EPJ Web of Conferences **25**, 01097 (2012)
DOI: 10.1051/epjconf/20122501097

AN EXPERIMENTAL TECHNIQUE TO MEASURE THE CAPILLARY WAVES IN ELECTRIFIED MICROJETS

Emilio José VEGA[*], José María MONTANERO[*] and Noelia REBOLLO-MUÑOZ[*]

Abstract: *Backlight optical imaging is an experimental technique with an enormous potential in microfluidics to study very varied fluid configurations and phenomena. In this paper, we show the capability of this technique to precisely characterize the capillary waves growing in electrified microjets. For this purpose, images of electrified liquid jets formed by electrospray were acquired and processed using a sub-pixel resolution technique. Our results reflect the validity and usefulness of optical imaging for this type of application.*

1 INTRODUCTION

The controlled production of liquid microjets has very relevant technological applications in varied fields such as Biotechnology, Medicine or Pharmacy [1]. Microjets constitute the natural gateway for the continuous production of very small droplets at a sufficiently high rate. The jet morphology and the liquid physical properties determine the outcome (droplets, bubbles, capsules, fibers, ...) obtained from a specific technique. Electrospray is one of the most popular jet production technique because it allows one to form droplets with diameters ranging from hundreds of microns down to a few nanometers [2, 3]. Working in the so-called cone-jet mode [2, 3], a microjet tapers from the conical meniscus attached to an electrified nozzle. The applied electrical potential decays mainly within a small region next to the meniscus tip. Then, an electrically charged microjet flows downstream within a region were the axial component of the electrical field can be neglected. The electrical charges accumulate on the jet's free surface even for relatively low electrical conductivities because the electrical relaxation time is much smaller than the characteristic hydrodynamic time in the tapering meniscus [3].

The small disturbances naturally appearing in capillary jets can be decomposed into oscillation modes with different azimuthal wave numbers m. Since the seminal works of Plateau [4] and Rayleigh [5], the axisymmetric $m = 0$ mode has been believed responsible for most instability transitions and breakage processes in capillary jets, essentially because in this case the surface tension leads to a decrease of the jet's free surface (energy) while preserving its volume. The jets steadily produced by the electrospray technique in the cone-jet mode break up due to the Plateau-Rayleigh instability in many practical situations. For sufficiently large electrical potentials, however, the jet breakup is dominated by either axisymmetric or whipping electrostatic modes, and the Plateau-Rayleigh instability becomes irrelevant [6]. Capillary waves grow on the jet's surface pinching the liquid ligament somewhere downstream. The wavelength of the axisymmetric linear perturbations appearing at the initial stage of the instability determine to a great extent the size of the droplets. A variety of theoretical models have been proposed to predict the spatial and temporal growth of axisymmetric capillary waves in electrified jets with very different electrical conductivities K and permitivities ϵ (see, e.g., [7, 8] and references therein).

The experimental characterization of the small-amplitude perturbations appearing on a micrometer jet is a complex problem. These perturbations evolve according to the characteristic capillary time $t_0 = (\rho R^3/\sigma)^{1/2}$, where ρ, R, and σ stand for the liquid density, the surface tension, and the jet's radius, respectively. This characteristic time is of the order of 10 μs, and thus digital images must be acquired in the course of the experiment at extremely high frame rates and low exposure times. The first condition limits the spatial resolution (number of pixels) of the images, while the last requirement reduces enormously the illumination intensity. This last obstacle is even more pronounced in our applications because of the very small size of the imaged objects. The lack of illumination intensity leads to very low-contrast

[*] Department of Mechanical, Energetic and Material Engineering, Extremadura University, Avda. de Elvas s/n, 06006 Badajoz, SPAIN. E-mail: ejvega@unex.es

images, which increases the noise/signal ratio in the images, and hinders the precise detection of the jet contours. Non-standard image processing techniques are to be used in order to get reliable results, especially for small-amplitude perturbations. Super-resolution methods for free surface detection have been sucesfully applied to both equilibrium [9, 10] and dynamic [11, 12] liquid shapes by using backlight illumination.
In the present paper, we describe an experimental method to produce micrometer electrically charged jets (Sec. 2), and an image processing technique (Sec. 3) to precisely measure the free surface deformation at the initial stage of the Plateau-Rayleigh instability. This experimental procedure allows one to precisely characterize the small-amplitude perturbations growing on micrometer jets with spatial and temporal resolutions not reached so far. Some preliminary results are shown (Sec. 4) to illustrate the capabilities of this technique.

2 EXPERIMENTAL SETUP

The experimental setup is sketched in Fig. 1. The liquid was injected at a constant flow rate Q by a stepper motor through a capillary (A) of inner diameter D located in front of a plate with an orifice (B) of 350 μm in diameter. The plate covered the upper face of a cubic cell (C). We used a high-precision orientation system (D) and a translation stage (E) to ensure the correct alignment of these metallic elements, and to set the capillary-to-orifice distance H. A electric potential V was applied to the end of the feeding capillary by a DC high voltage power supplier (BERTAN 205B-10R) (F), while the cubic cell was used as ground electrode. A liquid meniscus was formed in the open air and stretched by the action of the electrical field. A microjet tapered from the meniscus tip and moved vertically towards the plate orifice. A prescribed negative gauge pressure was applied in the cubic cell by using a suction pump to produce an air stream coflowing with the jet. Both the liquid jet and the coaxial air stream crossed the plate orifice, which prevented the liquid accumulation on the metallic plate (it must be noted that the mechanical stresses associated with the gas stream were much smaller than the electrical ones, and thus one can neglect the dynamical effects due to the gas stream). In this way, the electrical charges were collected in the cubic cell. The electrical current I transported by the liquid jet was measured by using a Picoammeter (KEITHLEY model 6485) (G) connected to the cell.

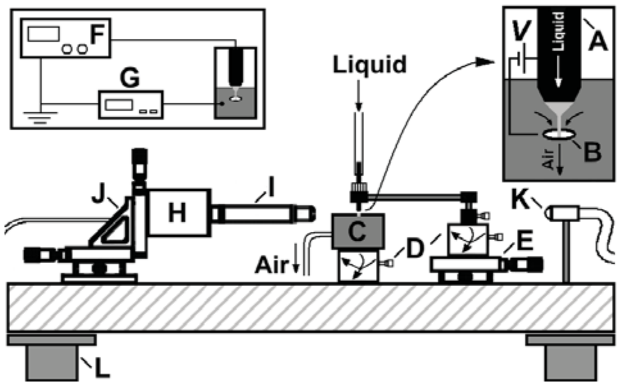

Figure 1: Experimental setup

Digital images of 128×160 pixels were acquired at 2.1$\times 10^5$ frames per second with an exposure time of 2 μs using a CMOS camera (Photron, FASTCAM SA5) (H) equipped with optical lens (a MITUTOYO 10$\times$ magnification zoom-objective and a NAVITAR 12$\times$ zoom) (I) providing a frame covering an area of about 70×88 μm. The magnification obtained was approximately 0.55 μm/pixel. The numerical aperture of the optical system was about 0.3. The camera could be displaced both horizontally and vertically using a triaxial translation stage (J) to focus the liquid jet. The fluid configuration was illuminated from the back side by cool white light provided by an optical fiber (K) connected to a continuous light source (STORZ xenon nova 300). To check that the fluid configuration was axisymmetric, we also acquired images of the liquid meniscus by using an auxiliary CCD camera (not shown in Fig. 1) with an optical axis perpendicular to that of the CMOS camera. All these elements were mounted on an optical table with a pneumatic anti-vibration isolation system (L) to damp the vibrations coming from the building.

Figure 2 shows the image of a 1-octanol ($\rho = 827$ kg/m^3, $\mu = 0.0081$ kg/ms, $\sigma = 0.027$ N/m) jet produced by the experimental setup described above with $Q = 4$ ml/h, $D = 200$ μm, $H = 1.72 \times 10^3$ μm, $V = 3.83$ kV and $I = 10.5$ nA.

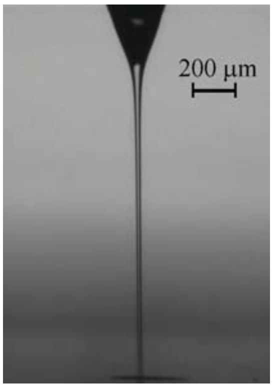

Figure 2: 1-octanol jet produced by the experimental setup described above. The image was acquired with the auxiliary CCD camera.

3 IMAGE PROCESSING TECHNIQUE

Here, we shall briefly describe the main aspects of the image processing technique. Details of the procedure can be found elsewhere [13]. The contours of the free surface in the image were detected using a two-stage procedure. In the first stage, a set of pixels probably corresponding to the contour being sought was extracted using Otsu's method [14]. The accuracy of Otsu's method is limited to the pixel size. In the second stage, the accuracy of the detected contours was improved to the sub-pixel level by analyzing the gray intensity profile along the direction normal to the contour. Fitting the sigmoid (Boltzmann) function [15] to the gray intensity values allowed us to obtain a continuous function in the transient region of the gray profile. The contour point was found by applying the local thresholding criterion. The number of resulting contour points was approximately equal to the number of pixels of the image along the vertical direction. Once the left x_l and right x_r contours had been detected, they were rotated to their vertical position (the rotation angle was less than 2° in all the cases analyzed), and the symmetry axis was found. The small non-axisymmetric perturbation of the free surface was partially removed by considering the axisymmetric contour $F = (x_r - x_l)/2$. Figure 3 shows the contour of a microjet detected by using the standard pixel-resolution method [14] (open symbols), and the sub-pixel resolution technique developed in our laboratory (filled symbols). The standard method fails to describe precisely the free surface deformation.

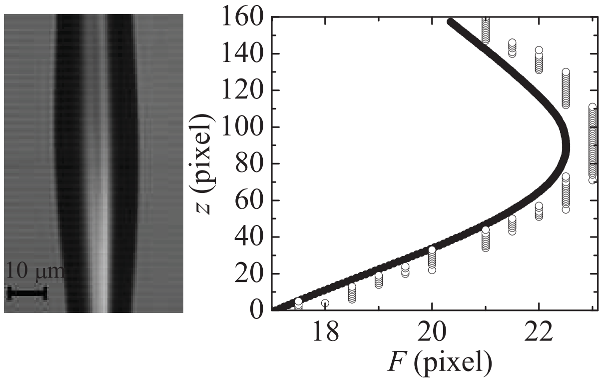

Figure 3: Contour of the microjet shown in the photograph detected by a standard pixel-resolution method (open symbols), and the sub-pixel resolution technique developed in our laboratory (filled symbols).

4 Results

In order to illustrate the capabilities of the experimental technique used in the present work, we show in Fig. 4 the results obtained at two instants for a microjets produced with $Q = 4$ ml/h, $D = 200$ μm, $H = 1.72 \times 10^3$ μm, $V = 3.83$ kV and $I = 10.5$ nA. In both cases the contour points are remarkably fitted by the a spatially growing wave $Ae^{i(kz+\omega t)}$, where A is the amplitude, $k = k_r + ik_i$ the complex wave number and ω the real frequency.

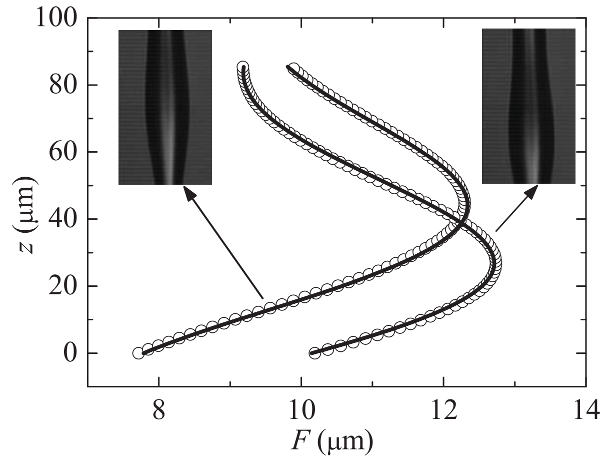

Figure 4: Contours of the microjet shown in the photographs (symbols). The solid lines correspond to the fit for $A = 3.06$ μm, $k = 0.0553 + i0.0104$ μm^{-1} and $\omega = -45.9$ μs^{-1}.

To summarize, we have described an experimental procedure to precisely measure the waves growing on electrified micrometer jets. The procedure provides accurate results which allows one to test, for instance, the dispersion relationships $k(\omega)$ calculated under different approximations. It must be noted that the surface charge density can be readily obtained in the experiments from the current intensity, flow rate, and jet radius values by assuming that the electrical charges are simply convected downstream within the region of interest. However and in order to simplify the analysis, it would be adequate to introduce artificially perturbations in the jet with a given frequency, and thus to isolate a single growing wave.

References

[1] O. A. Basaran, *Small-scale free surface flows with breakup: Drop formation and emerging applications*, AIChE J. **48**, 1842–1848, 2002.

[2] J. Fernández de la Mora, *The fluid dynamics of Taylor cones*, Annu. Rev. Fluid Mech. **39**, 217–243, 2007.

[3] A. M. Gañán-Calvo and J. M. Montanero, *Revision of capillary cone-jet physics: Electrospray and flow focusing*, Phys. Rev. E **79**, 066305, 2009.

[4] J. A. F. Plateau, *Statique expérimentale et théorique des liquides soumis aux seules forces moléculaires*, Gauthier-Villars, 1873.

[5] L. Rayleigh, *On the instability of jets*, Proc. Lond. Math. Soc. **10**, 4–13, 1879.

[6] Y. M. Shin, M. M. Hohman, M. P. Brenner, and G. C. Rutledge, *Experimental characterization of electrospinning: the electrically forced jet and instabilities*, Polymer **42**, 9955–9967, 2001.

[7] J. M. López-Herrera and A. M. Gañán-Calvo, *A note on charged capillary jet breakup of conducting liquids: experimental validation of a viscous one-dimensional model*, J. Fluid Mech. **501**, 303–326, 2004.

[8] F. Li, X.-Y. Yin, and X.-Z. Yin, *Instability of a viscous coflowing jet in a radial electric field*, J. Fluid Mech. **596**, 285–311, 2008.

[9] J. M. Montanero, E. J. Vega, and C. Ferrera, *Sub-micrometer precision of optical imaging to locate the free surface of a micrometer fluid shape*, J. Colloid Interface Sci. **339**, 271–274, 2009.

[10] E.J. Vega, J.M. Montanero, and C. Ferrera, *Exploring the precision of backlight optical imaging in microfluidics close to the diffraction limit*, Measurement **44**, 1300–1311, 2011.

[11] C. Ferrera, J. M. Montanero, A. Mialdun, V. M. Shevtsova, and M. G. Cabezas, *A new experimental technique for measuring the dynamical free surface deformation in liquid bridges due to thermal convection*, Meas. Sci. Technol. **19**, 015410, 2008.

[12] E. J. Vega, J. M. Montanero, and J. Fernández, *On the precision of optical imaging to study free surface dynamics at high frame rates*, Exp. Fluids **47**, 251–261, 2009.

[13] J. M. Montanero, C. Ferrera, and V. M. Shevtsova, *Experimental study of the free surface deformation due to thermal convection in liquid bridges*, Exp. Fluids **45**, 1087–1101, 2008.

[14] N. Otsu, *A threshold selection method from gray-level histograms*, IEEE Transactions on Systems, Man, and Cybernetics, 1979.

[15] B. Song and J. Springer, *Determination of interfacial tension from the profile of a pendant drop using computer-aided image processing: 2. Experimental*, J. Colloid Interface Sci. **184**, 77–91, 1996.