Gravimetric Traceability for Optical Measurements of Droplets-in-Flight

Michael Verkouteren, Jennifer Verkouteren, and Cynthia Zeissler

Material Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, MD 20899

ABSTRACT

We have improved the uncertainty of optical sizing methods of nanogram-sized droplets-in-flight by shifting measurement traceability from standard dimensional artifacts to droplet mass realized by inkjet dispensing and microgravimetry. In our case, total uncertainty was reduced from 6 % to 3 %. Using gravimetric and optical approaches, we compared measured diameters in droplets from fluids having a range of optical properties similar to those in specialized inks and nanoparticle suspensions. Results indicate that this mechanism of control may benefit inkjet applications in microscale additive manufacturing.

Keywords: calibration, gravimetry, inkjet, optics, traceability

1 INTRODUCTION

Inkjet technologies are finding wide application in additive manufacturing, drug discovery, microchemical engineering, low-cost display assembly, and small-volume analytical assays. Optical sizing of droplets-in-flight is critical to quantitative dispensing yet confounded by many factors. To start, the uncertainty in the standard artifact used for dimensional calibration is often significant; depending on the quality of the calibration this inherent baseline uncertainty can amount to 5 % or more. Secondly, optical measurements are influenced by optical refraction, reflections and scattering, image resolution limitations and pixelation, focusing effects, uneven illumination, and geometric asymmetries in the objects and detector arrays. Some of these factors, including object translucence and opacity, have been difficult to study quantitatively. For many optical systems, these factors can add another 5 % to the uncertainty [1]. While a precise potical system with a combined absolute uncertainty of 10 % may be tolerable, it places limitations on applications that may require accurate mass deposition control, such as printed microelectronics. We have attempted to address this issue by using inkjet deposition and microgravimetry [2] to calibrate an optical system, therby reducing its inherent baseline uncertainty to 1 %. Then, we designed a study to observe whether the fluidic properties of translucence and opacity influenced optical droplet measurements. We have utilized several fluids with exhibit extreme optical properties and compared droplet diameters as determined by gravimetry and optical tranmittance. Ray diagrams have been used to help explain results.

2 EXPERIMENTAL

Droplets were dispensed at 500 Hz from a drop-on-demand inkjet device having a 50 µm diameter orifice (Microfab Technologies) under a precise pressure control system [3]. (Certain commercial equipment, instruments, or materials are identified in this document. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.) Driving waveforms were adjusted to give simple droplets (without satellites) delivered at low velocities near 1 m/s in order to optimize droplet roundness (aspect ratio < 1.01). Mean droplet mass was determined by measuring 20,000 droplets on a submicrogram balance (Sartorius model SE 2-F) with correction for evaporation [2]. Droplet characteristics were measured using an advanced imaging microscope with strobed illumination (JetXpert, ImageXpert, Inc). Standard polystyrene spheres (Duke Scientific) mounted on a thin film provided the initial optical calibration. Fluids and inks with various levels of black and colored pigments were utilized, with densities determined through gravimetric measurements of aliquots dispensed through a calibrated pipette. Optical focusing was controlled using a piezoelectric nano-positioning device, and digital images were analyzed through ImagePro Plus (Media Cybernetics).

Our optical system gave backlit images of the droplets in flight. A droplet appeared as a dark object on a light background, and when the fluid was translucent, the droplet acted as a spherical lens with the illuminator focused in front of the droplet (such as the LED array seen with the polystyrene spheres in Figure 1). Even under the best conditions of focusing and illumination, droplet boundaries exhibited pixelation in high-resolution images and possessed a distribution of grayscale levels across the droplet boundary that acted to obscure the location (i.e. the grayscale threshold value) of the true boundary. Using gravimetry, we determined an estimate of the true diameter (hence the true grayscale value of the boundary) through Equation 1, where D is the gravimetrically-realized diameter, m is the droplet mass, and ρ is the fluid density.

$$D = \sqrt[3]{6m/\pi\rho} \tag{1}$$

The values of D were then compared with optical values of diameter d determined with translucent and opaque droplets.

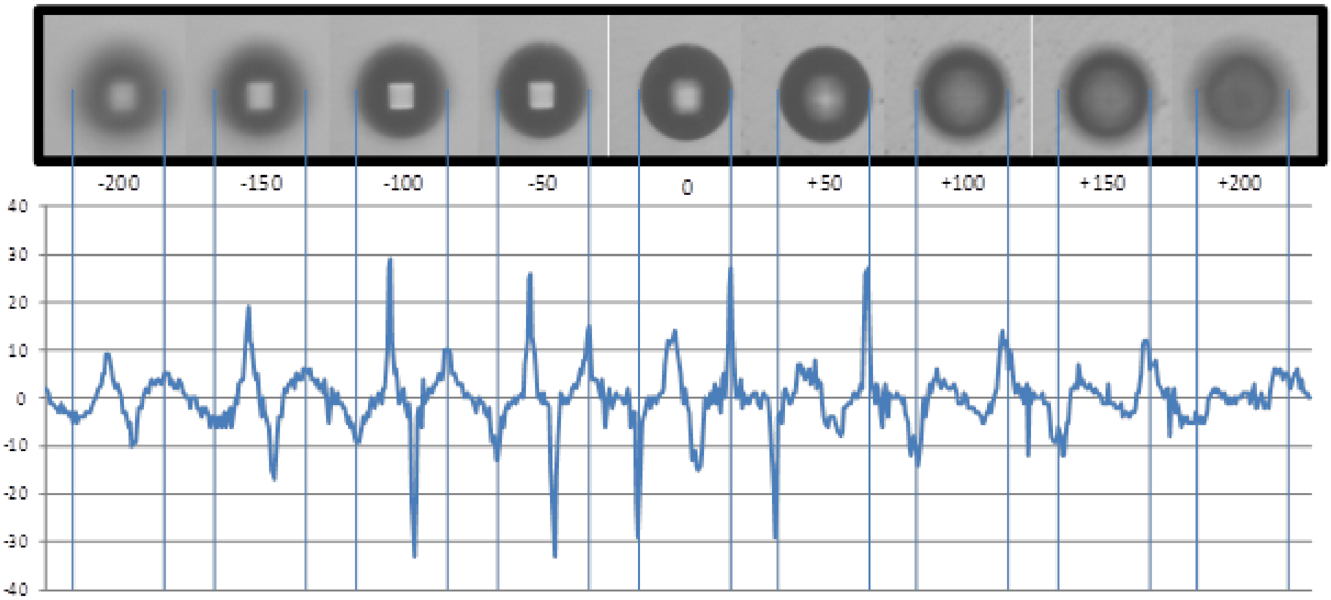

Figure 1: Focusing effects in objects illuminated by backlight transmission. At top are images of an 81.3 μm diameter polystyrene sphere at discrete displacements from the focal plane, and at bottom are the horizontal differential grayscale profile through the center of these images. Here, optimum focus of the sphere was between 0 and +50, while the optimum focus of the backlight array was between -50 and -100 (about 100 μm in front of the sphere).

Optical diameters were measured by two methods. The first method is depicted in Figure 2. A horizontal profile of grayscale pixel values was plotted from a well-focused image of a droplet-in-flight. Means of the lightest and darkest pixel grayscale populations were determined (G_{max} and G_{min}, respectively), then the grayscale values at 20 %, 50 %, and 80 % of the grayscale range were calculated through Equation 2, where x is the percentage value. The sub-pixel horizontal positions $P_L(x)$ and $P_R(x)$

$$G(x) = G_{\min} + \frac{x}{100}(G_{\max} - G_{\min}) \tag{2}$$

corresponding to the value of $G(x)$ on the left and right side of the droplet boundary were then determined through linear interpolation of the two pixels with grayscale values closest to $G(x)$. The optically-determined diameter $d(x)$ was then calculated through Equation 3, where k was the optical calibration factor.

$$d(x) = k\{P_R(x) - P_L(x)\} \tag{3}$$

The second method for determining optical diameter was the Otsu approach [4], which is the default method in many image analysis routines. The Otsu approach sets the boundary where the combined variance of the inside and outside pixel populations on either side of that boundary is minimized. This is difficult to envision in Figure 2, but results in the Otsu diameter being somewhat under the d(50) diameter when excessive pixel variation exists within the droplet boundary, such as when a central illumination peak exists.

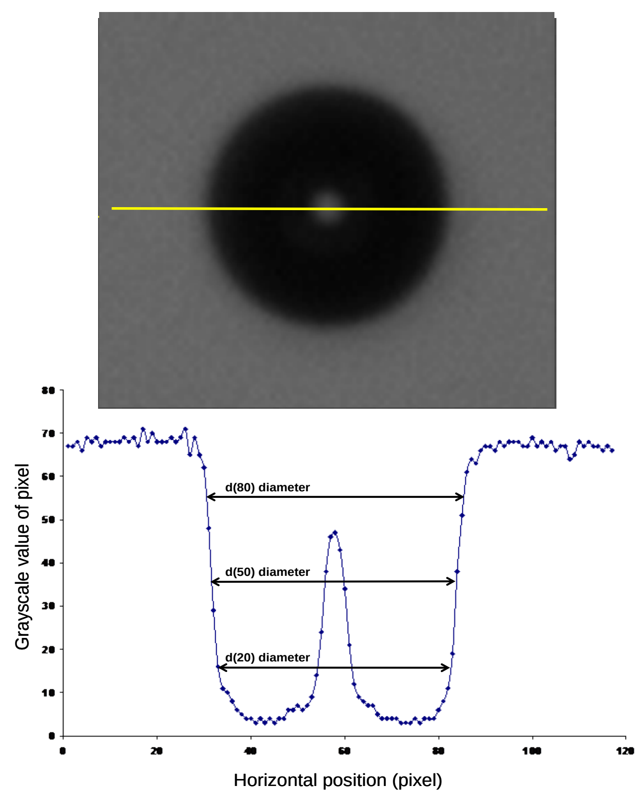

Figure 2: (top) An image of a translucent droplet-in-flight and location of the horizontal grayscale profile, and (bottom) that profile plotted along with values of the optical diameters at various grayscale thresholds.

Table 1: Gravimetric and optical measurement results for three fluids

Fluid	Droplet mass (m, ng) and standard uncertainty n=5	Density (ρ, g/mL) and standard uncertainty	Grav.diameter (D, μm) and expanded combined uncertainty	Optical thresholding method	Optical diameter (d, μm) and expanded combined uncertainty
Isobutanol (transparent)	34.65 (0.39)	0.806 (0.002)	43.46 (0.40)	*d* (80)	47.9 (2.9)
				d (50)	44.7 (2.7)
				d (20)	41.7 (2.5)
				Otsu	44.4 (2.7)
	35.05 (0.37)		43.63 (0.38)	*d* (80)	48.4 (2.9)
				d (50)	44.8 (2.7)
				d (20)	41.2 (2.5)
				Otsu	44.4 (2.7)
Spectra XL-30 blue ink (translucent)	75.73 (0.23)	1.06 (0.03)	51.48 (1.08)	*d* (80)	55.5 (3.3)
				d (50)	52.9 (3.2)
				d (20)	50.3 (3.0)
				Otsu	51.9 (3.1)
	75.44 (0.20)		51.42 (1.02)	*d* (80)	54.9 (3.3)
				d (50)	52.3 (3.1)
				d (20)	49.6 (3.0)
				Otsu	51.8 (3.1)
Generic black ink (opaque)	41.22 (0.17)	1.04 (0.01)	42.32 (0.43)	*d* (80)	46.6 (2.8)
				d (50)	43.6 (2.6)
				d (20)	40.4 (2.4)
				Otsu	42.9 (2.6)
	50.39 (0.26)		45.25 (0.54)	*d* (80)	50.1 (3.0)
				d (50)	46.6 (2.8)
				d (20)	42.8 (2.6)
				Otsu	45.4 (2.7)

3 RESULTS AND DISCUSSION

Gravimetric and optical measurement results are displayed in Table 1. Real differences in droplet size between the three fluids reported were due to differences in inkjet waveform parameters optimized for the distinct rheological properties of each fluid. These size differences were unimportant; rather, we were interested in comparing the droplet size estimates calculated through the different methods on the same droplets. All combined uncertainties include Type A and Type B evaluations of error [5]. As propagated through Eq. 1, the expanded combined uncertainty in a gravimetric D value was equal to one third of the combined relative uncertainty in m and ρ, which was then multiplied (expanded) by a factor of 2.

The resolution of our optical system was 1.003 μm per pixel. Since our droplets were about 40 to 50 μm in diameter, optical measurement resolution was limited to about 2 %. Imprecisions in repeated optical measurements reflected this value. The standard polystyrene microspheres that provided optical calibration had an inherent expanded uncertainty of 4.4 %. Without adding further factors, the expanded combined uncertainty of a d determination was estimated at 6 %.

Not surprisingly, inspection of results in Table 1 reveals that the gravimetric D values and optical d values are very similar; in fact, the D values fall in a narrow range between the d(20) and d(50) values. By plotting the each D value onto the d(20)-d(50)-d(80) linear relationship, all D values plotted between d(33) and d(41). To set the d(50) value to reflect the D diameter, the reference value of the optical artifact was lowered by 2.9 % (well within its reported uncertainty). The Otsu values perhaps were more influenced by the optical differences between the fluids: these values were about 2 % higher than the D values for the transparent fluid, yet only about 0.8 % higher for the opaque ink (they were 0.8 % higher in the translucent ink, but this result was less significant due to higher uncertainty stemming from difficulties with the density measurement). This subtle difference would probably be unnoticed except for the consistency in measurement that gravimetry affords. We theorize that the extra variation in the pixel grayscale population within the transparent droplets leads to a slight negative bias from measurements on opaque droplets when using the Otsu approach. The bias, if real, is still within the uncertainties of the measurements and therefore insignificant in most applications. Indeed, because the gravimetric and optical methods track well with each other across fluids with different optical properties, we have a strong indication that corrections for these optical differences are unneeded and perceived uncertainties linked to this source are unfounded. The only requirement to improve uncertainty is to shift the anchor point of the measurement traceability chain from a standard artifact to a gravimetric assessment of spherical droplet mass. We corrected the optical calibration of our system so that the automated *d*(50) approach (which is less sensitive to focusing effects) now gives optical values with uncertainties of about 3 % instead of 6 %, reflecting the lower baseline afforded by gravimetry over the artifact.

Given well-focused images of translucent and opaque droplets-in-flight having exactly the same dimensions (a very difficult scenerio to arrange!), our results suggest that there would be no significant difference between drop sizes measured by a consistent optical method. To gain perspective on the possible reasons for this outcome, we used ray diagrams to map the expected trajectories of the illumination source through and around the droplets. These maps are useful to explore the limitations of optical measurements from inkjet imaging systems that display gray-level profiles complicated by scattering, internal reflections and refraction of the illuminator rays. Opaque droplets, which absorb the internal light, are optically simpler than translucent droplets. The uncertainties introduced by these factors were modeled and empirically explored by comparing optical images of spheres of known diameter (Fig. 3 top) to analyze the 3-dimensional optical reconstruction of the effects (Fig. 3 bottom). Transitions at the drop edge corresponded to a 5 % variation in measured diameter, which is somewhat higher than the observed irreproducibility in the optical measurements when using any particular method to determine diameter at various positions around the droplet. These transitions, however, were reduced for opaque droplets, suggesting that the droplet boundries are better defined. This wasn't observed in the grayscale profiles between the translucent and opaque droplets, but does suggest that variation differences play a role in the subtle diameter differences observed between the *d*(x) and Otsu approaches.

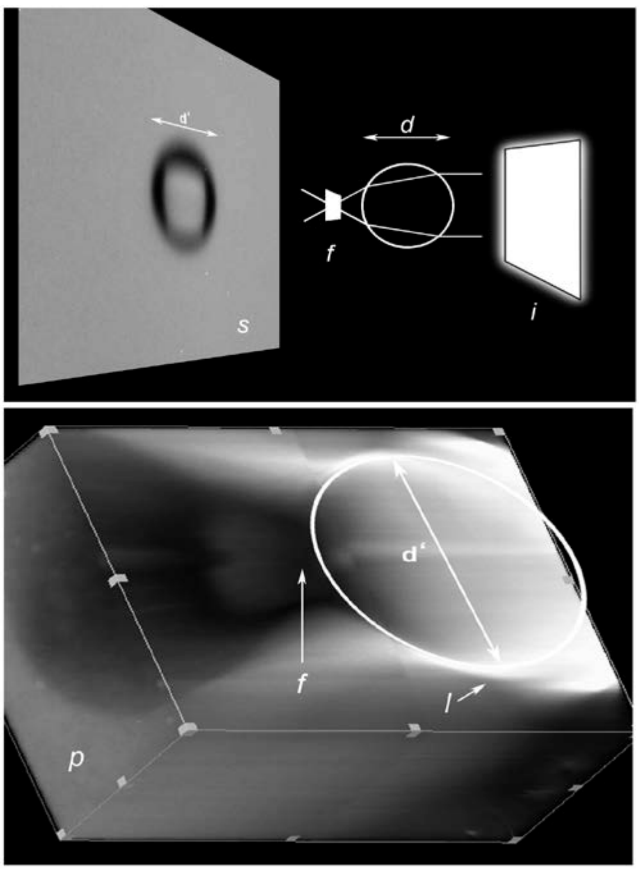

Figure 3: Top: schematic showing spherical droplet of true diameter (d), an illuminator (i), the image of the illuminator at the focal point of the sphere (f) and the resulting image (s) from which a measured diameter (d') is obtained. Bottom: 3-dimensional investigation of optical paths and effects around the perimeter of a sphere of known diameter (d) with diffraction effects (l) impacting measured diameter (d') and showing focal point (f) and appearance of sphere at a specific plane of focus (p).

4 CONCLUSIONS

Gravimetry provides one of the strongest traceability chains available in metrology, and our method of dimensional analysis now takes advantage of this improvement. Using fluids with differing optical properties, we determined accurately the diameters of inkjet-generated droplets-in-flight using gravimetry and compared these to those determined through optical methods. Because the biases observed between the gravimetric and optical diameters were consistent across the fluids tested, the degrees of fluid translucence and opacity does not appear to significantly influence optical sizing. This mechanism to improve uncertainty and control in dispensed droplet size may become vital to applications of microscale and nanoscale additive manufacturing where droplet size matters to the function of the deposit.

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