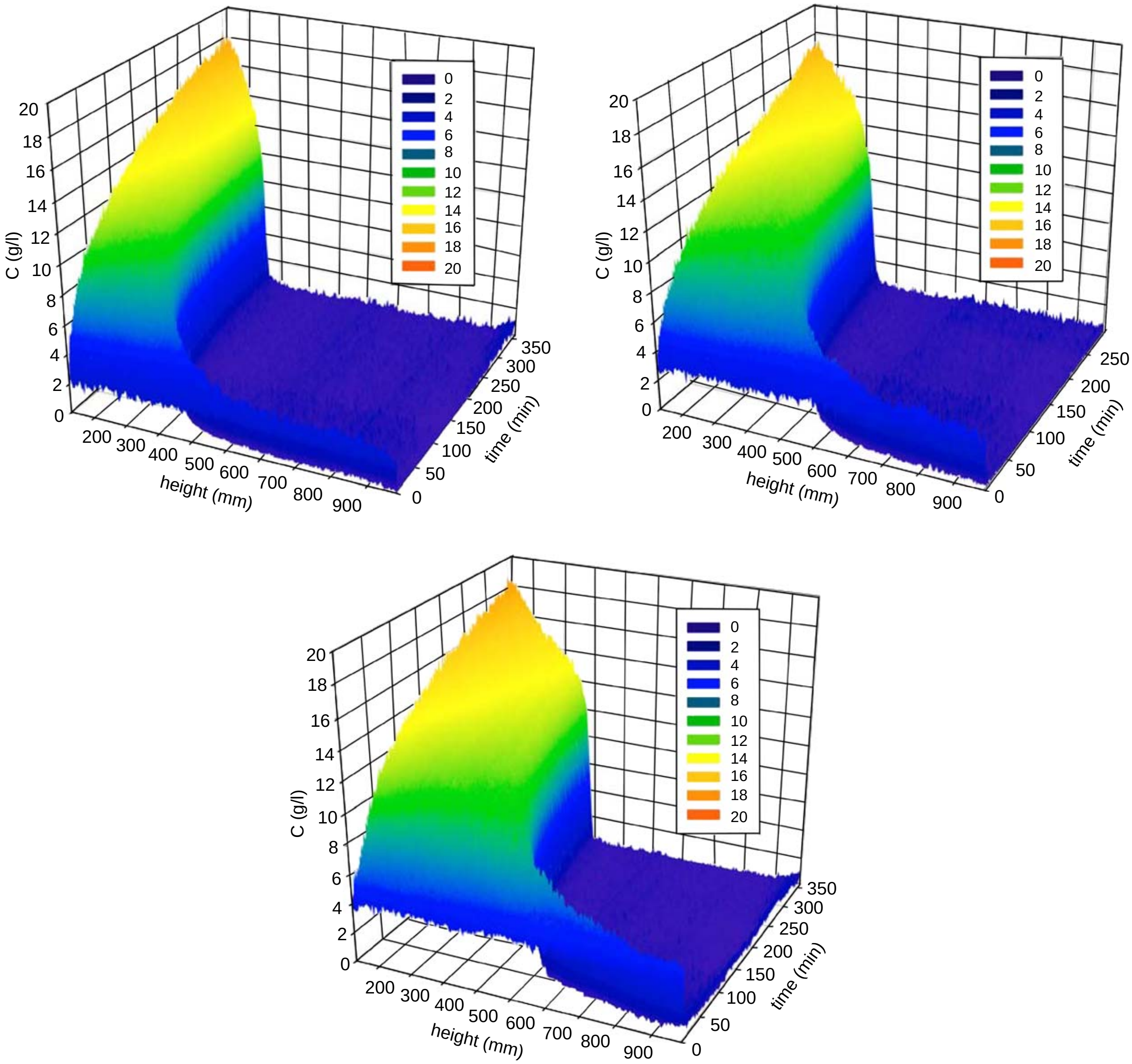


Fig. 5. Calculated solids concentration profile during batch settling of Destelbergen sludge (top left: $C = 2.40$ g/l; top right: $C = 3.23$ g/l; bottom: $C = 4.30$ g/l) (height = 0 mm corresponds to the bottom of the column).

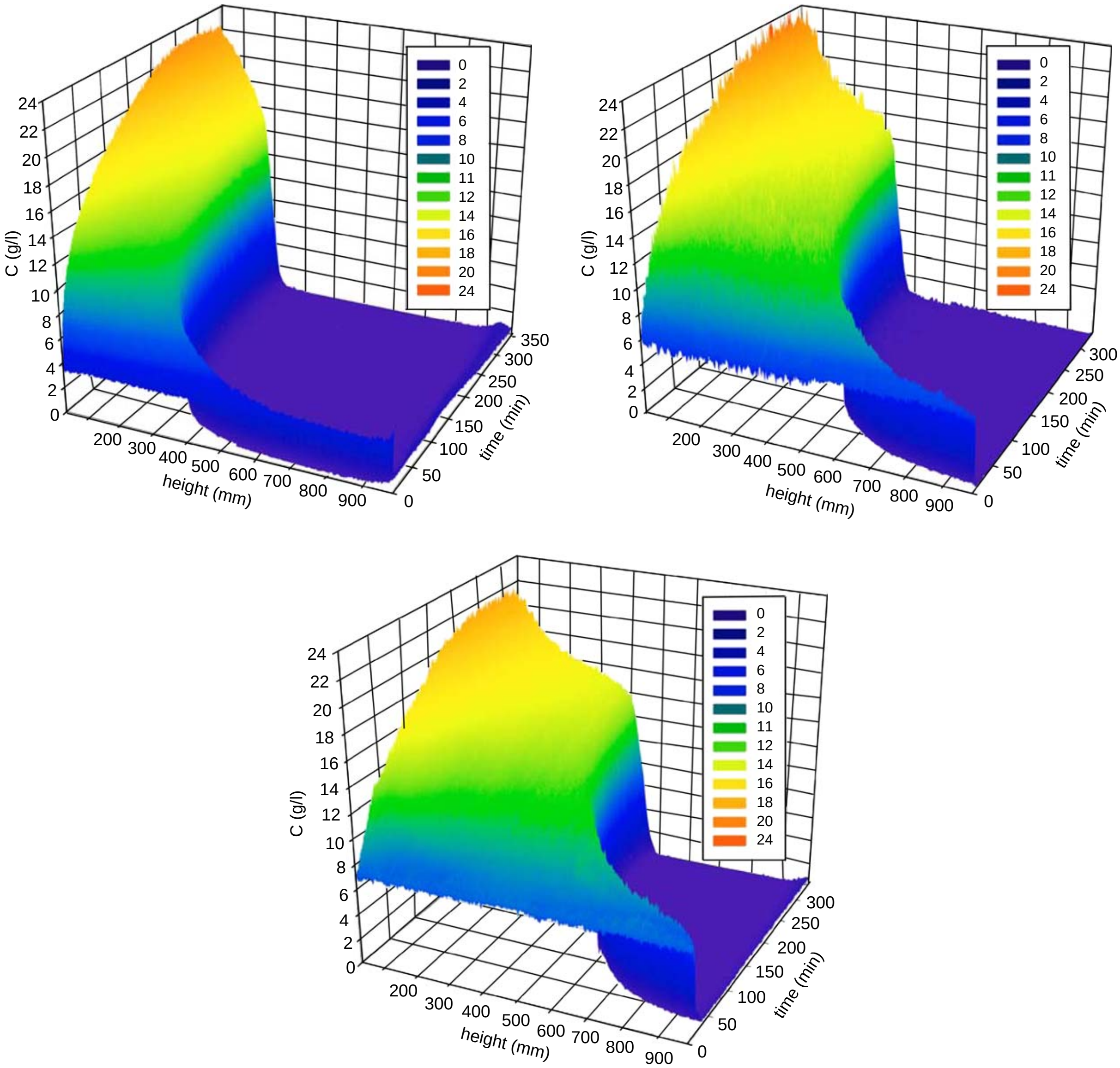


Fig. 6. Calculated solids concentration profile during batch settling of Deinze sludge (top left: $C = 3.67\,g/l$; top right: $C = 6.12\,g/l$; bottom: $C = 7.29\,g/l$) (height = 0 mm corresponds to the bottom of the column).

Deinze sludge shows more resistance to further thickening. This will be verified in upcoming studies by modelling the profiles. Equilibrium prevails at the end of the experiments on the Deinze sludge (the concentration profiles did not change anymore for a period of more than 20 min).

The equilibrium concentration profiles for the three different initial concentrations for the Deinze sludge are presented in Fig. 7. The equilibrated solids concentration at the base are around 22 g/l regardless of the initial solids concentration. The fact that the observed equilibrium profiles show an increasing concentration towards the base, indicates that besides hindered settling, compression settling occurs too. A suspension undergoing only hindered settling has an equilibrium profile existing of a sludge blanket with a constant maximum concentration (for which the hindered settling velocity equals zero). The different equilibrium profiles show approximately the same jump in concentration of about 15 g/l over a height of about 30 mm. It can be stated that for concentrations higher than 15 g/l compression is present. The compression solids concentration (boundary between hindered and hindered + compression settling) could be estimated by

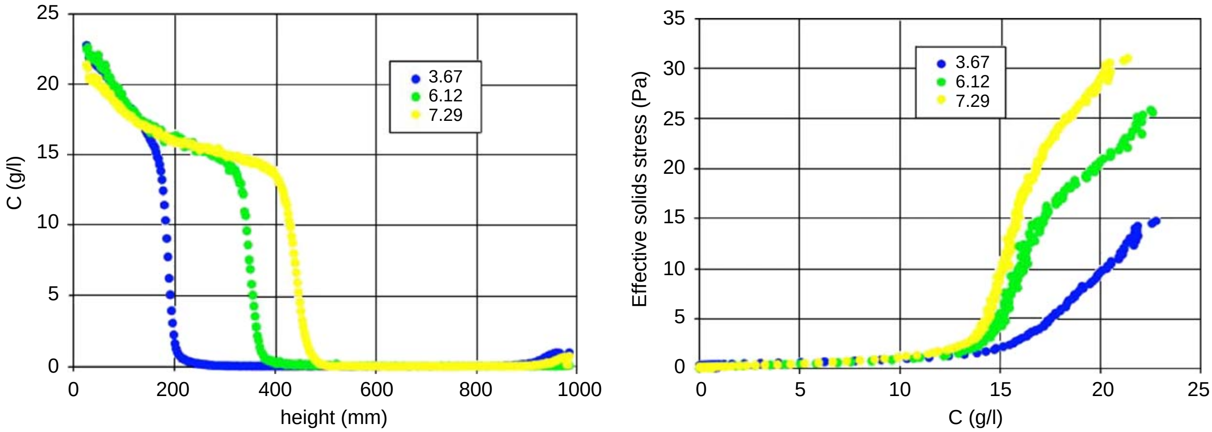


Fig. 7. Equilibrium solids concentration profiles (left) (height = 0 mm corresponds to the bottom of the bottom of the column) and effective solids stress versus solids concentration (right) for different initial concentrations (shown in g/l in legend) for the Deinze sludge.

averaging concentrations between 1 and 15 g/l of the equilibrium profiles; this gives values of 7.71, 8.58 and 9.09 for increasing initial concentration. Again, this statement needs to be verified by modelling the concentration profiles.

Since at equilibrium the solids pressure at a certain height equals the effective solids stress at that height, the effective solids stress $\sigma_e$ can be calculated as follows:

$$\sigma_e(C(z, t_{\text{equilibrium}})) = \frac{\Delta\rho g}{\rho_S} \int_z^H C(z, t_{\text{equilibrium}})\, \partial z$$

in which $\rho_S$ is the solids density, $\Delta\rho$ is the difference between solids and fluid density and $H$ is the total suspension height. The calculated effective solids stress is shown in Fig. 7 (right) for the different experiments. The effective solids stress increases with the concentration and is different for the three experiments. The stress at the highest concentration (i.e. at the base of the column) agrees with the pressure of the buoyant weight of solids calculated from the initial solids concentration. This confirms again the accuracy of the proposed measurement technique. At concentrations lower than 15 g/l, the solids do not offer considerable resistance to further settling (effective solids stress lower than 5 Pa), but for concentrations higher than 15 g/l, the solids yield more slowly with the applied pressure. The results are comparable with those reported by Chu et al. (2002) for kaolin and clay. Fig. 7 (right) can be used to find an empirical relationship correlating the effective solids stress and solids concentration. The measurements clearly show compression during the batch settling of the Deinze sludge since there is a solids concentration profile at equilibrium. If only hindered settling would occur, the sludge bed at equilibrium would consist of one uniform solids concentration, namely the concentration at which the settling velocity equals zero.

The existence of compression during the batch settling of the Destelbergen sludge is evidenced by the form of the iso-concentration lines observed. When only hindered settling occurs, the iso-concentration lines are straight lines; when compression occurs also, the iso-concentration lines become curved (Bürger et al., 2004). This is clearly the case for all experiments. Fig. 8 shows the Destelbergen results after scatter-diagram smoothing.

Although equilibrium is not reached yet for the Destelbergen experiments, the profiles at the end, show the same trend as the Deinze equilibrium profiles. There is a jump in concentration of about 12 g/l over a height of 40 mm. Calculating the average concentrations between 1 and 12 g/l of the Destelbergen profiles at the end gives estimated compression solids concentrations of 6.88, 7.34 and 8.85 g/l for increasing initial concentration.

Modelling of these data needs to illuminate this further. It is clear that a lot more information can be gained from the measurement of the solids concentration profiles than the batch settling curve during the batch settling of activated sludge.

## 5. Conclusion

This study utilized a solids radiotracer and gamma cameras to obtain high time and spatial resolution solids concentration profiles during the batch settling of activated sludge in a pilot-scale column with a height of 1 m. It is the first time that such detailed pilot-scale dynamic solids concentration profiles have been

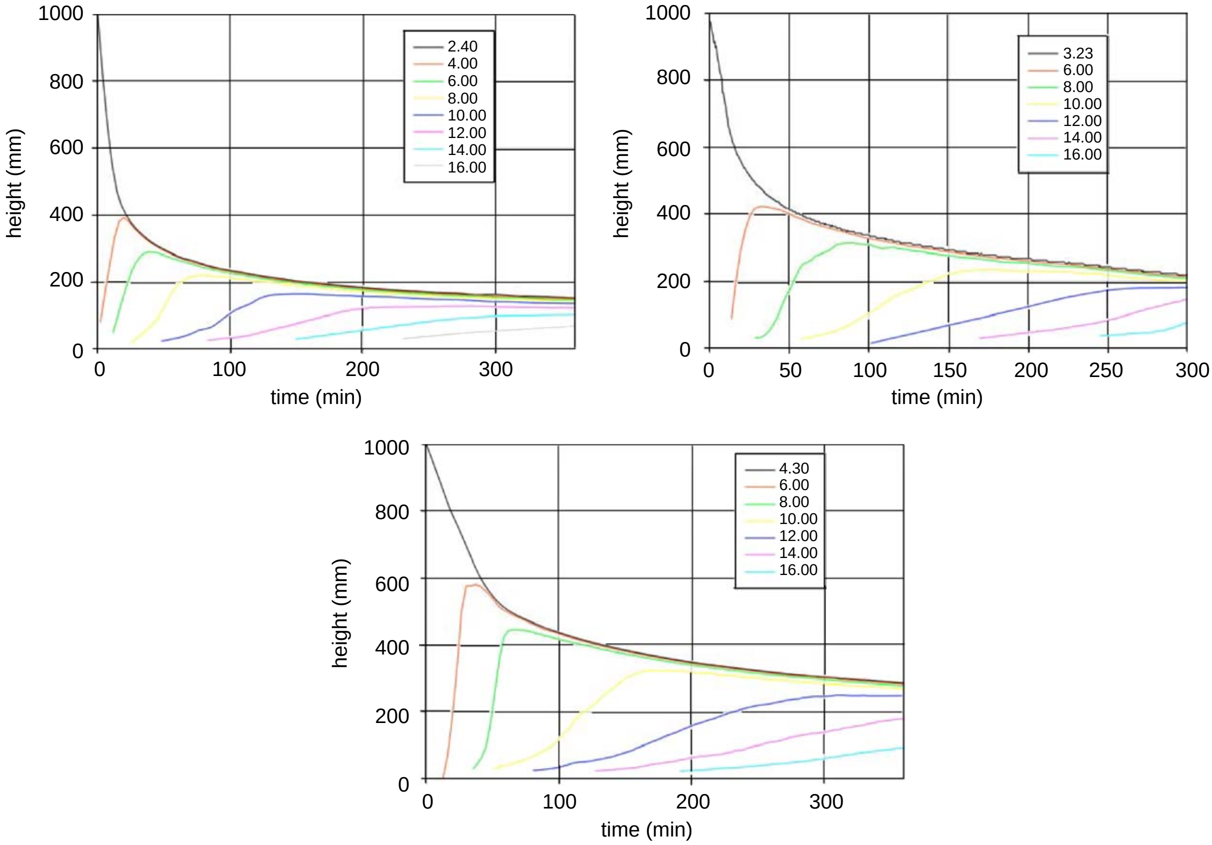


Fig. 8. Iso-concentration lines (different concentrations shown in g/l in legend) during batch settling of Destelbergen sludge (top left: $C = 2.40$ g/l; top right: $C = 3.23$ g/l; bottom: $C = 4.30$ g/l) (height = 0 mm corresponds to the bottom of the column).

reported. This non-invasive technique does not disturb the settling process, does not alter the settling characteristics, gives profiles every minute, and is capable of measuring in a range of 0–25 g/l with high accuracy.

The pilot-scale dynamic solids concentration profiles give a detailed quantitative representation of the settling process. They confirm that hindered settling rates decrease with increasing initial solids concentrations. The profiles show not only hindered settling but the equilibrium concentration profiles and the iso-concentration lines clearly show that compression too is taking place. Compression solids concentrations can be estimated from the equilibrium profiles and are between 7 and 10 g/l depending on the origin of the sludge and the initial solids concentration. The equilibrated solids concentration at the base is the same for the same sludge regardless of the initial solids concentration.

The obtained profiles are very appropriate data to increase our understanding of the settling behaviour of activated sludge and to validate models.

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