How to measure the volume fraction of granular assemblies using x-ray radiography

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Abstract

When investigating dynamical processes in granular systems, it is frequently necessary to measure the time-resolved local material density. Recently, x-ray radiography facilities became available in many laboratories and can be used to measure the volume fraction via the attenuation of x-ray radiation along the beam direction. Naïve application of the Beer-Lambert law yields, however, unacceptably large systematic errors due to beam hardening. We present a calibration protocol which allows to reliably measure the local volume fraction based exclusively on reference measurement of known packing fraction.

Keywords: volume fraction, x-ray radiography, beam hardening

1. Introduction

In many cases, the dynamics of granular matter depend sensitively on the local volume fraction of the system. A prominent example is the formation of shear bands which requires local reduction of density due to Reynolds dilatancy. Reliable measurements of volume density are necessary for the quantitative investigation of many granular systems such as irregular granular pipe and hopper flow, fluidized beds, multi-phase flows, etc. which are essential for many industrial applications. In some cases, such as dense shear flows, the dynamical properties vary drastically due to short term variations of the local volume density, resulting in a demand for reliable and precise measurements. The attenuation of x-ray radiation allows to measure the material density. Nowadays x-ray setups are available as standard devices in many laboratories. Due to the progress of modern flat panel detectors large frame rates with high signal-to-noise ratio can be reached. This allows the study of dynamic granular systems using x-ray radiography. The naïve application of Beer-Lambert’s law will, however, lead to inaccurate measurements due to beam hardening.

In this short communication, we propose a protocol for calibration of x-ray radiography measurements which relies exclusively on reference measurement of a sample of known volume fraction. We exemplify the method for the case of shear bands in a horizontally vibrated granular system and obtain a correction for the packing density. Naïve measurements, where no correction with respect to beam hardening is applied, lead to large systematic errors.

2. Measurement of volume fraction and errors due to beam hardening

When monochromatic x-rays penetrate matter, the incident intensity, \(I_0\), emitted from the source is attenuated exponentially,

\[ I = I_0 e^{-\mu \Delta x}, \]

known as Beer-Lambert law (Beer, 1852), where \(\Delta x\) is the thickness of the material, and \(\mu\) its specific attenuation coefficient. In the grain material, the intensity of the beam decreases due to Rayleigh scattering, photoelectric absorption, and Compton scattering by the electrons of the material. For photon energies above \(2m_e c^2 = 1.022\) MeV, electron-positron pair production also occurs. All these contributions to the attenuation coefficient depend on the photon energy \(E\). Furthermore, the attenuation depends on the number of electrons, which is given by the atomic number \(Z\) of the grain material. This allows to distinguish the internal structure of objects, even if from outside no structure is noticeable. The attenuation can be also used to measure the density of a granular system, which is connected to the path length of the beam in the grains, see Fig. 1: Beam (a) arrives at the detector with an intensity \(I_0\) due to the fact that the attenuation by the ambient air can be neglected with respect to the attenuation of solid matter. Beam (b) is attenuated by passing only the container walls (discussed below) and beam (c) by passing the container walls and the granular material. Hereby, only the
When it comes to quantitative measurements of granular densities beyond gray-scale imaging, however, we are faced with a problem: from the above mentioned energy dependent interaction probability of x-ray photons with the electrons of the grain material, we see immediately that the Beer Lambert law, Eq. (1), is valid only for monochromatic x-rays where all photons have the same energy, $E$. Common x-ray tubes generate, however, polychromatic x-rays, thus, the spectral composition of the x-rays alters as a function of the penetration length, $\Delta x$, through the material. More specifically, the low-energy (long wavelength) photons undergo scattering and absorption events at a higher probability than high-energy photons with the consequence that the spectrum of the x-ray beam shifts to high-energy, also called hard radiation. This effect is, therefore, referred to as beam hardening. In many applications, x-ray radiography is used to identify structural features from images. Here, beam hardening may be tolerable or even exploited to enhance contrast. For quantitative investigations, however, beam hardening is critical as it results in erroneous information about the sample’s composition and density.

A simple solution would be to calibrate the detector at different volume fractions $\phi(x, y)$ to deliver the correct value of $l_g(x, y)$ from a measured intensity, $I(x, y)$, irrespective of beam hardening and other sources of error. The problem here is that there is no simple way to produce a granular system at all values of densities which are of interest for the measurement, in order to calibrate our device.

3. Analyzing x-ray radiograms

In order to determine $\phi(x, y)$ from Eqs. (1) and (2), we need to take two effects into account: beam hardening and the extinction due to the container walls. Beam hardening implies that the attenuation coefficient of the granular material $\mu_g$ is a function of $l_g$. The actual functional form depends on the details of the x-ray generation (target material, acceleration voltage) and on the energy resolved sensitivity of the detector. While there is no closed theory for the derivation of $\mu_g(l_g)$, an empirical expression proposed by Yu et al. (1997) will be used here as an approximation to interpolate experimental data:

$$\mu_g(l_g) = \frac{\mu_0}{1 + \lambda l_g},$$

with open parameters $\mu_0$ and $\lambda$. Therefore, we obtain Lambert Beer’s law in the form

$$I = I_0 e^{-\mu_g l_g} e^{-\mu_w l_w} = I_0 e^{-\mu_g l_g} e^{-M},$$

where $\mu_w$ is the attenuation coefficient of the wall material and $l_w$ is the path length through the sidewalls. In our case, sketched in Fig. 1, the wall thickness is independent of $(x, y)$, therefore, we can introduce the constant $M$ to characterize the attenuation due to the container walls.
Its value can be determined from a single measurement of the empty container (beam (b) in Fig. 1):

\[ M = \mu_0 l_w = -\ln \frac{I^{(b)}}{I_0} \]

where \( I^{(b)} \) is the intensity of beam (b), and \( I_0 \) can be measured in an area of the radiogram which is outside of the object (beam (a) in Fig. 1),

\[ I_0 = I^{(a)}. \]

Knowing \( M \) and \( I_0 \), as well as Eq. (6) and (7), from Eqs. (4) and (5) we obtain \( l_g \). Keeping in mind that \( l_g \) is a function of the position \((x, y)\) at the detector, we obtain

\[ l_g(x, y) = \frac{1 + \lambda \mu_0 (x, y)}{\mu_0} \left( \ln \frac{I(x, y)}{I_0} - M \right). \]

With Eq. (2) we get,

\[ \varphi(x, y) = \frac{1}{l_{\text{box}}} \frac{1}{\mu_0} \left( \ln \frac{I(x, y)}{I_0} - M \right) + \lambda \ln \frac{I(x, y)}{I_0} + \lambda M, \]

which allows to determine the density field, \( \varphi(x, y) \), from the measured field of intensities, \( I(x, y) \). Equation (9) contains the yet unknown parameters \( \mu_0 \) and \( \lambda \) which can be determined by a fit to a calibration measurements. For the case of a rectangular container of different side length, it is convenient to measure the intensities for the same packing by just rotating the container by 90 degrees.

4. Example

We consider an horizontally vibrated container (amplitude 18 mm, frequency 18 Hz) partially filled by granular material such that the upper part of the granulate sloshes back and forth, see Fig. 2. A full size paper on this experiment including all details of the experiment will be published elsewhere (Kollmer et al., 2018). The system develops recurrent shear bands which give rise to a complicated spatio-temporal phenomenology (Pöschel and Rosenkranz, 1998; Pöschel et al., 2012). X-ray radiography with the calibration protocol described above allows to quantitatively analyze the gray-scale image shown in Fig. 2(right), we obtain the volume fraction profile shown in Fig. 2(left).

![X-ray radiogram of a horizontally vibrated container partially filled with granular material.](image)

Obviously \( \mu_0 \) is depending on \( l_g \) significantly. Neglecting the effect of beam hardening leads to wrong packing fractions.

Using Eq. (9) together with these parameters and averaging in horizontal direction over the area highlighted in Fig. 2(right), we obtain the volume fraction profile shown in Fig. 2(left).

![Attenuation coefficient \( \mu_0(l_g) \) obtained from the calibration measurements (symbols). The abscissa shows values of \( l_g = l_{\text{box}} \varphi \), where \( l_{\text{box}} = \{2, 3, 4\} \) cm and \( \varphi = 0.6 \). The dashed line shows Eq. (4) using the calibrated parameters.](image)

The obtained calibration, \( \mu_0(l_g) \), can be used to analyze the gray-scale image shown in Fig. 2 (right) to obtain the volume fraction as a function of height shown in Fig. 2 (left). The correction with respect to beam hardening outlined in this paper, delivers a packing fraction of \( \varphi = 0.50 \) in the shear band. Since \( \mu_0(l_g) \) is far from constant neglecting the effect of beam hardening leads to large deviations from that value (Fig. 3). The full description of the effect of self-organized migrating shear bands in horizontally shaken granular matter, including a detailed discussion on the packing density in shear bands, the error analysis and the physical significance of the correction with respect to beam hardening will be given in Ref. (Kollmer et al., 2018).
5. Conclusion

X-ray radiography can be used as an efficient and precise method to determine the spatio-temporal field of density in dynamical granular systems. For quantitative investigations, beam hardening that is the change of the spectral composition of the x-rays passing the object, must be taken into account. In this paper, we describe a protocol for the calibration of the x-ray recordings that relies on measurements of a packing at a single volume fraction which is essential for granular system because it is difficult to prepare a system at a well defined volume fraction. In this short communication, we interpolated our data with a simple function to correct beam hardening.

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References