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Bubbles and liquid films resisting drainage, evaporation and nuclei-induced bursting.

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Soap bubbles are by essence fragile and ephemeral. Depending on their composition and environment, bubble bursting can be triggered by gravity-induced drainage and/or the evaporation of the liquid and/or the presence of nuclei. In this paper, we design bubbles made of a composite liquid shell able to neutralize all these effects and keep their integrity in a standard atmosphere. This composite material is obtained in a simple way by replacing surfactants by partially-wetting micro-particles and water by a water/glycerol mixture. A nonlinear model able to predict the evolution of these composite bubbles toward an equilibrium state is proposed and quantitatively compared to experimental data. This work unveils a composite liquid film with unique robustness, which can easily be manufactured to design complex objects.

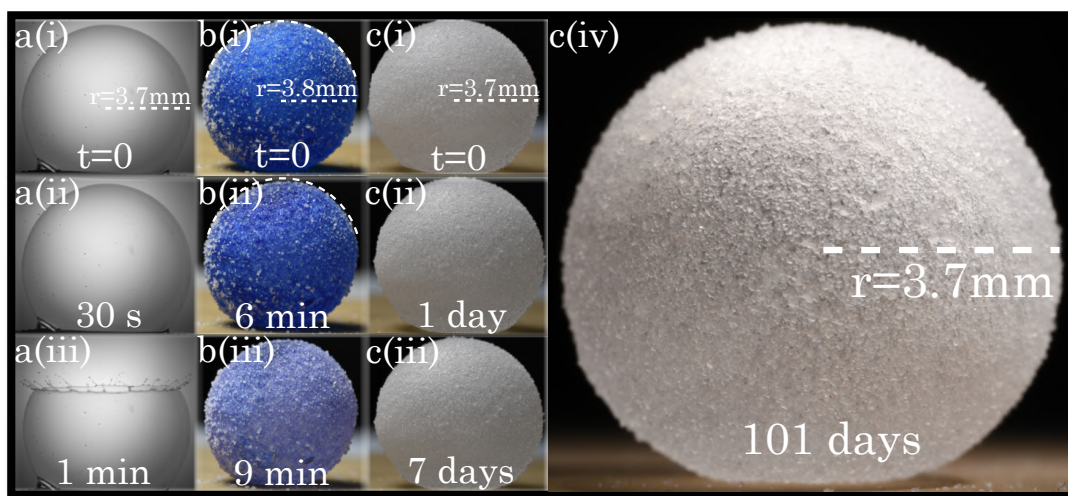


FIG. 1. Comparison of the lifetimes of three types of bubbles: a) Soap bubble bursting after 1 min. b) Water gas marble covered by partially-wetting micro-particles, whose shell rupture is observed after 6 min and complete drying after 9 min. Blue dye is added to visualize liquid drying. c) Water/glycerol gas marble, whose integrity remains unaffected after at least 101 days. As can be seen on Fig.1 in SI, the shell of a water gas marble once dried collapses like a sand pile (see Movie M1), while the shell of the water/glycerol gas marble is still liquid and spherical after 101 days and reacts as a liquid film when punctured (see Movie M2).

I. INTRODUCTION

Under a standard atmosphere, air bubbles typically burst in minutes (see Fig.1a) due to the effect of gravity-induced drainage and/or liquid evaporation and/or nuclei induced inception depending on their composition. "Bare" viscous bubbles bursting follows gravity-induced drainage of the liquid, resulting in shell thinning down to a limit thickness of the order of tens of nanometers, wherein spontaneous breaking occurs⁹. The sliding condition of the liquid on air leads to plug flow in the shell and hence characteristic bursting time $\tau = \mu/\rho_l g R$, which does not rely on the liquid shell thickness e but rather on the radius of curvature of the bubble R , with ρ_l the liquid density, g the gravitational acceleration and μ the liquid dynamic viscosity. The addition of

surfactants to produce soap bubbles can lead to completely different pictures depending on the concentration of surfactants^{17,19}. At intermediate surfactant concentrations, bubble ageing results from a complex interplay between gravity and capillary induced drainage, Marangoni stresses due to gradients of surfactant concentration and bubble evaporation. This picture can be even more complex for bubbles at the surface of a liquid bath¹⁴. In this case, localized film thinning occurs close to the surface of the bath (so-called "marginal pinching"²), whose instability results in some convective plumes rising on the cap referred to as "marginal regeneration"¹⁷. At large surfactant concentration, the surfactant rigidifies the film surface leading to no-slip boundary condition, Poiseuille flow and hence considerably slowed down drainage. In addition, surfactants stabilize thin films so that bubbles

lifetime is drastically increased. But eventually, the evaporation and/or the presence of nuclei will end up the life of these bubbles. Even the most skilled scientist or bubble artist meticulously adjusting the liquid/surfactant composition is not able to design soap bubbles keeping

their integrity for more than a couple of hours under a standard atmosphere. Long bubbles lifetimes are only reported in carefully controlled atmosphere¹², wherein dusts, the "archenemy of bubbles", are suppressed.

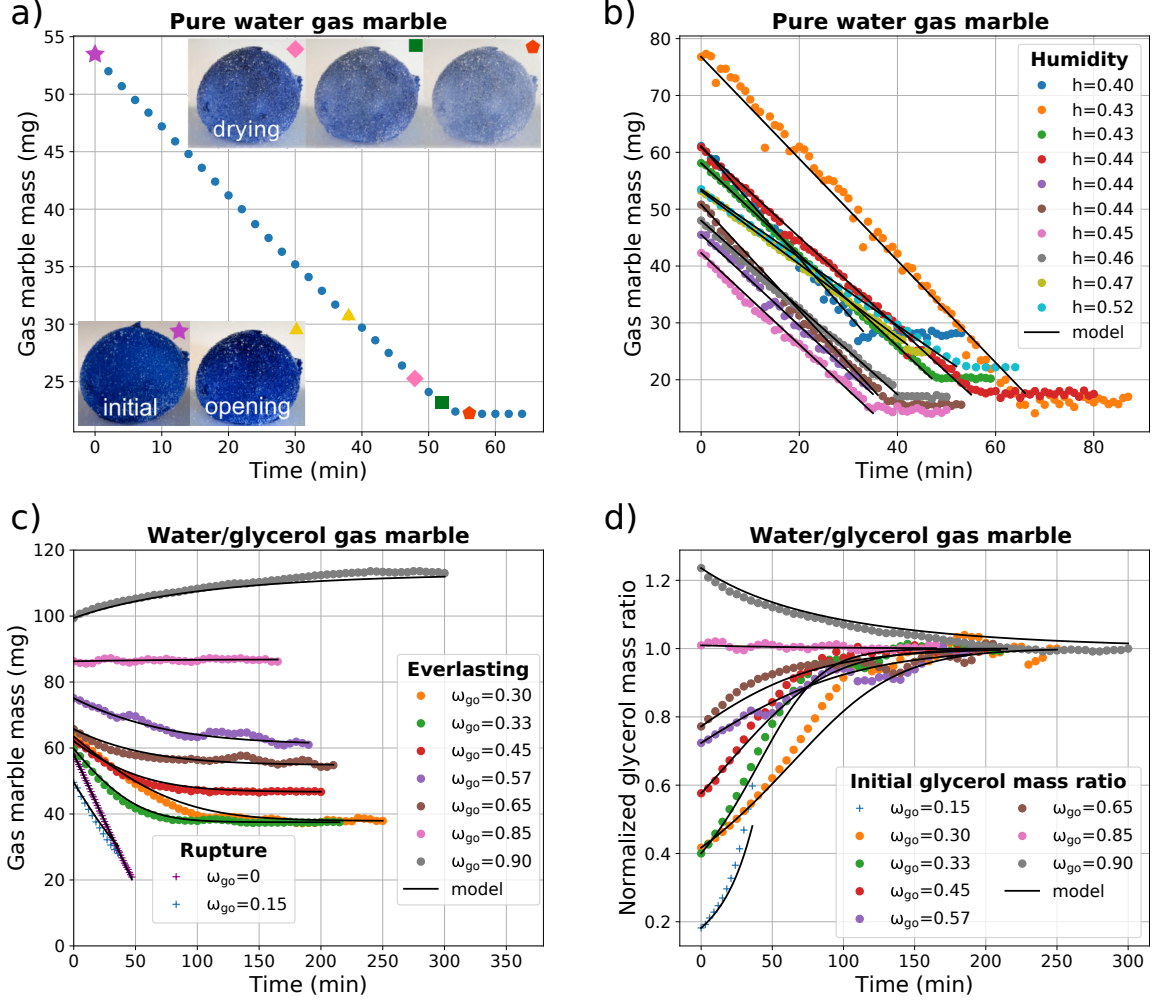


FIG. 2. Evolution of gas marbles of radius 5.1 ± 0.1 mm under a standard atmosphere (temperature $T = 21.0 \pm 0.5^\circ\text{C}$). a) Evolution of the mass of a single water gas marble as a function of time at ambient humidity $h = 0.52 \pm 0.01$. A blue dye is used to visualise drying, reflected by the change in the bubble color. The bubble opening can be visualized by the brutal change in the bubble shape (see also Movie M4). It is marked with a yellow triangle on the curve. The plateau region (after 54 min) occurs when the water is entirely evaporated. b) Evolution of the mass of pure water gas marbles as a function of time at different relative humidity conditions. c) Evolution of the mass of water/glycerol gas marbles as a function of time for different initial glycerol mass ratio ω_{go} . Low glycerol content leads to the rupture of the gas marble, while higher glycerol mass ratio leads to everlasting gas marbles. d) Normalized glycerol mass ratio (ω_g/ω_{geq}) content of gas marble as a function of time. The experimental data are the same as in Fig. 2c but with a different representation. It shows that the glycerol mass content tends toward a constant value, which depends on the ambient air humidity. Experimental data of Fig. 2b, 2c and 2d are fitted with the evaporation/absorption model introduced in section IV (black continuous line).

Adding partially-wetting particles to liquid/air interfaces⁶ has been investigated as a mean to strengthen their resistance to mechanical stresses providing an interfacial material with properties at the crossroad between solids and liquids²³. When drops and bubbles are covered with such a composite interface, some extraor-

dinary properties have been reported such as: non-stick droplets (so-called "liquid marbles")^{4,8,15} able to roll on solid surfaces, "armoured bubbles"¹³ in a liquid stable to dissolution¹ and which can sustain metastable¹⁸ non-spherical shapes^{20,24} or finally "gas marbles" (air bubbles)^{21,22} supporting positive and negative relative

pressure one order of magnitude larger than the Laplace pressure. The addition of particles to an interface can also lead to some counter-intuitive behaviours such as surface energy driven fingering instability in reversed Saffman-Taylor configuration⁵ or films growing ahead of a liquid meniscus moving in a capillary tube²⁴.

Nevertheless, (i) the effect of such a composite interface on the lifetime of air bubbles has not been investigated and (ii) the single addition of particles to a bubble shell cannot prevent its evaporation (Fig.1b) and hence its bursting. In this paper, we show that covering a bubble water shell with micro-particles inhibit gravity-induced drainage and that further adding glycerol leads to a stable state, wherein the evaporation of water is counterbalanced by the hygroscopicity of glycerol, which absorbs water molecules contained in ambient air. This results in bubbles with lifetimes of at least several months in a standard atmosphere since these bubbles are still intact in our laboratory 101 days after their synthesis (Fig.1c). This behaviour is rationalized by a nonlinear model able to reproduce quantitatively the evolution of the mass of the bubble. In addition, we show that this composite material can be used to tailor liquid films of various shapes, which also keep their integrity.

II. NEUTRALIZING DRAINAGE

"Gas marbles" are by definition gas bubbles whose composite shell is made of liquid and partially-wetting particles and surrounded by an ambient gas^{21,22}. Here we study first the mass evolution and lifetime of water gas marbles for different ambient humidities (Fig.2a and 2b). Gas marbles of radius 5.1 ± 0.1 mm are produced with the simple following process (see supplementary movie M3): First, some partially-wetting particles (polyamide-11 particles, average radius $r_p = 80 \pm 20$ μ m, contact angle with water $71 \pm 3^\circ$ ²⁴), are spread at the surface of a water liquid bath to form a jammed granular raft. Second, a controlled volume of air is injected with a syringe below the granular raft, leading to the formation of air bubbles with a spherical dome shape and whose upper interface is covered with particle. Third, the bubble is pushed with a spoon toward the surrounding particle raft and made roll over this raft to cover its whole surface with particles. Finally, since the particles on the bubble surface and the remaining particles on the raft prevent contact between the liquid bath and the liquid film, the bubble can be safely extracted with the spoon and placed over a hydrophobic (Teflon) or superhydrophobic (nanopillar) substrate. These surfaces prevent wetting of the substrate by the liquid film. The evolution of the gas marble mass (Fig.2a, 2b) and shape are respectively monitored with a FAS224 Fisherbrand high precision balance and a Nikon D850 camera with a 25 mm ultra macro Laowa lens at 2 frames per minute (Fig.2a), 1 frames per minute (Fig.2b) or 1 frame per 5 minutes (Fig.2c, 2d). The relative humidity is measured with a A+life TH1818

hygrometer. Fig.2a shows the evolution of the mass of a single water gas marble. Bubble drying is visualized with the addition of a blue dye and associated color change, while the bubble opening (marked with a yellow triangle on Fig.2a) can be seen by the brutal change in the bubble shape when the bubble shell is no more airtight (see Movie M4). These data show (i) that the bubble mass decreases linearly until its complete drying (plateau region after 54 minutes on Fig.2a) and (ii) that the gas marble opening (yellow triangle) occurs a few minutes before its complete drying, thus underlying that the water gas marble lifetime is mainly determined by the evaporation process. This scenario is further confirmed by numerous experiments performed for different ambient humidities (Fig. 2b). Hence, the presence of the particle shell inhibits the drainage of the liquid, which, in absence of the particles would provoke bubble bursting after a characteristic time of the order of $\tau = \mu/\rho_l g R \sim 20$ μ s. Note that the bubble lifetime variability on Fig.2b at same ambient humidity can be attributed to the fact that we do not control the initial amount of liquid in the shell during the gas marble formation process, which can result in different initial film thicknesses. This can be seen by the difference in the initial mass of the gas marble (see e.g. the orange and green curves at humidity 0.43), while the final mass (corresponding to particles) is relatively similar.

III. NEUTRALIZING EVAPORATION

To achieve "everlasting" bubbles, i.e. bubbles with very long lifetimes (exceeding at least several months in a standard atmosphere), we must further neutralize the evaporation of the liquid contained in the bubble shell. This can be achieved by adding glycerol to water. Indeed, glycerol is a liquid with a high concentration of hydroxyl groups (one for each of its carbon atoms). These hydroxyl groups have a strong affinity with water molecules, with whom they create hydrogen bonds⁷. This mechanism is at the origin of glycerol's hygroscopicity (ability to absorb water molecules contained in air), which can compensate for the water evaporation. To test this hypothesis, bubbles were synthesized with the same process as described in the previous section but water is replaced by a water/glycerol mixture. The evolution of the mass of water/glycerol gas marbles for different initial glycerol mass ratio $\omega_{go} = m_{go}/(m_{wo} + m_{go})$ (with m_{go} and m_{wo} the glycerol and water mass content of the mixture) and different values of the ambient relative humidity (given in SI) is shown on Fig.2c. This figure shows that the shell of water/glycerol gas marbles with the lowest initial glycerol concentrations ($\omega_{go} \in [0, 0.15]$) ruptures after lifetimes of the order of 50 min (similarly to water gas marble), while the bubbles with higher glycerol concentration tend toward a steady state either by initially losing some water (for $\omega_{go} \in [0.30, 0.65]$), keeping a mass relatively constant (for $\omega_{go} = 0.85$) or absorbing some water from the

IV. MODEL

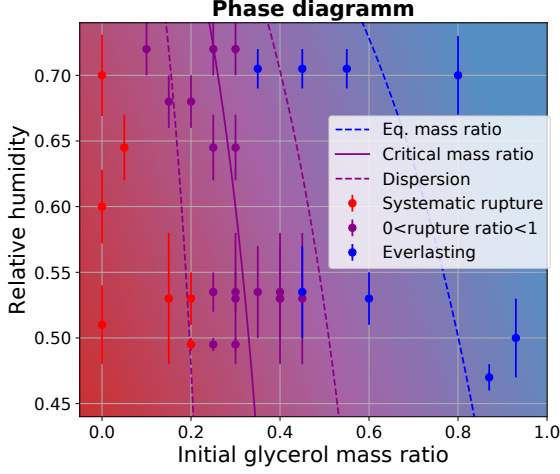


FIG. 3. Diagram of gas marble fate after six hours as a function of the initial glycerol mass ratio and the relative humidity. Each point corresponds to statistics made on at least ten gas marbles. Red: Systematic rupture of the bubble. Purple: Conditions leading to part of the bubble sample rupture (rupture ratio $\in]0, 1[$). Blue: gas marble keeping their integrity after six hours. The vertical bar indicates the variation of the relative humidity for each set of experiment. Note that six hours was chosen as a reference time since our data (cf. Fig.2d) indicate that the time required to reach a stable state is always significantly lower than this value. The dashed blue curve entitled "Eq. mass ratio" represents the equilibrium glycerol mass ratio $\omega_{geq}(h)$ and the purple continuous line the critical glycerol mass ratio ω_{geo} as defined in section IV.

humidity contained in the air (for $\omega_{go} = 0.9$). Indeed, since glycerol is a non-volatile liquid, the mass variations of these gas marbles are solely due to water evaporation or absorption. The evolution toward a steady state with an equilibrium between the water and glycerol content is even clearer when we study the evolution of the normalized glycerol mass ratio (Fig.2d), which tends toward a constant value (slight fluctuations toward the equilibrium value may be attributed to recorded fluctuations in the ambient air humidity). We further studied the fate of gas marbles as a function of the initial glycerol mass ratio and relative humidity (Fig.3). An important element to understand these results is that when a water/glycerol mixture is in contact with humid air with a relative humidity h , it will tend toward an equilibrium glycerol mass ratio ω_{geq} materialized by the blue dotted line on Fig.3. The diagram shows that when the initial glycerol mass ratio ω_{go} is close to this equilibrium value, the gas marble evolves toward a stable equilibrium state (everlasting bubbles) either by losing some liquid (when $\omega_{go} < \omega_{geq}$) or absorbing some liquid (when $\omega_{go} > \omega_{geq}$). When the initial glycerol mass ratio is however too far from this equilibrium value, the gas marble will systematically rupture.

To rationalise this behaviour, we introduce a simple model able to quantitatively reproduce the experimental trends described in Fig.2 and 3 without any adjustable parameter. Since glycerol is a non-volatile liquid, the liquid mass m_l evolution of a water/glycerol gas marble is set by an equilibrium between the flux of water evaporation and the flux of water absorption due to the hygroscopicity of glycerol: $dm_l/dt = \phi_{abs} - \phi_{evap}$. Water evaporation flux is driven by the vapor concentration gradient and hence is proportional to $(1 - h)$, with h the relative humidity of air far from the bubble^{11,16}. Furthermore, since the shell liquid is a mixture of glycerol and water, the water evaporation flux will be proportional to the quantity of water molecules present at the bubble surface. Assuming that the diffusion time τ_d of water molecules into the water/glycerol mixture is short compared to the characteristic time τ_c required for a bubble to reach its equilibrium state, the water/glycerol mixture in the film can be supposed to be homogeneous at the bubble mass evolution timescale. This hypothesis is well verified since a rough estimation of the diffusion time $\tau_d = e^2/D_{wg}$, with $e \sim 150 \mu\text{m}$ (estimated from the surface of the bubble and its mass) and $D_{wg} \gtrsim 10^{-10} \text{ m}^2\text{s}^{-1}$ the diffusion coefficient of water in a water/glycerol mixture¹⁰ gives $\tau_d \lesssim 3 \text{ min}$, which remains relatively small compared to the bubble mass evolution characteristic time $\tau_c \gtrsim 100 \text{ min}$. Hence, the water evaporation rate will be proportional to the concentration of water in the water/glycerol mixture $(1 - \omega_g)$ with $\omega_g = m_g/m_l$, m_g the glycerol mass, $m_l = m_g + m_w$ the liquid mass and m_w the water mass. We thus obtain: $\phi_{evap} = k_e(1 - h)(1 - \omega_g)$, with k_e a constant to be determined. Conversely the absorption of water through the formation of hydrogen bonds with glycerol molecules will be proportional to the concentration of water in the liquid film ω_g , so that $\phi_{abs} = k_a h \omega_g$. The final equation: $dm_l/dt = k_a h \omega_g - k_e(1 - h)(1 - \omega_g)$ relies on two unknown coefficients k_e and k_a , which depend on the evaporation regime, absorption efficiency and bubble geometry. The first coefficient can be determined by examining the case of pure water gas marble. In this case, $\phi_{abs} = 0$ and $\phi_{evap} = k_e(1 - h)$. A first achievement of this model is that the linear trend is well recovered on Fig.2b. The value of the coefficient $k_e = 2.3 \pm 0.2 \times 10^{-8} \text{ kg s}^{-1}$ is obtained by taking the median value of the slopes in Fig.2b, which enables to obtain good fits for all the curves. The second coefficient is determined by examining the equilibrium state $dm_l/dt = 0$. In this case, the mass concentration of glycerol in the water/glycerol mixture reaches a well established equilibrium value³, which depends on the relative humidity of the ambient air. Our model predicts a relation between the equilibrium glycerol mass ratio ω_{geq} and the relative humidity given by: $\omega_{geq} = [1 + k_a h / k_e(1 - h)]^{-1}$. A second achievement of this model is that this law fits well the abacus available in the literature³ (see Fig.2 in SI), with $k_a/k_e = 0.248$,

giving good confidence in the dependence of the evaporation and absorption fluxes on the glycerol concentration ω_g and relative humidity h . Now, that the two coefficients k_a and k_e , are determined we can compare the model predictions to the experimentally measured gas marble mass evolution. The results show that this simple model recovers quantitatively the trends observed on Fig.2c and 2d. From this model, we can also derive a criterion for the transition between ephemeral and everlasting bubbles (Fig.3) depending on the initial glycerol concentration ω_{go} and relative humidity h . Assuming that bubbles rupture occurs when the bubble shell liquid mass reaches a critical value m_{lc} , a bubble will be everlasting if its equilibrium liquid mass m_{leq} is larger than m_{lc} and ephemeral if m_{leq} smaller than m_{lc} . The transition between these two regime hence occurs for $m_{leq} = m_{lc}$. Since the mass of glycerol m_g is constant, we have $m_g = m_{leq}\omega_{geq} = m_{lo}\omega_{go}$. Hence for $m_{leq} = m_{lc}$, we obtain $\omega_{goc} = m_{lc}\omega_{geq}(h)/m_{lo}$ with $\omega_{geq}(h)$ determined previously. The critical liquid mass $m_{lc} = 14 \pm 4$ mg is determined by measuring the bubble total mass $m_c = 32 \pm 3$ when its rupture occurs (measured here for 4 different gas marbles) and subtracting the particle mass $m_p = 18 \pm 2$ (value extracted from Fig.2b after total evaporation of water). Since, as mentioned before, we cannot control the liquid content during the gas marble formation process, an ephemeral gas marble initial content m_{lo} varies typically between 22 mg and 57 mg. If we plot the equation $\omega_{goc} = m_{lc}\omega_{geq}(h)/m_{lo}$ on Fig.3, we see that this model gives a good criterion for the transition between ephemeral and everlasting bubble (the continuous purple line corresponds to the average value $m_{lo} = 34$ mg, while the purple dashed lines correspond to $m_{lo} = 22$ mg and

57mg respectively and account for the dispersion of the initial state).

V. PERSPECTIVES: COMPOSITE FILMS

In this paper, we have shown that air bubbles keeping their integrity for at least several months can be produced in a simple way by replacing surfactants by partially-wetting particles and water by a water/glycerol mixture. Indeed, partially-wetting particles prevent the drainage of the liquid due to gravity, while the hygroscopicity of glycerol-water mixtures counterbalances the evaporation, which are the two main mechanisms at the origin of classical bubble bursting. In addition, the neutralisation of these two phenomena enables to make bubble insensitive to the nuclei contained in the air. But beyond bubbles, this work unveils a robust composite liquid film, which can be used to create a wealth of different objects. Indeed, when a metallic frame is dipped below a liquid surface covered with a layer of jammed particles and lifted slowly up to the surface, the metallic frame captures the particle-covered films (see Fig.4), similarly to what happens with surfactants. While marginal pinching leads to soap films bursting near the edge after a few seconds (Fig.4.a), the replacement of surfactants by partially-wetting particles leads to the apparition of evaporation induced cracks after several minutes only (Fig.4.b), and the further addition of glycerol leads to long lasting liquid film stable for at least 5 weeks (Fig.4.c). Following this process, complex objects such as everlasting pyramidal films can easily be manufactured (Fig.4.d) paving the way toward the design of new fluidic objects with unexplored physical and chemical properties.

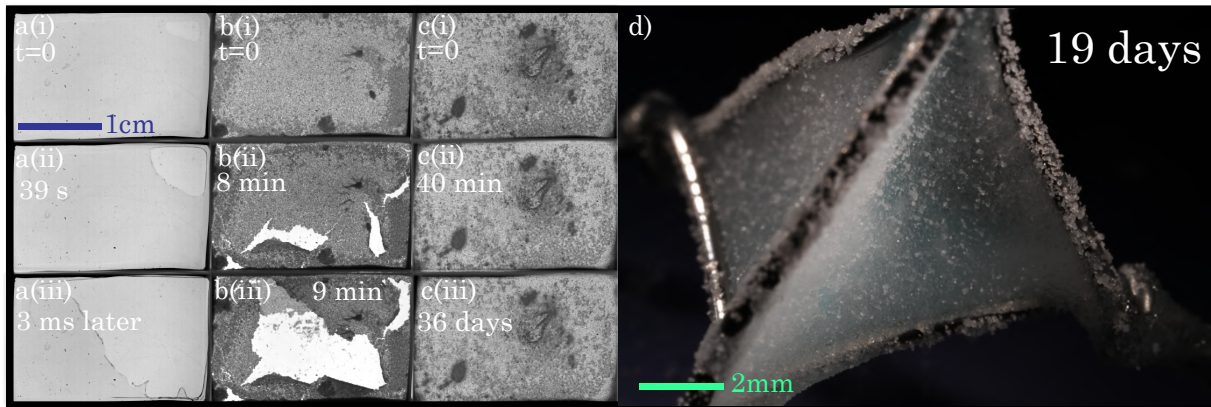


FIG. 4. Lifetime of liquid films attached to a metallic frame (a,b,c: horizontal frame. d. pyramidal frame) a) Soap film made of water and surfactant. b) Water film covered with partially wetting particles. c) Water/glycerol liquid film (initial glycerol mass concentration $\omega_{go} = 0.80$) covered with particles. d) 3D object created with a pyramidal frame supporting a water/glycerol liquid film covered with particles.

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VII. ADDITIONAL INFORMATION

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