Stationary CBoat: Experimental Notes

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Details of the experimental protocols and measurement procedures for the Stationary camphoric acid boat experiments.

Two types of measurements were conducted:
1) The transient time-evolution of the radially spreading front $R(t)$ versus time t.
2) Measurement of radial velocity $u(r)$ versus radial distance r in the stationary state.

All experiments were conducted with camphoric acid in agarose gel tablets of 3 mm diameter and 1 mm thickness. Trouble with this method is the drift in velocity with time, especially for steady state measurements of $u(r)$. However, I cut down the total measurement time by automating the entire measurement and by simultaneously employing 2 LDV measurement probes.

I. PETRI DISH

I constructed a square petri dish by gluing glass plates (bottom plate was specifically chosen to be optically flat). Final dimensions of the dish were square side length $L = 25$ cm and height $H = 8$ cm.

Cleaning procedures for petri dish prior to each experimental run:
Step 1: Wash dish in acetone followed by methanol 3 times and dry in oven for 10 minutes at 100 °C.
Step 2: Soak dish in sulphochromic acid bath for 10 minutes.
Step 3: Thorough rinse with de-ionized water.
Step 4: Dry dish in oven for 30 minutes at 100 °C.
Step 5: Irradiate dish in plasma to remove any residual organic impurities.

All experiments were conducted with interface exposed to room environment to maintain unsaturated solvent vapor conditions at air-water interface. To avoid surface contamination by airborne dust and particulate matter, all cleaning procedures and experiments were performed in a small clean room with portable particulate air filters maintained at 25 ± 1°C.

II. TRANSIENT MEASUREMENTS OF $R(t)$

Transient measurements of initial spreading were performed with 50 μm borosilicate hollow glass spheres (specific gravity 0.25) as our visualization medium.

Cleaning procedures for the glass particles:
Step 1: Wash particles in methanol followed by acetone 3 times and dry in oven for 10 minutes at 100 °C.
Step 2: Thoroughly rinse particles in de-ionized water and dry again in oven for 30 minutes at 100 °C.
Step 3: Before each experiment, weigh out 0.1 g of particles, irradiate with plasma to break down residual organic impurities.

A. Setup

The square petri dish was placed atop an LED illumination source. The petri dish surface was closed with a clean glass plate acting as a lid and de-ionized water was introduced in the petri dish by a pump upto depth $D = 5$ cm to ensure all experiments conformed to the deep layer limit. The setup was left for 15 minutes to allow flow circulation to subside. The light source was turned on and the glass plate was removed from top of the dish just prior to experiments. The camphoric acid tablet in methanol saturated solution was washed in de-ionized water to remove methanol and precipitate camphoric acid. The tablet was then glued to a rigid needle connected to a motorized translation stage as shown in Sathish's figure (copied in fig. 1 below).

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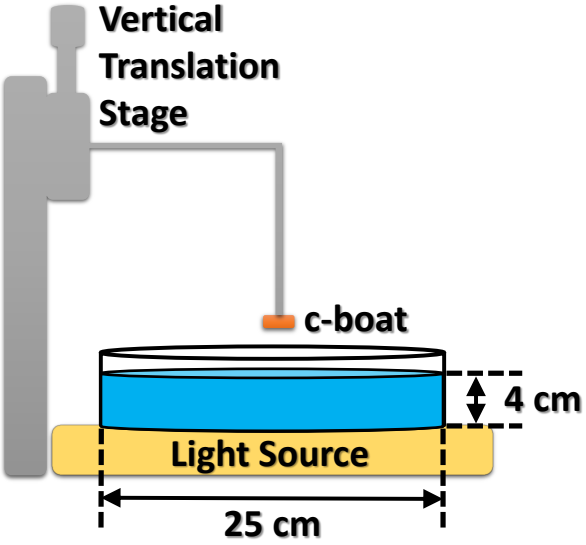

FIG. 1: Sathish's sketch of the setup for transient measurements. Only difference is I used a square petri dish (25 cm side length and height 8 cm).

A high speed camera (Phantom v641, Vision Research Inc.) was mounted vertically above the setup and images were collected with 1152×1152 pixel resolution at 3000 frames per second, thus setting the temporal resolution at $\Delta t = 1/1000 = 0.001$ s for the transient measurements.

B. Experimental Procedure

Following the 15 minute wait for the pumped de-ionized water to stop circulation in the petri dish, the experiment was conducted in the following stages:
Step 1: Position tablet 500 μm above interface using a motorized translation stage.
Step 2: Take 10 successive snapshots of bare setup to use later as background images for image processing.
Step 3: Sprinkle 0.1 g of hollow glass sphere tracer particles with fine mesh sieve onto air-water interface.
Step 4: Set high speed camera to constantly collect images at 1000 frames per second.
Step 5: Move motorized translation stage to bring tablet in contact with surface. Collect data and hit trigger.

C. Image Processing

The images were processed with the following protocol:
1) Due to the backlit illumination mode, the particles show up dark in a bright background.
2) The 10 background images collected prior to experiment were averaged to obtain a single average background intensity image.
3) Average background subtracted from all experimental images to reveal the negative where particles show up as grayscale specks on a dark background. This removes all spatial illumination inhomogeneity.
5) For each image, taking tablet center as origin, obtain azimuthally averaged intensity as function of radial distance $I(r)$.
6) In a plot of $I(r)$ vs. r for each instant, the radially spreading particulate front shows up as a bump. Radial position of spreading front $R(t)$ was calculated from this azimuthally smoothed bump for each time instant.
7) Plotting $R(t)$ vs. t in log-log scale gives exponent for transient spreading front. I subtracted 1.5 mm from Radial distance R(t) to re-position R = 0 at edge of camphor boat.
8) Exponent was calculated from log-derivative.

III. STATIONARY MEASUREMENTS $u(r)$

Measurements for the stationary state radial velocity $u(r)$ were performed using Laser Doppler Velocimetry (LDV). I used a Laser Doppler Velocimeter from TSI Inc. (LDV 9253-120 fiberoptic probe, Colorlink 9230 and IFA 655 Digital Burst Correlator) coupled to a Spectraphysics Laser (wavelength 488 nm, 35 mW continuous output). The LDV's Bragg Cell splits the incoming beam to give the original and a second frequency shifted beam (shift frequency $\Delta f = 40$ MHz).

For LDV seed particles, I used 1 μm polystyrene particles (Bangs Laboratories Catalog No.: PS04N, mean diameter 1.04 μm, 10% solid fraction of particles suspended in water). I suspended 10 μl of particle suspension per 100 ml of camphoric acid saturated methanol solution for the measurements.

A. Experimental Setup

The LDV method does not require high speed imaging and backlit LED illumination. The LDV fiberoptic probes were vertically aligned above petri dish such that the two beams exiting a probe traveled through air and intersected at the interface. The beams were aligned such that fringes resulting from the beating of the two frequencies were aligned normal to the radial direction so that colloidal particles traveling along the radial direction would cut the fringes when transiting the beam spot. The beam spot had a diameter of 124 μm. Each LDV probe was mounted on a motorized translation stage (Newport Corp. Model XMS160 with 160 mm travel, load capacity of 100 N with on-axis accuracy of 1.5 μm). Both translation stages were independently controlled via LabView. The LabView interface was programmed to move the stage 200 μm distance at 300 μm/s speed and collect measurement data for radial velocity $u(r)$ for 1 second. In other words the radial velocity $u(r)$ was measured in 200 micrometer steps of radial distance from the center. The photomultiplier tubes in the LDV fiberoptic probe registered on average $N = 30,000$ particle counts per second. A temporal average over the 1 second of data provided the value of $u(r)$ for each radial location. Since particles arrive at random, assuming Poisson statistics, we estimate measurement error for each value of temporally averaged (over 1 second) radial velocity $u(r)$ is $\delta u(r)/u(r) = 1/\sqrt{N} = 1/\sqrt{30000} \simeq 0.006$ or 0.6%. Both LDV probes were manually moved to c-boat perimeter. From there the 2nd probe was moved a radial distance of 2.02 cm. Under steady-state conditions i.e. 2 minutes after introduction of camphoric acid boat at interface, the probes started collecting data. Probe 1 collected data from $r = 0$ to $r = 2$cm in 200 micron steps. Probe 2 collected data from $r = 2.02$ cm to the edge in 200 micron steps. Each measurement of $u(r)$ is obtained by temporally averaging all particle counts over a 1 second duration. The LDV registered upto 70000 particle counts per second close to $r = 0$ cm, but this fell off with increasing radial distance since the particles travel in a radially diverging geometry. The error I take is the worst case at maximum radial distance where the LDV registered about 30000 particle counts per second. Assuming random arrival of particles, this gives me an error $\delta u(r)/u(r) \sim 1/\sqrt{30000} = 0.0057$ or 0.57%.