Development of a non-invasive optical technique to study liquid evaporation in gas-solid fluidized beds

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Abstract

A non-invasive experimental technique based on particle image velocimetry and digital image analysis on images acquired with high-speed cameras operating in the visual and infrared wavelengths has been developed. With this, simultaneously whole-field data on the evolution of flow patterns and particle temperature distributions in a gas-fluidized bed with and without liquid injection can be obtained. A dedicated pseudo-2D gas-solid-fluidized bed was constructed and operated with liquid injection via a nozzle spraying onto the fluidized bed.

It was found that for proper processing of the data recorded with the high-speed infrared camera, combination with digital image analysis on images acquired from the visual camera is essential. The application of infrared thermography to gas fluidized beds suffers from the effects of interparticle reflections. The paper addresses the calibration procedure in detail and it is shown how to correct for this. The temperature-dependent effect of the setup window in the calibration is evaluated. To demonstrate the potential of the technique, it has been applied to dry fluidization and fluidization with liquid injection.

Development of a non-invasive optical technique to study liquid evaporation in gas-solid fluidized beds

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22 July 2016

1 Introduction

Gas-solid fluidized beds are known for the favorable property of operation at relatively uniform temperature, even when significant sources and sinks of heat are present. Liquid injection in gas fluidized beds has been employed industrially for decades, for example in evaporative cooling of polyolefin reactors [16] and in catalytic cracking [15]. For a long time, the mechanism of distribution and subsequent evaporation of liquid after injection into a gas fluidized bed remained difficult to ascertain [9] and this made it difficult to, for example, increase the amount of liquid injected (to evaporate) per bed volume. In part, this is due to difficulties in the interpretation of results from (single point) invasive probes (Table 1), for example for thermocouples [8, 19], which leaves questions in the field of heat transfer limitations open. Non-invasive techniques monitoring phenomena on the scale of the bed have amongst others allowed to distinguish between “free liquid” and liquid bound in liquid-solid agglomerates, and identified typical conditions for agglomerate breakage, if formed (Table 2).

Whole-field camera recordings of relevant phenomena have shown enormous value in enhancing our understanding. Modern techniques allow to observe phenomena that until recently were inaccessible, e.g. [10]. Automated quantitative analysis of images obtained allows for quantification, ensuring proper interpretation and for revealing system features that might otherwise remain unnoticed. Optical techniques can employ (high-speed) cameras that detect visual light, but also cameras that detect wavelengths in the infrared. Non-invasive determination of the temperature via infrared, in combination with visual techniques (e.g. applied by Yamada et al. [35] to detect stagnant solids), brings opportunity for the study of liquid injection into gas-fluidized beds and associated effects such as (non)agglomeration of solids and heat transfer limitations.

This work presents a methodology for simultaneous application of infrared thermography, high speed visual imaging and digital post-processing, to a gas fluidized bed of particles opaque in the infrared. This was tailored to the application of liquid injection, for which a suitable experimental setup is presented. Anticipating a wide distribution of temperatures inside the gas fluidized bed, the calibration method unlike the related work of Patil et al. [25] does account for the temperature-dependent effect of the setup window. This work has a particular attention to the width of the distribution of signals that is obtained in

Table 1: Concise overview of experimental techniques employing invasive probes to study liquid injection in dense gas-fluidized beds. This work employs a non-invasive method, instead.

Technique	Features
Temperature probes [1, 9, 12, 22]	<ul style="list-style-type: none"> • Fast and robust • Invasive • Sticking, difficult interpretation [9] and [12] • Mostly employed for time averages
Suction probes [9, 14]	<ul style="list-style-type: none"> • Mainly for time averaged gas composition profiles • Invasive • Potential for bias by sticking liquid
Pressure probes [2, 21, 20, 36]	<ul style="list-style-type: none"> • Common indicator of change of fluidization regime [2, 21, 20] • Other techniques required to confirm interpretation [20, 36]
Tribo-electric probes [3, 6, 26]	<ul style="list-style-type: none"> • Invasive • Sensitive to local hydrodynamics

the infrared, and presents a method to detect the dense emulsion phase using the camera detecting visual light, to ensure proper application of the calibration. Therefore, this method, unlike that by [25], also works for wide temperature distributions in the solid phase. The methodology to determine the solids flux is similar to that employed by Patil et al. [25], opposed to the detection of single particles by Tsuji et al. [30], which in the current application with large particle numbers is less practical. This work presents and discusses calibration results showing typical phenomena associated with the use of the technique including a demonstration for wet and dry fluidization.

2 Apparatus for measurement

The employed apparatus consists of a high-speed visual camera, a high-speed infrared camera and a pseudo-2D gas fluidized bed with a window of suitable material. The pseudo-2D geometry is required for optimal visual access. Although for this geometry the tendency of fluidized particles to stick was considered to make investigations with liquid injection problematic [3], it was found in this work that it actually can be done (also [10]). However, electrostatic charging would lead to particles sticking to the front window. In this work this was avoided by treating the particle material with alkylamine ethoxylate (EINECS 276-014-8) and the window with Catanac.

Figure 1 shows the arrangement of the cameras relative to the setup. The infrared camera is positioned with an angle to the setup to avoid self-reflection.

Table 2: Concise overview of experimental techniques for the study of the effect of liquid injection on fluidization, requiring intermittent fluidization. In this work measurement is done during fluidization.

Electro capacitance [23, 13]	<ul style="list-style-type: none"> • Detection of liquid via capacitance • Signal affected by passing bubbles • Poor spatial resolution
Conductivity measurement [18, 27, 37]	<ul style="list-style-type: none"> • Liquid detected via conductivity • Signal disturbed by fluidization • Detection of distribution of “free liquid”
Bed excavation [15]	<ul style="list-style-type: none"> • Recovery of liquid-solid agglomerates by digging • Breakage cannot be directly observed
Artificial agglomerates with RFID trackers [24]	<ul style="list-style-type: none"> • Artificial agglomerates held together by permanent magnets • Breakage can be directly observed • Agglomerates re-assembled between experiments

At the same time direct strong reflection of the LED lights into the infrared camera is avoided. Table 3 lists the main features of the cameras employed in this work. The cameras are triggered by a pulse generator to simultaneously take images. At each pulse the infrared camera takes a single image and the visual camera takes a pair at a predefined interval. In all measurements a pulse frequency of 5 Hz was employed. At this frequency the snapshots are estimated to be statistically independent for the hydrodynamics, since the bubble residence time is lower than 0.2 seconds.

The dimensions and main functional components of the center section of the experimental setup are shown schematically in Figure 2. Table 4 describes the main properties and underlying design choices. Nitrogen was selected as flu-

Table 3: Characteristics of the cameras employed in the experiments

Camera	Visual	Infrared
Wavelength range	Visual (CCD)	2.5-5.0 μm
Integr. time (μs)	370	800
Pair interval (ms)	2	not appl.
Vert. resolution	1024	512
Hor. resolution	1280	640
Bits per pixel	16	14
Memory max	2GB	PC RAM
Brand	Lavision	FLIR
Type	ImagerPro	SC7000

Figure 1: Layout of the cameras relative to the setup.

idization agent. The particle material is 1.0-1.3 mm spherical LLDPE material with a settled bulk density of 0.37 kg/L, kindly provided by Lyondell-Basell.

2.1 Selection of window material

Since the window to be used in the experimental setup needs to be transparent in wavelengths observed by either camera, the selection of these is tightly connected and predates detailed design of the experimental setup.

The material selected for the window is optically polished sapphire with a thickness of 3 mm. This material is transparent in the visual waverange and (partially) into the infrared up to about 5 μm . In addition it has high thermal conductivity of 35 W/m/K [28] and thermal diffusivity. The Knoop hardness of sapphire is higher than that of quartz [28], giving it scratch resistance. It is resistant to water, unlike some other optical materials that may have transparency over a wider range of wavelengths, found from e.g. Brown et al. [7]. The maximum size of window material commercially available has been an important factor limiting the size of the setup.

3 Interpretation and processing of thermographic images

3.1 Introduction

Theory on infrared thermography is available from a number of sources, e.g. [11]. The primary principle is that the emission of infrared radiation from an object is dependent on its temperature. This radiation can be detected and quantified. Then the detected signal can be used to obtain information on the object's

Table 4: Specification of key components of the pseudo-2D gas fluidized bed (see also Figure 2)

Nozzle	<ul style="list-style-type: none"> • Airbrush Sogolee AB200 • Orifice diameter 0.2 mm, cone angle from center-line $\pm 15^\circ$, tip pressure drop 1.5-2.5 bar • Modified for forced liquid feed instead of gravity feed • Liquid flow rate measured via coriolis meter, gas flowrate measured via mass flow controller • Connected liquid flow meter is Bronckhorst Liqui-flow with maximum capacity 600 g/hr • Capacity of connected Bronckhorst gas mass flow controller: maximum 30 NL/min
Back plate	<ul style="list-style-type: none"> • 15 mm thick aluminium, selected to provide a uniform temperature back wall that acts as a heat source to provide the heat of evaporation • Anodized black for visual contrast with white particles
Heater rods	<ul style="list-style-type: none"> • Electric, 150 Watt each • Watlow KEBG0120D008A • Combined with thermocouple and PID control to set back plate temperature (typically 80 °C)
Gas preheater	<ul style="list-style-type: none"> • Via heat tracing (versus inline) for safety • PID control to achieve preset gas inlet temperature, matching back plate temperature • Gas flowrate measured via mass flow controller
Bottom plate	<ul style="list-style-type: none"> • Sintered metal • Typical pressure drop 0.1 bar to ensure uniform fluidization • Feeding gas mass flow controller (Bronckhorst) capacity: maximum 120 NL/min
Chimney	<ul style="list-style-type: none"> • Atmospheric pressure • With arrangement to reduce the concentration of flammables below their lower explosion limits in air
Conditions	<ul style="list-style-type: none"> • Pressure: atmospheric pressure in chimney • Lower temperature limit: approximately 40 °C, below this temperature infrared reflections from the environment become significant • Upper temperature limit: approximately 100 °C, to safeguard the sealing glue

Figure 2: The experimental setup used in this work. (a) The main components of the center section of the experimental setup. (b) Photograph of the experimental setup. (c) Detail of Figure (b), showing the secondary window used in calibration (see also Section 3.4).

temperature without making direct contact with it. There are, however, some complications, as will be outlined in the following.

To introduce the complications, Figure 3 shows, side by side, two images taken at the same moment. The one on the left is the raw image taken with the camera operating in the visual wavelengths. It shows the black-colored back wall of the setup showing through the bubbles. Shadows reveal that the white emulsion phase does not have uniform density. To the right, the corresponding infrared image is shown. In the experiment the particle temperature is not uniform due to a heat sink present. Comparison of the two reveals the following phenomena, that will be taken into account in automatic image processing.

1. The heated back wall shows up as a zone of high temperature in infrared images. When determining per-position statistics for the temperature of the emulsion phase, it should be excluded.
2. In the infrared, individual particles can be discerned and around each particle there is a zone of apparently higher temperature. This perceived temperature difference is not necessarily there. The most likely physical explanation for this are reflections, causing a higher effective emissivity of cavities versus the flat particle front surface. Since attempts to reduce the reflectivity via “black body paint” seem to give disappointing results [30], this effect is intrinsic in the data for the dense emulsion phase.
3. Zones with relatively low particle density in the emulsion phase appear to have a relatively high temperature. Likely, reflections are involved and this observation cannot be trusted to be correct. Thus, such dilute zones need to be excluded from consideration for gathering automated time-averaged statistics.

Figure 3: Comparison of visual image and infrared image taken at the same instant. Particle temperature in the bed is nonuniform.

Thus, only data for the dense emulsion phase is accumulated and this data contains intrinsic variation.

In infrared thermography an electrical signal is ultimately related to the temperature of an object of interest. Figure 4 illustrates that the infrared signal as it impinges on the detector is composed of contributions not only of the object of interest, but also from the optical components passed on the way to the detector. In the present arrangement, there are two components as shown in Figure 4 for which the temperature, and therefore signal, can vary between experiments:

1. The object of interest. The relation between the object temperature and the camera signal is primarily the subject of Section 3.2.
2. The window in the setup. The interference of the window is discussed in Section 3.3.

The humidity in the air also could have a minor influence, but the laboratory is climate controlled and the camera distance is set, so that any influence is constant.

The use of a thermography model, with the assumption of constant (apparent) emissivity, is common to relate camera signal to object temperature [11]. However, since in this work the system configuration stays the same, a direct calibration approach [29] is adopted.

In the next sections the relation between the bed temperature and the camera signal and the influence of the window of the setup and the final calibration is described. The automated detection of dilute zones in the fluidized bed,

Figure 4: Illustration of the steps from infrared emission to digital signal. The window and lens absorb a fraction of incoming radiation, and emit themselves, too.

Table 5: Coefficients determined for the camera signal preconversion, equation (2).

Coefficient	Value
a_z	-3.356×10^{-5}
b_z	1.210
c_z	-287.95

such that these can be excluded from the temperature statistics, is discussed in Section 4.

3.2 Relation between camera signal and bed temperature

The power law (1) is employed to relate the average value of signal s and the window temperature T_w to the bed temperature.

$$F(T_w, s) = as^2 + bs + c + dT_w^2 + eT_w \quad (1)$$

In order to avoid the introduction of a bias into the temperature statistics as determined from the data processing (see A), a pre-correction is applied to the camera signal z to make it linear with temperature. This can be done because the camera signal recorded is essentially an arbitrary number, however monotonously increasing with increasing black body radiation intensity, so typically with object temperature. The *desired* signal value s_d is (arbitrarily) chosen to obey the linear relationship $s_d = 100T_b$.

$$s = a_z z^2 + b_z z + c_z \quad (2)$$

The constant coefficients a_z , b_z and c_z were determined in this work in preliminary data processing and are given in Table 5.

3.3 Interference of a window in thermography

The window used in the setup interferes with the infrared radiation received [30, 7], see also Figure 4. Since the window also emits radiation, this interference is dependent on the local temperature of the window. This section describes how this is accounted for in the calibration and subsequent data analysis. The analysis is based on Kirchhoff's law, which states that the sum of an object's emissivity, transmissivity and reflectivity for infrared radiation equals one. Two assumptions have been made to allow for calibration via a secondary window.

1. The effective emissivity of the object is equal to one. This is assumed since the cavities between the particles resemble the geometry by which an artificial black body object is constructed [11]. It is therefore expected that the emissivity is mostly close to one for the observed bumpy bed surface.
2. The reflectivity of the window w is assumed to be zero.

The received intensity at the lens (Figure 4) is now given by equation (3), based on Kirchhoff's law. The black body intensity $I_{b\lambda}$ is the intensity, for a particular wavelength λ , described by Planck's equation at a given temperature T_o for the object and T_w for the window. The transmissivity $\tau_{\lambda w}$ gives the fraction of the radiation coming from the object that, as shown in Figure 4, is absorbed by the window, for the given wavelength λ .

$$I_\lambda = \tau_{\lambda w} I_{b\lambda}(T_o) + (1 - \tau_{\lambda w}) I_{b\lambda}(T_w) \quad (3)$$

If for the calibration experiment it is assumed that $T_w = T_o$, equation (3) allows for the introduction of a second window in the calibration experiment, to determine the coefficients in equation (1), such that the contribution of the window is not incorrectly lumped as originating from the object.

In actual experiments a significant temperature difference during liquid injection can be expected over the different sections of the bed. In experiments in which temperature distributions within the emulsion phase are to be observed, there is usually no temperature measurement of the window since this would hinder visual access. Therefore, the preferred assumption is that the temperature of the window is locally equal to the time averaged temperature and constant. The following explains how the local contribution, to be applied to account for the window, is determined.

Calibration experiments will yield the coefficients in equation (1). In subsequent experiments the time-averaged temperature profile in the fluidized bed is to be determined. The experiment yields a distribution of camera signals $r(s)$, for a given position. The time-averaged temperature follows from equation (5).

$$\langle T_b \rangle = dT_w^2 + eT_w + \int_s r(s) (as^2 + bs + c) ds \quad (4)$$

$$= \langle as^2 + bs + c \rangle + dT_w^2 + eT_w \quad (5)$$

In absense of an estimate for T_w , equation (5) cannot be evaluated. However, substituting the assumption $T_w = \langle T_b \rangle$ into equation (5) yields equation (6).

$$0 = \langle as^2 + bs + c \rangle + dT_w^2 + (e - 1) T_w \quad (6)$$

For a given experiment, all terms in equation (6) except $T_w (= \langle T_b \rangle)$ are known. The equation has two solutions for T_w . The solution to be selected is the one for which T_w falls within the range of calibrated temperatures.

3.4 Calibration procedure

Using the procedure outlined above detailing how a secondary window can be introduced to delump the coefficients in equation (1), this section describes the resulting calibration procedure. The calibration procedure essentially gathers signal data s for various values of T_b and T_w . Steps in the infrared calibration experiment are as follows:

1. Let the bed fluidize at a predetermined temperature.
2. Arrange the cameras, setup and secondary window as shown in Figure 2. The secondary window in front of the setup, preheated in an oven, is cooling to surroundings.
3. Monitor the temperature of the cooling secondary window. Take a sequence of images for each in the set of predefined temperatures of interest for the secondary window. Measurement is started when the temperature is just above the predefined temperature, and stopped when it is just below.
4. Repeat for the next prescribed temperature of the particle bed.

Figure 2 demonstrates the experimental arrangement employed. As value for the temperature of the particle bed, the temperature of the window at the front of the experimental setup is used, since it is in thermal contact with the front particles. For both the particle bed and the window in front the temperature is measured with a microthermocouple taped to the respective window. Figure 5 shows the custom-made interface employed on the computer for extracting the infrared signal for the calibration measurements. The user indicates the zone in which the window interferes with the bed signal, shown as a square. The other contours shown indicate zones in which bubbles are detected, and from which no samples are taken by the software, detailed in the next section. Due to light normalization, the thermocouple tape (silver colored, see Figure 2) now has practically the same brightness as the bed in the visual image.

4 Digital image processing and analysis

4.1 Introduction

Digital image processing is applied to select and extract data from the images obtained with the visual and the infrared camera. Information from the simultaneously taken visual images is applied in processing the infrared images. The first required step to transfer information from the visual images to the infrared images, is alignment to a standardized grid, corresponding to the lower part of the bed, width and height 80 mm, via a user-provided affine transform (for the head-on visual camera) or perspective transform (for the infrared camera). In both cases this yields a smaller-than-original image, so that only bilinear [5]

(a)

(b)

Figure 5: User interface for infrared calibration. For infrared calibration, the arrangement of the experimental apparatus (Figure 2) is such that the secondary window interferes in the right half of the infrared image (left image in (a) and (b)). The user is to indicate a subzone for sampling (the square). On the right the corresponding visual images. In (a) the temperature of the secondary window is higher than that of the bed, in (b) it is lower.

interpolation is done. The size is 512×512 pixels for visual images, 256×256 pixels for infrared images. Because of the latter lower resolution, the masks obtained from visual images (Section 4.2), representing the same alignment with the experimental setup, are scaled down, before they are applied to determine whether a measured value needs to be discarded in the determination of statistics. With both the visual and infrared image aligned to the same representation of the experimental setup, they can also be conveniently shown side-by-side by scaling, e.g. Figure 5.

The visual images in this work have three distinct applications, each with its own image pre-processing steps. The steps for each method are contained in Table 6. Two applications are related to detection of dense emulsion phase, via thresholding and width of signal distribution. These are introduced in Figure 6 and explained further in Section 4.2. The third application is the particle image velocimetry (PIV), another form of image analysis (Section 4.4). Common to all is light normalization (see e.g. [17]), to correct for nonuniform lighting. Processing of infrared images is discussed in Section 4.5. All image processing operations were achieved via custom-written software employing the open source OpenCV library [4] in C++. The single exception is the application of PIV software, for which Davis by LaVision was used.

4.2 Dense emulsion phase detection

Detection of dense emulsion phase is achieved in this work by two simultaneously employed methods.

The first method is standard per-pixel thresholding (e.g. [17]) on intensity, after blurring with a mask of 3×3 pixels, corresponding with a quarter of the size of a particle in the image. Thresholding is based on a user-supplied threshold value. Subsequently erosion and dilation are applied primarily to enlarge the single pixels detected by the thresholding into zones including the borders of the bubble. This method works well for deep bubbles and is mainly used as pre-processing of images to be loaded for the particle image velocimetry analysis.

The second method has its primary application for discarding zones for which the infrared calibration will be invalid. Figure 7 demonstrates the principle of the method. It is based on the width of distribution of signals in a given interrogation zone, for which the standard deviation is a suitable quantitative measure. Only if this width surpasses a user-defined value, the center pixel of the zone is considered to be part of the dilute emulsion phase. In other words, thresholding is applied onto the width of signal distribution in the enveloping zone, instead of light intensity. Since within zones in which only the single black color of the back plate shows this is zero, the method would be expected to fail for large bubbles. However, in combination with contour detection (Section 4.3), it also allows for detection of large bubbles, but cannot distinguish those from shallow ones, because the borders of large bubbles are detected as being dilute emulsion phase.

4.3 The use of contours

The thresholding operations of Section 4.2 yield binary pictures, in which zones with pixels to be disregarded in subsequent analysis are zero, and others are

Figure 6: Demonstration of sub-steps in visual image processing (Table 6), for the same case as image 3. Figure (a) shows the raw image. Figures (b) and (c) shows the image aligned to the standard grid and with light intensity correction applied. In addition, Figure (b) shows the outlines of dilute zones detected from thresholding on intensity, as well as detection of dilute zones from the distribution of signals. Figure (c) is a raw image as it was loaded into the PIV analysis. Inside the contours of zones detected from thresholding (see Figure (b)), intensity was set to zero, setting the image locally dark.

Table 6: Summary of settings for analysis of visual images.

Method	Threshold	Distrib.	PIV
Image	512×512 px	Same	Same
Size	80x80 mm	Same	Same
Pre	Affine	Same	Same
	Normalize	Same	Same
	Blur (3x3px)	Mirror	Threshold
Zone	1x1 px	15x15px	32x32 px
Post	Erode (3px, 3it)		Weighing
	Dilate (3px, 1it)		
	Fill contours	Same	
Used in	Infrared & PIV	Infrared	Result

With:

Pre	Pretreatment before main method is applied
Zone	Source data zone for single point result
Post	Methods allways applied after main method
px	Pixel
Threshold	Ordinary per-pixel thresholding
Distrib.	Emulsion phase detection from distribution
PIV	Particle image velocimetry
Affine	Affine transform, aligns image to grid
Normalize	Per-pixel correction for light conditions
Blur	Replace a pixel intensity by a local average
Erode	Operation expanding dark zones of image
Dilate	Operation expanding light zones of image
Fill	Set bubble interiors to zero intensity
Mirror	Expand image by mirroring at original edges
Weighing	Accounting for solid phase fraction

Figure 7: Principle of dense emulsion phase detection via width of distribution. Dense and dilute emulsion phase can be distinguished by evaluating the width of the histogram obtained for a small sub-rectangle, here colored black to pinpoint it, of the image. A suitable quantitative method is thresholding for the standard deviation calculated for the values composing the histogram.

Figure 8: The upper left quarter of the 16x16 grid applied for particle image velocimetry on the processed image as fed to the PIV software. See also Figure 6 to see how the image was composed. The fraction non-black pixels f is employed to correct the solids flux for the amount of solids locally present (equation (7)).

one. Around these zones contours can be drawn via automated image analysis. These subsequently can be conveniently projected onto the original image to provide user feedback, as in Figure 6, or similarly onto the aligned infrared image as in Figure 5. Contours resulting from thresholding on the width of signal distribution in this work are shown in purple, those from thresholding on light intensity in blue.

Automated contour detection also provides the means to discard heavy particle raining through the bubbles. In this work those are also discarded from statistics for infrared thermography because of their potential for reflection onto particles moving not close to the front window.

4.4 Image velocimetry and solids flux

Particle image velocimetry employs two images of a collection of particles taken in quick succession at time interval Δt_p . Between these images cross-correlation analysis of the intensity I is used to determine the most probable displacement $\bar{x}$ of the particles between the images [34].

This work reports the emulsion phase solids flux instead of the solids velocity. For this purpose it is essential to have an estimate of the amount of solids found in the sub-image. Therefore, from the first image in the pair the fraction f_e representing emulsion phase is determined from thresholding, as indicated in Figure 8. In line with prior work [17] the observed solids flux is reported in the unit of velocity. The emulsion phase solids flux $\bar{u}$ is calculated from equation (7).

$$\bar{u} = \frac{f_e}{\Delta t_p} \bar{x} \quad (7)$$

Figure 9: Calibration data for the infrared camera at distance 110cm.

4.5 Image processing on infrared images

The infrared images contain raw camera data and show little contrast when displayed directly on screen. Therefore, in cases where only a qualitative display of the infrared data is required, a histogram equalization has been applied. This is carried out for all grayscale representations of infrared data reported in this work, also e.g. Figure 5.

5 Results for calibration

5.1 Infrared calibration for T_b and T_w

In order to obtain raw data to develop the relation between window temperature, received infrared signal and bed temperature, the procedure outlined in Section 3.4 was performed. The background fluidization gas mass flux was set to $0.382 \text{ kg}/(\text{m}^2\text{s})$, giving bubbling fluidization with significantly large zones of dense emulsion phase. The secondary window in front of the setup was pre-heated in an oven to 100°C . The raw data was filtered to yield data relating to dense emulsion phase only using the method outlined in Section 4.2.

The data thus gathered is used to fit F (equation (1)). The raw calibration data is summarized in Figures 9 and 10, for infrared camera distances 55 cm and 110 cm (see Figure 1), respectively. The box and whisker plots indicate the first, second and third quartile, and the minimum and maximum. Also shown are isocontours, at intervals for the bed temperature of 10°C for the calibration

Figure 10: Calibration data for the infrared camera at distance 55 cm.

Table 7: Coefficients for equation (1), for distances L between the camera and the fluidized bed. Temperature in $^{\circ}\text{C}$.

L (cm)	55	110
a	7.5350×10^{-8}	2.1520×10^{-7}
b	0.01321	0.01160
c	-4.6328	2.5427
d	-1.5549×10^{-3}	-7.5061×10^{-4}
e	-0.1630	-0.2593

plane fitted. It is seen that the nonlinearity is limited and the used second order polynomial serves well to describe the pre-corrected (Section 3.2) response of the camera to the temperature of the bed and window. Table 7 gives the coefficients as determined for use in equation (1).

Figures 9 and 10 present calibration diagrams for two distances of the infrared camera to the setup, giving a very similar calibration curve for either case. It shows that the calibration can be obtained repeatedly and additionally that there is not any major influence that can be ascribed to the distance of the infrared camera to the setup. Figure 10 shows a duplicate measurement for distance 55 cm, as well. Reproducibility is reasonable, in particular for the region with bed- and window temperature in the range of $50 - 70^{\circ}\text{C}$.

However, there is also an increase in the width of the distribution of signals with bed temperature. Figures 9 and 10 suggest that this is dependent on the bed temperature, and not the window temperature. Table 8 shows that the width of the distribution increases much stronger with bed temperature than does the slope of signal versus bed temperature. The increase in the width of the distribution, together with increasing the difference of bed temperature relative to the environment, is possibly related to an increasing width of distribution of the actual particle temperature, due to particles cooling at the setup window, at which the bed temperature is measured. It was verified that indeed in raw instantaneous infrared images a distribution is found for the signals originating from positions showing the centers of particles, at which the influence of reflections is expected to be minor (as exploited by Patil et al. [25]). In other words, the inherent width of distribution is possibly only in small part due to cavities.

Table 8 also shows that the width of the distribution is poorly reproducible. The origin of this difference is not known. It was verified that it is not clearly dependent on the location at the front window for which the samples are taken. The implication is that not only there is always an inherent background fluctuation in the signal, which will be translated into a temperature fluctuation, even if the bed temperature is uniform. This background fluctuation is also dependent on bed temperature (distribution) and, possibly, camera arrangement. Temperature statistics other than the average can therefore only serve as a qualitative indicator.

5.2 Effect of reflectivity of the window

In Section 3.3, a simplification was made by omitting the reflectivity of the windows, while simultaneously assuming $e_b \approx 1$. In order to evaluate the effect of this assumption, data was sampled for images with known bed temperature

Figure 11: Comparison between calibration curve and measured signals, in absence of the secondary window introduced for the calibration procedure. Measured signal distributions are shown via boxplots (see Section 5.1) for various bed temperatures as measured at the bed window. The correlation F determined in the calibration, giving the relation between bed temperature and expected average signal. Also shown is a correlation Fc that was fitted to the experimental signal distributions as shown.

Table 8: Evaluation of change of width of signal distribution with temperature, for the signal distributions as given in Figures 9 and 10.

	T bed °C	Median σ s	Median average	ds/dT_b °C ⁻¹
L=55cm	60	134.6	6101.4	70.8
	70	255.6	6382.9	70.5
	80	267.5	7469.0	69.7
L=55cm repeat	60	98.0	5850.1	70.7
	70	110.4	6532.0	73.9
	80	119.6	7379.0	78.2
L=110cm	60	49.6	5840.5	70.8
	70	65.7	6624.3	69.2
	80	82.8	7248.9	67.9

from the zone without interference of the secondary window. This is compared with the correlation obtained from equation (1) while imposing $T_w = T_b$. The correlation should now closely predict the average of the distribution of signals obtained for each bed temperature. This results in Figure 11 for the calibration experiment with the camera positioned at a distance of 110 cm.

As expected for nonzero reflectivity and $e_b \approx 1$, the correlation F underpredicts the signal distribution as actually found for any given bed temperature and the deviation increases as the difference of bed temperature and room temperature increases. It could therefore be proposed to perform a correction for the coefficients in equation (1), such that the coefficients for the window (d and e) are unaffected.

However, since the correlation F crosses the lower quarter of the signal distribution for each bed temperature T_b , the deviation due to the simplification on reflectivity counterbalances the deviation due to particles at actually higher temperature having been present during calibration (Section 5.1). For this reason, the coefficients a , b , c , d and e as determined in Section 5.1 are left in place and are used in the remainder of this work.

6 Results for fluidization

6.1 Statistical definitions for PIV notation

This section clarifies the notation to be used in particular to represent the PIV results in the following sections. The background is that in fluidized beds the flow characteristics vary considerably both in space and time [31]. The dynamics are of interest by themselves, for example for evaluating whether the average is representative for the typical condition of the system. Before proceeding to results for fluidized bed dynamics, the definition of some key parameters will be given together with their visual representation.

As a measure for the spread and the skewness for samples taken from a population of individual measurements, in this work the standard deviation σ_i

Figure 12: Application of equations (8), (9) and (11) on a flux distribution. The standard deviation σ has no sign and is drawn symmetrically around the average μ . The modified third moment τ , represented as a star, is drawn relative to μ . Its negative value represents the leftward skewness of the shown distribution.

and the parameter τ_i , based on the third moment [33], are used.

$$\mu = \frac{1}{n} \sum x_i \quad (8)$$

$$\sigma = \sqrt{\frac{1}{n-1} \left(-n\mu^2 + \sum x_i^2 \right)} \quad (9)$$

$$\begin{aligned} M_3 &= \frac{1}{n-1} \sum (x_i - \mu)^3 \\ &= \frac{1}{n-1} \left(\sum x_i^3 - 3\mu \sum x_i^2 + 2n\mu^3 \right) \end{aligned} \quad (10)$$

$$\tau = \text{sgn}(M_3) \sqrt[3]{\text{sgn}(M_3) M_3} \quad (11)$$

Figure 12 demonstrates the application of equations (8), (9) and (11) on a typical measured one-dimensional flux distribution in a single direction. In particular, it illustrates the modified third moment as a parameter to indicate the skewness of a distribution. Figure 13 shows the notation for a decomposition of two distributions into its orthogonal components. The average is shown as a single vector. For visualization of the two components of the standard deviation, σ_x and σ_y , a box of corresponding relative size in the x and y direction is used. The modified third moment is represented as two orthogonal vectors.

6.2 Notation for temperature statistics

For the temperature statistics, results obtained from equations (8), (9) and (11) are presented on a truncated color scale. For the determination of σ and τ

Figure 13: Notation of μ , σ and τ for combination in two dimensions of two single distributions as shown in Figure 12. μ is represented as a single arrow, σ as a rectangle and τ as a combination of two orthogonal arrows.

Table 9: Key numbers for demonstration cases presented in sections 6.3 and 6.4.

Case	Dry	Wet
# raw IR images	1020	835
Sample rate (Hz)	5	5
Measured time span (s)	204	167
Bottom N ₂ flux (kg/m ² /s)	0.764	0.764
Nozzle N ₂ flow (g/min)	0	3.44
Nozzle liq. flow (g/min)	0	6.3
Back wall T set (°C)	70	80
Settled bulk volume (mL)	90	60
Camera distance (cm)	55	110

the intrinsic fluctuations cannot be readily filtered out and these statistics are only useful, as qualitative indicator, if the fluctuation due to actual temperature differences exceeds that due to the intrinsic signal distribution.

6.3 Application to dry fluidization

The first example of the application of the technique is fluidization without any injected liquid, i.e. “dry” fluidization. The goal is to evaluate whether, as would be expected, a uniform temperature is found. Also, this provides a reference case for comparison with temperature data with liquid injection, which will be reported in Section 6.4. Table 9 summarizes key conditions for the demonstration cases.

The digital image analysis provides information on typical paths followed by bubbles rising in the fluidized bed. Figure 14 shows this for dry fluidization. Positions for which no dense emulsion phase samples was detected at all, are shown in light blue (in all figures). The number of samples found near the left and right wall is low, due to low density of the emulsion phase detected in this region. An exception is seen in the lower left corner, showing a large fraction of samples as dense emulsion phase. This is due to glue on the window and

Figure 14: Fraction of samples for which dense emulsion phase was detected, per position, for dry fluidization. See Table 9 for conditions.

this zone should be ignored for this experiment. Also near the bottom plate, in particular on the lower right, there is a zone in which no dense emulsion phase was detected. From Figure 14 it is apparent that the flow pattern is not entirely symmetrical which most likely is due to non-uniformities in the bottom distributor plate.

The emulsion phase flow pattern (Figure 15) exhibits the same asymmetry, suggesting more gas enters the bed on the right than on the left. The standard deviation σ in Figure 15 shows that fluctuations are of the same order of magnitude as the average. These are significant and will therefore also be reported for the case with liquid injection. The modified third moment indicates that the distribution of the solids flux is skewed towards the direction of the average solids flux.

Calibration experiments (Section 5.1) revealed that also when there is no forced cooling (by liquid evaporation), the instantaneous particle temperature in the bed is not uniform. In addition to that, Figure 16 reveals that minor (time-averaged) temperature gradients exist in the bed. The local variation in particle temperature, along with the effect of cavities, causes the standard deviation seen in Figure 16 (b). The result for the modified third moment is shown to serve as reference for a case with liquid injection, see Section 6.4. Its value, being generally positive, corresponds with the density distribution of observed temperatures with skewness towards higher temperatures (Figure 17). The density distribution shows a wide distribution of temperatures calculated for single data points. After averaging, the intrinsic distribution averages out and an average temperature of 68.6°C is found.

6.4 Application to fluidization with liquid injection

As explained in the introduction, it is desired to have an experimental technique that can non-invasively pinpoint any heat transfer limitations and solid flux

Figure 15: Statistics for solids flux, for fluidization without liquid injection with the wall temperature set at 70 °C. Conditions can be found in Table 9.

Figure 16: Temperature statistics found for dry fluidization. Same conditions as Figure 15.

Figure 17: Probability density distribution for the temperatures calculated to compose Figure 16. The blue line indicates the mean.

associated with liquid injection in gas fluidized beds. For this purpose the setup and technique were developed. This section demonstrates the injection can be done, and an example of results is obtained. It explores the effect of injection of isopentane (boiling point 28°C [32]) on the temperature distribution in the fluidized bed. Details on the conditions applied in the experiment with liquid injection can be found in Table 9. The liquid injection nozzle is mounted as shown in Figure 2 and sprays down onto the particle bed from the top right.

The fraction of images that contains dense emulsion phase, per position, is not shown for brevity. It indicates that as in Section 6.3, there is an asymmetry in gas distribution in the bottom plate. The emulsion phase flow characteristics are shown in Figure 18 and show the expected typical circulation pattern in the emulsion phase with upflow in the center and downflow in the vicinity of the walls. The liquid injection does not lead to a strong asymmetry in the emulsion phase flow pattern. As in the dry fluidization case, there is again a slight asymmetry that may be expected from the non-uniform distribution of gas.

At the same time, the effect of liquid injection is very clearly noticeable in the observed temperature distribution. To demonstrate the added value of the combination of visual and infrared, Figure 19 shows a snapshot of the bed during the injection of isopentane. In the visual, nothing special is observed. On the contrary, in the infrared clusters of cold particles and a temperature gradient from the walls to the bed centre are observed. The sampling frequency (Table 9) was too low to track such clusters, which also tend to move behind the front layer (out of sight), in relation to the point of liquid injection. However, their abundance (and absence without liquid injection) makes it likely that they form as a consequence of liquid injection.

The modified third moment (Figure 20) shows very negative values along the side walls, where colder particles originating from the top of the bed descend. This is more pronounced on the right, at which side the liquid impinges on top of

the bed, than on the left. A negative value of the modified third moment means that the temperature distribution observed for the particular position is skewed towards lower-than average temperatures. This is for example the case if single isolated strongly cooled particles move around an emulsion phase of otherwise rather uniform temperature. A strong negative value was not observed for dry fluidization (Figure 20); the skew in the distribution of observed temperatures changes when liquid injection is introduced.

Contrary to the case without liquid injection, in which the standard deviation was rather uniform throughout the bed, the fluctuation of particle temperature, as found from the standard deviation, now exhibits a clear maximum. It is now strongly biased to the right, in particular the top right, featuring there relatively high values.

The time-average of particle temperature shows a local minimum at the top right and a very strong gradient near the wall. This together demonstrates that there are heat transfer limitations in the fluidized bed in supplying heat to the region of liquid injection where reheating of the particles occurs after they have been convected into the bulk, away from the region of injection.

7 Conclusions

A powerful non-invasive monitoring technique was developed for the simultaneous study of hydrodynamic and thermal effects of liquid injection in dense gas-fluidized beds. The technique is based on the simultaneous application of a high speed visual camera and a high-speed infrared camera. A direct calibration approach was adopted for thermography. Due to intrinsic variation in the data, it is strongly beneficial if the camera signal is linear with respect to temperature for statistics to be derived. Simultaneous application of a visual camera and an infrared camera, followed by digital image analysis, is necessary to avoid improper interpretation. A further improvement would be the ability to directly determine, and correct for, window temperature, which was found to have a noticeable effect on the average temperature determined.

Liquid could successfully be injected into a pseudo-2D experimental arrangement. The mechanism of local cooling and re-heating of the emulsion phase was identified and quantified with the developed technique.

The demonstrated non-invasive technique can be employed to derive instantaneous whole-field quantitative data for model verification. The technique provides observations that enhance the understanding of the mechanism of liquid injection and subsequent evaporation in the bed.

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Figure 18: Solids flux for fluidization with liquid injection. Conditions can be found in Table 9.

Figure 19: Temperature distribution as determined from the signal distribution in an instantaneous infrared image. No emulsion detection has been applied. Single cooled particles, as well as clusters of these, are seen to move about the emulsion phase when liquid injection is applied. Same conditions as for Figure 18.

A Temperature statistics from data from the emulsion phase

All equipment being the same in every experiment, the signal is expected to be dependent on the temperature of the particle bed, T_b and that of the window, T_w (assumed constant here). This is supplemented by a variation due to the effect of cavities, as discussed before. The latter leads to a probability distribution of finding a certain signal for a given bed and window temperature. This is conceptually illustrated for four bed temperatures in Figure 21. The relation $F(T_w, s)$, also shown in Figure 21, gives the probability-average value of T_b corresponding to a single signal value s . In this work F is obtained via least squares fitting of a power law equation (1), with set s and T_w , and T_b the dependent variable. Although equation (1) allows for nonlinearity, it is advantageous if it is linear in s for the determination of statistics. For example, from experimental data, given as distribution $r(s)$ the average would be determined by integration as given by equation (12), if f would be known. Instead, avoiding the need to determine f , approximation (13) is employed.

$$\langle T_b \rangle = \int_s \int_{T_b} r(s) T_b f(T_b, T_w, s) dT_b ds \quad (12)$$

$$\approx \int_s r(s) F(T_w, s) ds \quad (13)$$

Figure 20: Statistics on temperature dynamics for isopentane injection from the top. Conditions can be found in Table 9. Same conditions as for Figure 18.

Probability for finding bed signal with given T_b

Figure 21: Probability distribution for finding a bed signal for given bed temperature T_b . Sampled for 3 bed temperatures and two signals. Also shown is the calibration relation F , crossing the probability-averaged value of T_b for each s . For this example, T_w was assumed to have a single value.

If F also represents the probability-average of signal s for given T_b , in addition to the probability-average T_b for given s , then the approximation given in equation (13) is exact. This is the case when F is linear in s . This can be achieved by the introduction of a pre-correction to the raw infrared camera signal z , using equation (2).

List of symbols

Greek symbols

λ	Wavelength
σ	Standard deviation
τ	Equation (11)
τ	Transmissivity

Alphabetical symbols

a	Coefficient	n	Number of samples
b	Coefficient	r	Signal distribution
b	Bed	s	Signal (pre-corrected)
c	Coefficient	t	Time
d	Coefficient	T	Temperature
e	Coefficient	u	Velocity
e	Emissivity	x	Displacement
f	Volume fraction	x	Sample value
f	Probability density	x	Horizontal direction
F	Equation (1)	y	Vertical direction
i	Index	w	Window
I	Infrared flux	z	Raw camera signal
j	Index		

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