AQUEOUS FOAM EXPERIMENTS IN THE MAXUS 6 ROCKET: TOWARDS THE DEVELOPMENT OF AN ISS MODULE

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ABSTRACT

We report results of aqueous foam experiments performed in the Maxus 6 sounding rocket, dealing both with technological and scientific issues. On the technological side, the performed tests concern the methods for controlled foam generation and destruction. On the scientific side, we present the results of an imbibition experiment, in which some liquid is injected into the foam and its propagation followed by electrical conductimetry. Comparisons with numerical simulations are made, evidencing a good agreement.

1. BACKGROUND AND MOTIVATION

Aqueous foams are dispersions of a gas into a liquid, stabilized by the presence of surfactant molecules adsorbed at the air-liquid interface [1]. Aqueous foams are widely used in our everyday life: in detergency, cosmetics, food, etc... They are also present in many industrial applications (flotation, petroleum, paper). They are also very interesting for the development of low-weight materials, potentially useful for space applications. Despite the use of surfactant molecules, aqueous foams irreversibly evolve on Earth, mainly because of gravity: the liquid drains downward out of a foam, as gas and liquid tend to separate. Thus, the amount of liquid inside a foam (its liquid fraction, \( \varepsilon = V_{\text{liquid}}/V_{\text{foam}} \)) cannot be kept constant on long time scales, and always decreases. This effect, called drainage, has been widely studied on ground, as it is a crucial effect regarding the foam lifetime [1-4]. It also appears that many foam properties (the optical, electrical or mechanical ones, for instance) strongly depend on the liquid fraction, so that understanding the properties of foams in the widest range of liquid fractions is one of the main issue of the physics and chemistry of foams. However, due to drainage, it is almost impossible to study wet foams at constant liquid fraction on ground.

In fact, only microgravity offers the opportunity to study such wet foams. Starting a few years ago and under the supervision of ESA, the FOAM module for the ISS is under development. It will be dedicated to studying the rheology (mechanical properties), stability, coarsening and structure of wet aqueous foams. The development of an ISS facility is quite complex: to help us to achieve that goal, we can use other means to access microgravity conditions to test crucial parts and apparatus, and to reduce the risk for the final ISS module. In that spirit, we performed experiments in the 34th, 35th and 37th ESA parabolic flight campaigns, with the help of EADS-ST in Friedrichshafen [5]. We have also performed experiments in the Maxus 6 sounding rocket, launched in Kiruna, on Nov. 22nd 2004. With this rocket, one can have access to about 12 minutes of extremely good low gravity conditions. The experiments can be followed from the ground, and commands can be sent during the flight. Our sub-module, developed by EADS-ST (Friedrichshafen), was installed within the “FOAM 2” module developed by the Swedish Space Corporation (SSC), where it shares the same baseplate with 2 other foam experiments, more dedicated to study the stability of foams. A complete description of this FOAM 2 module can be found in a companion paper from the SSC engineers [6]. For our setup, and within the ISS project, the goals for this Maxus rocket were both technical (foam handling, see section 2) and scientific (performing an imbibition experiment, see section 3).

2. TECHNICAL ISSUES AND RESULTS

Many complex technical issues are associated with the development of the ISS FOAM module. A major one concerns the “handling”, or the management, of the foam samples. First of all, we have to produce the samples in-situ. Due to the measurements foreseen (especially rheometry), strong requirements on the foam parameters are also given. The bubble size has to be well controlled (low polydispersity, and a mean value of 100\( \mu \)m) and foams of liquid fraction ranging from 3% to 40% have to be produced. In addition, the foam homogeneity and uniformity has to be high, meaning no holes or voids inside the samples. Beside the requirements for the sample, one also has to consider the typical cell geometry required for performing the best rheological measurements: the foam sample is constricted between a base plate and a

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top cone (Fig. 1), in a discoid shape cell. For rheology purposes, the cone must eventually rotate in order to apply a controlled shear on the foam. At this stage of development, we have kept the cone fixed, so that it makes a single piece with the base plate. Finally, as it is foreseen to study dozens of different foams (with cleaning sequences in between), the final automatic device will have to be very robust, minimizing the chances of failure with time, and also minimizing the amount of solution needed (with as much liquid recovery as possible).

From both the results of previous parabolic flights [5] and following an original idea from D. Durian at UCLA, a method based on a closed loop circulation was developed. The basic idea is to create a closed circuit, including the cone-plate cell, in which the gas and the liquid are driven by a pump: both fluids get mixed along the circulation (in the pump, the valves and tubes), and eventually a uniform foam is produced. Controlling the initial amount of liquid and gas gives the final foam liquid fraction. In our setup, the foam enters the cell from the bottom, at its center (Fig. 1). It is collected out of the cell on its edges by eight holes, connected to tubes bringing back the foam below the cell to the center port (two of these holes/tubes can be seen on Fig. 1). During the running of the loop circulation, a major concern is to ensure that there is always some foam flowing in all the volume of the cell, without leaving any foam pieces out of the circulation, or empty parts of the cell. To avoid such problems, the cell is rapidly rotating on its vertical axis during the production process. The tubes which collect the foam out of the cell are also rotating with it: they are thus connected one after the other to the fixed port at the center (at any time, there is always one tube bringing back some foam inside the loop). This rotation method first provides that the injected foam spreads very uniformly in all directions; secondly, the foam is sucked out in the same way from all the eight holes on the edges. More technical details can be found in [6].

Once a foam has been made and studied, it is in principle quite simple to make a new sample from this previous one with a different liquid fraction, by adding the right amount of solution or gas inside the loop and running it to homogenize the foam. We can also empty the cell by strong centrifugation, replacing the foam inside the circuit by only gas (and collecting the old foam into a reservoir).

To follow the global behavior inside the cell, we observe it from below with a CCD camera (the cell is illuminated from the other side). Only a bit less than ¼ of the cell is optically accessible, though its rotation allows us to visualize the whole cell. The amount of light transmitted is also in principle a way to quantify the liquid fraction, and to evidence radial gradients [4,5]. The foam electrical conductivity, which provides the liquid fraction [1,4], is also measured along a cell radius by 6 pairs of electrodes.

![Fig. 2. Photograph of the “cone-plate” sub-module (also called “drainage” cell in [6]).](image)

Figure 2 is a photograph of our sub-module cell: the cell is on the bottom right with a diameter of 10 cm (and a cone angle of 0.2 rad); one can see some tubing from the closed loop, the main motor for the cell rotation (on the left, linked to the cell axis by a rubber band) and pieces of the slip-ring on the top of the cell (for the conductivity measurements). This sub-module was then integrated inside the global FOAM 2 module [6], with the camera, VCRs, electronics, gas and fluid injectors and suppliers. In these experiments, we used an aqueous solution containing the surfactant Sodium Dodecyl Sulfate (SDS) and the co-surfactant dodecanol (DOH) to make very stable foams. Such a mixture provides rather rigid and immobile bubble interfacial layers [4]. The gas used was nitrogen.

The whole sub-module was thoroughly tested and validated on ground: starting from an empty cell, the foam production takes roughly 2 minutes of closed loop circulation. In microgravity, the same time was given in the timeline for the foam production. In figure 3, four pictures of the visible part of the cell are given during these 2 first minutes of microgravity. The cell is seen from below, and its center is on the top middle of each photograph (the black vertical lines corresponds to the tubes coming from the edges back to the center, so they indicate the cell radius). At short time (top left) the cell is almost empty, and a maximum of light is transmitted. As the mixing and circulation occur, more and more foam can be seen inside the cell (it appears grey, as the light transmission decreases). At the end of
the process, the whole cell is filled with an homogeneous and uniform foam, with small bubble sizes of typically 150-200μm.

![Fig.3. Four views of the cell during foam formation.](image)

An important result here is that the foam production and cell filling appear to be completely similar to what was found during the ground tests. After the imbibition experiment (see next section) the foam sample was no longer uniform (with radial liquid gradient): we have found during the flight that running again the loop circulation has successfully re-created a new uniform and wetter sample. Regarding the cell emptying, one can see in figure 4 that the process is also very efficient as all the foam is removed within 2 minutes of centrifugation and gas injection (as on ground).

![Fig.4. Four views of the cell during foam removal.](image)

Moreover, during all these experiment phases, the monitoring of temperatures and pressures showed that all the parameters were under control, without any unexpected behavior.

So these results show that we have successfully and automatically produced a foam (within the required conditions), correctly filled a cell of complicated geometry, re-homogenized it after some first experiments so that it can be re-used for new measurements, and followed all the modifications by direct visualization, finally removing the foam from the cell.

### 3. IMBIBITION EXPERIMENTS

As already said, on Earth liquid drains out of a foam, and accumulates below it: due to gravity, the liquid flows down, and this flow takes place inside a network of liquid channels between the bubbles, called the Plateau borders (PB) [1]. These PB have specific triangular-like section (shape of the interstice between three adjacent bubbles), and are connected fours at "nodes". Capillarity also plays a role in liquid propagation inside aqueous foams: differences in the PB pressure between wet and dry foam induce a liquid displacement from the wet foam part to the dry one, and tend to remove liquid fraction gradients. Getting rid of gravity is a way to study only these capillary effects. In the Maxus rocket, we performed an imbibition experiment: some liquid is injected at a controlled continuous rate inside an initially dry foam. One can then follow how the liquid spreads inside the foam by capillarity, which is a way to "scan" the structure of the foam, test the drainage equations in new conditions, validate some timescales and hypothesis made in the models [7], and to possibly find out unexpected behavior; all this issues are very important for the future ISS module. Related experiments on capillary imbibition have already been reported, but first they only dealt with 2D foams, and secondly there is no controlled liquid injection (but only the simple situation of a foam in contact with a liquid reservoir) [8-9]. For 3D foams, we also performed imbibition experiments on parabolic flights, but on shorter timescales, and not in that same cone-plate geometry [5].

In the Maxus experiment, the injection is at a fixed flow rate Q= 2.5 ml/min, during 150s, and we follow the liquid distribution in the foam by the electrical conductivities (calibrations were done between the foam conductivity $C_{foam}$, solution conductivity $C_{liquid}$, and the liquid fractions). In Figure 5, we have plotted the raw data of the conductivity as function of the flight time for the 6 pairs of electrodes (the electrode 1 is close to the center at a radius $r = 7mm$, and electrode 6 is close to the edge, at $r = 42mm$). At time A, the liquid injection starts, it is stopped at time B, and the closed loop is re-activated at time C to homogenize the foam. On this graph, one can easily see the spreading of liquid inside the foam: the pairs of electrodes detect the increases of liquid fraction, one after the other.
Also, one can see that the signals on the first three electrodes (closest to the center of the cell) abruptly reach a very high and constant value, after a first initial slower increase.

![Graph](image1)

Fig.5. Conductivity vs time, on the six pairs of electrodes.

Indeed, this abrupt rise corresponds to the transition from a measurement inside a foam to a measurement inside the pure liquid state (note however that the transition is not as discontinuous and sharp as the foam-liquid interface is, since the measurement is an average over some radial distance). Optically, we verified that, at these electrodes, only pure solution is actually found, and no longer a foam. As the liquid is injected, the foam becomes so wet that the liquid fraction goes over the critical liquid fraction corresponding to the foam-liquid interface, $\varepsilon = 36\%$.

This is a direct effect of the experiment geometry: close to the injection point, the radius and the thickness are small so that the flow rate per unit of foam surface is always extremely high, providing very a high local wetness, and a pure liquid pool (even at low flow rates). After the end of the injection, the conductivities close to the center decrease back: the liquid pool in the center reduces its size, and eventually vanishes, as all the liquid is dispersed into the foam.

Now we can compare these results to numerical simulations. Drainage equations describing the time and position evolution of the liquid fraction have been derived, and widely discussed for the case with gravity [1-4]. In the case of microgravity, simulations corresponding to different experimental situations have been recently presented in [7]. Following that work, we present here the simulations corresponding best to the Maxus case: it takes into account the initial liquid fraction of 5%, the exact flow rate, the bubble size, the 3D cone-plate geometry, and a physical cutoff at 36% (foam-solution interface). For the bubble size, a mean diameter of $D = 300\mu m$ was chosen: due to the strong coarsening with nitrogen, the bubble size increases significantly within the first minutes. In that spirit, during the maxus experiment, we have let some time for the foam coarsening and stresses relaxation after the end of the foam production, before the beginning of the imbibition experiment. During the imbibition, the bubble size was then no longer varying much and $D = 300\mu m$ was a mean value.

In order to compare our data, we also have to restrict them to the liquid fractions corresponding to the foam ($\varepsilon < 36\%$). From calibration tests, we know that the conductivity of a foam at $36\%$ is $\frac{4}{3}$ of the conductivity of the pure solution ($C_{\text{foam}}(\varepsilon=0.36) / C_{\text{liquid}} = 0.25$). So, the figures 6a and 6b show the experimental data and simulations, focusing only below this limit of $36\%$ (just before the sharp variation in the experimental curves). Note that the time has been rescaled so that $t=0$ at the beginning of the injection, and that these time evolutions are given in both parts of figure 6 for the same 6 radial positions.

![Graphs](image2)

Fig.6. Time evolution at 6 different radii: (a) experimental results, and (b) simulations.

It is clear that the two graphs have many features in common, and that the agreement between experiments and simulations is fairly good. Qualitatively, during the injection, one recovers the different rises of the signals, delayed in time, and with a smaller and smaller rate of increase (initial slope) as the radius increases. We will return to the quantitative features in the following. For $t > 150s$, after the injection has been stopped, the qualitative features of experiments and simulations are again similar, although there are some more differences.
than for \( t < 150s \). It turns out that they are connected to the size and shape of the pure liquid pool at low radii. Here visual observation is also important for the understanding: it appears that the liquid pool does not remain symmetric around the injection port, but is slightly shifted out of the center, and in this Maxus experiment it is unexpectedly displaced along the direction where the electrodes are. So, along that radius, the size of the liquid pool appears bigger than it should be if it had remained centered and symmetric. This explains for instance that experimentally the fourth pair of electrodes is finally measuring in the pure liquid, in disagreement with the simulations. This asymmetry of the liquid pool could also be linked to the differences seen in the way the liquid pool retracts and vanishes. In any case, for \( t > 150s \), it appears that the dynamics of the homogenization of the liquid fraction seems to be slower in the experiments than in the simulations (where the liquid pool seem to vanish extremely quickly).

Fig. 7. liquid profiles at different times:
(a) experimental results, and (b) simulations. The arrow indicates the time direction.

Another way to present these data is to look at the profiles along a radius, at different times (Figure 7a and b). For clarity, only data and simulations for time up to 150s (during the injection) are reported. Once again it is quite clear that the simulations capture most of the features of the experiments. One can also recover here that, in the maxus experiment, the liquid-foam interface propagates to a greater radius than what is predicted in the case of a symmetric liquid pool. One can only speculate on the shift of the liquid pool out of the center, and a possible explanation is related to the wetting properties of the cone and plate surfaces, and to the contact angle on these surfaces. Beside these qualitative comparisons, one can quantitatively compare the experimental and numerical results on some specific features. At any time, the liquid profile in the foam decreases down to the initial liquid fraction (5%) with the cell radius. The point where it reaches that initial liquid fraction can be considered as the position of the fastest liquid propagation, or the position of the liquid "front". From Fig. 6a, it is then possible to extract this front position as a function of time (practically, it is done by detecting the time at which some liquid is detected at a given position).

Fig. 8. front position: experiments (squares), and simulation (dashed line).

In Fig. 8, we have plotted this front position deduced from the experiments and simulations. In simulations, considering that our conductivity setup is quite accurate, we have chosen to report the position corresponding to \( \epsilon = 5.1\% \), slightly above the initial foam liquid fraction at 5%. It turns out that the agreement is good: the simulations reproduce rather well how fast the liquid spreads into the foam. Adjusting slightly the bubble size or the foam permeability (which depends on the surface mobility [4]) can provide a perfect agreement, removing the small discrepancy. Note, in that sense, that despite that small discrepancy, one finds a similar power law behavior for the experiments and simulations, with an equal exponent of 3/7 (solid line through the experimental points). An exponent 1/2 would have been found in the situation of constant cell thickness. Finally, it is interesting to note how fast the liquid is actually spreading into the foam: in about a minute some liquid has already reached the cell edge.

Lastly, we show in Fig. 9 how the initial rates of liquid fraction increase depend on the cell radius \( r \), and make
the comparison between experiments and simulations. For simplification, at each radial position and following the detection of liquid at that position, we have considered the observed conductance and liquid fraction variations as being linear. This is valid here as we only have the variations over a small range of time (at long times, a power law behavior is expected [7]). Extracting these linear slopes $\alpha$ from figure 6a and 6b, and normalizing them by the value $\alpha_1$ found at the first electrode allows us to compare them in Fig. 9. The agreement between experiments and simulations is here quite good, showing how strongly the slopes change with the radius, and how well the simulations reproduce the data during the liquid injection ($t < 150s$).

![Graph showing slopes $\alpha$ normalized by the slope $\alpha_1$ at electrode 1.](image)

Moreover, the variation of $\alpha/\alpha_1$ is approximatively like $1/r^2$. This is understandable knowing that first, the foam surface $S$ through which the liquid is spreading goes with $r^2$, so that the flow rate per unit surface $Q_0 = Q/S$ is in $1/r^2$, and secondly, the liquid fraction at given time and position goes like $Q_0^b$, with $b = 0.8$ or 1, depending on the surface properties.

4. CONCLUSIONS

Within the FOAM 2 module in the Maxus 6 rocket, the tests and experiments on our sub-module have been successful and very instructive. For technological issues, we have clearly validated some foam handling methods. Still, the device can be optimised, but the main concepts appear completely reliable, and can be used for the design of the future ISS facility.

For the imbibition experiment, we have been able to follow the evolution of the liquid inside the foam during and after some injection. The comparison with the simulations show that we are able to capture and explain almost all the features of the liquid propagation. This means that the equations and hypothesis are quite robust and valid in these microgravity conditions, and this gives us some important insights regarding the choice of experiments to be performed in the ISS module.

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6 REFERENCES


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