Supporting Information
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Stabilization of Liquid Foams through the Synergistic Action of Particles and an Immiscible Liquid**
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Supporting Information

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1. Materials

Polyvinyl chloride particles with different size and wettability (Vinnolit SA 1062/7 and Vinnolit P70F) were obtained from Vinnolit, Germany. Polyethylene (PE) particles were purchased from Polysciences, Inc., USA. The silica samples (Aerosil 200) were obtained from Evonik, USA and then treated with trimethoxymethylsilane as provided by Sigma-Aldrich. Cellulose powder was purchased from Sigma-Aldrich and then modified with poly(maleic anhydride-1-octadecene) obtained from Polysciences, Inc., USA. The glass samples were obtained from Potters Industries LLC as Spheriglass 5000 solid glass microspheres. The glass was cleaned with acetone. The aluminum oxide particles were purchased from Sigma-Aldrich. Monodisperse silica spheres (SS03N, 0.96µm) were obtained from Bangs Laboratories, Inc. Scanning electron micrographs of particles used in this study and particles size distribution are shown in Figure S1 and Figure S2 respectively. The secondary fluids used in this study were trimethylolpropane trimethacrylate (TMPTMA, Sigma-Aldrich), paraffin (Sigma-Aldrich), and diisononyl phthalate (DINP, Sigma-Aldrich). Polyvinyl chloride particles (Vinnolit SA1062/7) were washed with deionized water several times before use. The gelling agent for the water phase used in the gel trapping method was gellan gum (Kelcogel, CPKelco). The hot gelling solution was purified by passing it twice through a C18–silica chromatographic column (Phenomenex) preactivated with an acetonitrile (Sigma-Aldrich)-water (80:20) mixture. Sulfuric acid and hydrogen peroxide were purchased from VWR international and EMD Millipore respectively. All the chemicals were used as received, unless otherwise noted. Ultrapure water with a resistivity of 18.2 MΩ·cm (Barnstead) was used.
Figure S1. SEM images of the particles used in this study. a) Modified silica (Aerosil 200), mean diameter: 619.15 nm. b) Modified cellulose powder, mean diameter: 33.90 µm. c) PVC Vinnolit SA/1062/7, mean diameter: 29.59 µm. d) Aluminum oxide particles, mean diameter: 5.27 µm. e) Glass (Spheriglass 5000), mean diameter: 4.44 µm. f) Polyethylene particles, mean diameter: 9.65 µm. g) Monodisperse silica spheres (SS03N), diameter: 0.96 µm. h) PVC Vinnolit P70F, mean diameter: 0.93 µm.

Figure S2. Particle size distributions.

2. Contact angle measurements

The recently developed gel-trapping technique (GTT) has proven to be a powerful method for determining particle contact angles at a fluid-fluid interface and was used in this work.\textsuperscript{S1,S2} The GTT involves spreading colloidal particles at a water-oil or water-air interface and subsequent gelling of the aqueous phase with a non-surface-active gelling agent.\textsuperscript{S1} The top phase is removed and replaced with curable polydimethylsiloxane (PDMS), which is then cured.
and peeled off the gel. The PDMS replica of the interface with entrapped particles was imaged by Scanning Electron Microscope (SEM). The height of protrusion, $h$, and the equatorial radius, $R$, of particles at the interface were obtained from the SEM images (Figure S3). The particle contact angle is given by \[^{83}\]

$$\theta = \arccos \left( \frac{h}{R} - 1 \right)$$  \hspace{1cm} (1)

The value of the particle contact angle was determined by averaging over a large number of particles.

**Figure S3. Schematic representation of a PDMS replica with trapped particle.** a) Contact angle smaller than 90°. b) Contact angle larger than 90°.

The GTT protocol reported in the literature typically starts with the introduction of particles to interface using a spreading solvent such as isopropanol (IPA).\[^{81,82}\] Here, the particles were delivered to the interface using mechanical force instead of a spreading solvent, because spreading solvents have been demonstrated to affect the particle wetting properties, IPA for instance making particles appear more hydrophobic.\[^{84}\] Our sample preparation protocol for GTT proceeded as follows: in the case of the air-water interface, 0.001 wt% particles were dispersed in water by sonication (VWR Model B2500A- MT ultrasonic cleaner) for 30 minutes and then delivered to the air-water interface by homogenization (IKA UltraTurrax T10, stator diameter of 8 mm and rotor diameter of 6.1 mm) in a Petri dish. Air bubbles were removed with a mild vacuum (100 mbar), and the sample temperature was raised to 50–55 °C. An aqueous 3 wt% solution of gel solution was added to the hot aqueous phase. The system was then cooled quickly to 25 °C to set the gel and immobilize the particles at the interface. PDMS was spread on the top of the aqueous gel and cured to generate a PDMS replica with trapped particles. For the case of oil-water interface, the same procedure was used to prepare the aqueous dispersion, deliver the particles to the interface, and remove air bubbles. Then the oil was carefully added at the air-
water interface. The sample was moved to the oven at 50-55 °C. An aqueous 3 wt% solution of gel solution was added to the hot aqueous phase. The system was cooled quickly to 25 °C to set the gel. Then, the oil phase was carefully removed and immediately replaced with PDMS.

After curing the PDMS layer at room temperature for 48 h, it was peeled off the aqueous gel (along with the entrapped particles at the interface) and washed with pure hot water to remove any gel residues from the PDMS surface. The PDMS replica with trapped particles were prepared for imaging with SEM by coating with a gold nanolayer in a Hummer 5 Gold/Palladium Sputter and the contact angle was calculated from the geometric parameters obtained by SEM for at least 3 and up to 13 particles trapped in the interface.

The hydrophilic silica particles (SS03N) used in this study could not be trapped in the PDMS replica of the oil-water interface because of their low affinity for the interface. In this case the contact angle was estimated using macroscopic contact angle measurements with a Ramé-Hart goniometer on a glass slide pretreated with “piranha solution” (a 7:3 mixture of concentrated sulfuric acid and 30 % hydrogen peroxide) to mimic the surface wettability of bare silica particle. For the contact angle of oil/water interface, the glass substrate was submerged in a quartz cell filled with water and contact angle measurements were carried out on inverted sessile oil drops. The contact angle for the air/water interface was determined from observation of sessile water drops in air. Reported contact angle values are averages of data for 5 different drops.

3. Surface tension and interfacial tension measurements

To measure the equilibrium surface and interfacial tensions, equal volumes of water and oil were vigorously stirred overnight to achieve mutual saturation, and separated by centrifuge prior to the experiment. The surface tension of oil against air was measured with a Lauda TVT-2 drop volume tensiometer. Interfacial tensions of water against air and oil were measured via shape analysis of pendant drops with a Ramé-hart model 250 goniometer. The surface and interfacial tensions are reported in Table S1. Reported surface and interfacial tensions are averages over five independent measurements, the experimental uncertainty is on the order of 1 mN/m.

<table>
<thead>
<tr>
<th>Table S1. Surface and interfacial tensions of fluids used in this study.</th>
<th>Equilibrium surface and interfacial tensions</th>
<th>Surface and interfacial tensions of pure liquids</th>
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</thead>
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<td>$\gamma_{ao}$ (mN/m)</td>
<td>$\gamma_{aw}$ (mN/m)</td>
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<td>51.74</td>
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<td>DINP</td>
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<td>57.43</td>
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<td>Paraffin oil</td>
<td>32.95</td>
<td>72.00</td>
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</table>
4. Evidence that secondary liquid alone cannot stabilize foams

We report that the addition of a secondary liquid can lead to stable foams even when the particles do not have the required wetting behavior to stabilize air bubbles by themselves (Figure 1 in the main manuscript) and find that stabilization of bubbles requires both particles and a secondary liquid. In order to further support the synergistic action of particles and a secondary fluid on the stabilization of capillary foams, here we investigate whether the foam bubbles can be stabilized in the presence of only a secondary fluid. Figure S4 shows that no foam head is produced when frothing a particle-free systems with only a secondary fluid, such as Paraffin, TMPTMA or DINP (14.16 wt.%).

Figure S4. Attempts of producing foam via frothing in the presence of secondary liquids only (14.16 wt. %).

5. Evidence that the formation of capillary particle networks in the liquid bulk is not sufficient to ensure good stabilization of foam bubbles

As mentioned in the main manuscript, we find that capillary particle networks in the liquid bulk are not sufficient to ensure good stabilization of foam bubbles. This is also evident in Figure S5a, a picture of capillary foams resulting from aluminum oxide particles and TMPTMA as the oil. It can be seen that foams are only formed in the upper region, while most aluminum oxide particles settle down at the bottom of the container. For both regions, particles networks are formed. The only difference between the two regions is that in the upper region, a thin oil film is adsorbed at the surface of air bubbles. This experimental result further supports that the formation of capillary particle networks in the liquid bulk is not sufficient to ensure good stabilization of foam bubbles.
Figure S5. Attempts of producing capillary foam from aluminum oxide particles suspension. a) Attempts of producing capillary foams from aluminum oxide particles suspension (30 wt.%) in the presence of a secondary fluid TMPTMA (3 wt.%, with respect to the amount of bulk phase water). b) An SEM image of a dried capillary foam. The secondary fluid was solidified by photo-polymerization, and the water was then removed by drying. c) An SEM image of region among different bubbles. d) An SEM image of particles settle region. e) Close-up of particles network.

6. The interfacial free energy of capillary foams and the effective oil spreading coefficient

Ordinary liquid foams are two phase systems in which a large volume of gas is dispersed as bubbles in a continuous liquid phase. We consider the case of air as the gas phase and water as the continuous liquid. Aqueous foams are thermodynamically unstable due to the large interfacial free energy cost associated with the bubble surfaces. If the continuous water phase contains suspended colloidal particles of appropriate wettability, these particles can adsorb strongly to the air-water interface and hinder the coalescence of foam bubbles kinetically as described in the manuscript; such particle-stabilized foams are often referred to as Pickering foams in analogy to the particle-stabilized Pickering emulsions.

In the capillary foams investigated in our study, the air-water interface is replaced (at least to a large extent, if not entirely) by a film of oil (the secondary liquid), where particles now adsorb to the oil film interface with the air bubble or with the continuous water phase. The adsorption state (oil-water interface or the air-oil interface) yielding the lower interfacial energy was
considered the preferred (predicted) state of the bubble surface in the capillary foams. Here we estimate the total interfacial energy change (inverse of effective spreading coefficient) for capillary foams from different particles and secondary fluid combinations. To this end the following simplifying assumptions and notations are adopted:

(1) The internal energy, pressure, and temperature are assumed constant, and the small entropy changes due to particle adsorption are neglected.

(2) Particles are considered spherical and effects of particle surface charge and size polydispersity are neglected.

(3) Gravitational force and particle-particle interactions are neglected against surface and interfacial tension forces.

(4) Particles are initially dispersed in the water phase.

(5) Effects of bubble curvature are neglected.

(6) The interfacial tensions of the oil/water, air/oil, air/water, air/particle, water/particle and oil/particle interfaces are denoted as $\gamma_{ow}$, $\gamma_{oa}$, $\gamma_{aw}$, $\gamma_{ap}$, $\gamma_{wp}$ and $\gamma_{op}$. In systems containing both water and oil, mutual saturation of the two liquid phases is assumed.

(7) $\theta_{wop}$ and $\theta_{aop}$ denote the particle contact angle at the water/oil and air/oil interfaces measured through the oil phase as is customary in the literature on particle-assisted wetting. $\theta_{awp}$ is the particle contact angle at the air/water interface measured through the water phase as customary in the literature on Pickering emulsions.

The interfacial energy per unit bubble area in a Pickering foam is the adsorption energy of particles attaching to the bubble surfaces,

$$\frac{G_{\text{Pickering}}^{\text{inf}}}{A} = \gamma_{aw} + \frac{n_p}{A} \Delta G_{\text{Pickering}}$$

where $n_p$ is the number of particles attached onto the air-water interface, and $\Delta G_{\text{pickering}}$ is the adsorption energy per particle, given by

$$\Delta G_{\text{Pickering}} = -\pi R^2 \gamma_{aw} \left(1 - \cos \theta_{awp}\right)^2.$$

Combining Eqs. 1 and 2 and normalizing by the bubble surface area yields for the interfacial energy per unit bubble area of the Pickering foam

$$\frac{G_{\text{Pickering}}^{\text{inf}}}{A} = \gamma_{aw} \left[1 - \phi \left(1 - \cos \theta_{awp}\right)^2\right],$$

where $\phi$ is the volume fraction of particles in the water phase.
where $\phi = n_p \pi R^2 / A$ is the packing density (area fraction) of the particles adsorbed in the interface. Similar considerations can be applied to capillary foams. The interfacial energy change per unit bubble area in capillary foam is the sum of the free energy required for spreading an oil film at that interface with the assistance of adsorbed particles.

$$
\frac{G_{\text{capillary}}^{\text{inf}}}{A} = \gamma_{aw} - S_{\text{eff}} = \gamma_{aw} - S_o + \frac{n_p}{A} \Delta G,
$$

(4)

where $S_{\text{eff}}$ is the effective spreading coefficient accounting for particles adsorption at one of the oil interfaces, $S_o = \gamma_{aw} - (\gamma_{oa} + \gamma_{ow})$ is the standard spreading coefficient of the oil, and $\Delta G$ is again a particle’s energy change upon transfer from the water bulk to an interface.

Spreading of the oil around the air bubbles requires that particles adsorb at the oil-water interface (case 1), or the oil-air interface (case 2), or at both interfaces; and the particle wettability dictates which, if any, of these scenarios are realized. For the case 1 of particles adsorbing at the oil-water interface, the particle adsorption energy $\Delta G_1$, is given by

$$
\Delta G_1 = -\pi R^2 \gamma_{ow} \left(1 + \cos \theta_{wop}\right)^2.
$$

(5)

Therefore, the interfacial energy of the capillary foam with particles adsorbed at the oil-water interface is given by

$$
\frac{G_{\text{capillary}}^{\text{inf}}}{A} = \gamma_{aw} - S_{\text{eff}, 1} = \gamma_{aw} - S_o - \phi \gamma_{ow} \left(1 + \cos \theta_{wop}\right)^2,
$$

(6)

where $\phi$ again stands for the particle packing fraction, this time in the oil-water interface. For the case 2 of particles adsorbed at the air-oil interface, the energy change upon bringing particles from the water bulk to the air-oil interface, $\Delta G_2$, is given by

$$
\frac{-\Delta G_2}{\pi R^2} = \gamma_{oa} \sin^2 \theta_{aop} + 2 \left[ \cos \theta_{aop} (\gamma_{ap} - \gamma_{op}) + (\gamma_{wp} - \gamma_{ap}) + (\gamma_{wp} - \gamma_{op}) \right].
$$

(7)

The interfacial tensions involving the particle surface are not directly accessible experimentally, but can be eliminated using Young’s equations

$$
\gamma_{ap} - \gamma_{op} = \gamma_{oa} \cos \theta_{aop},
$$

(8)

$$
\gamma_{wp} - \gamma_{ap} = -\gamma_{aw} \cos \theta_{awp}.
$$

(9)

$$
\gamma_{wp} - \gamma_{op} = \gamma_{ow} \cos \theta_{wop}.
$$

(10)
Equation 7 then takes the form

$$-\frac{\Delta G}{\pi R^2} = \gamma_{oa} (1 + \cos^2 \theta_{awp}) + 2 \left( \gamma_{w} \cos \theta_{wop} - \gamma_{aw} \cos \theta_{awp} \right).$$

(11)

Hence, the interfacial energy per unit bubble area for a capillary foam with particles adsorbed at the air-oil interface is

$$\frac{G_{\text{capillary,2}}^{\text{intf}}}{A} = \gamma_{aw} - S_{\text{eff,2}} = \gamma_{aw} - S_o - \phi \left[ \gamma_{oa} (1 + \cos^2 \theta_{awp}) + 2 \left( \gamma_{w} \cos \theta_{wop} - \gamma_{aw} \cos \theta_{awp} \right) \right].$$

(12)

The particle contact angles $\theta_{wop}$ and $\theta_{awp}$ were measured by GTT, and the contact angle $\theta_{awp}$ was inferred from the measured contact angles ($\theta_{wop}$, $\theta_{awp}$) and the measured interfacial tensions ($\gamma_{w}$, $\gamma_{oa}$, $\gamma_{aw}$) via Young’s relation:

$$\gamma_{oa} \cos \theta_{awp} = \gamma_{aw} \cos \theta_{awp} + \gamma_{w} \cos \theta_{wop}$$

(13)

Equations (6) and (12) were used to calculate the interfacial free energy per unit bubble surface area and effective spreading coefficient for scenarios 1 (particles adsorb at the oil-water interface) and 2 (particles adsorb at the air-oil interface), assuming for both cases an interfacial particle packing density (area fraction) $\phi$ of 70% based on a crude estimate from microscopic observations. A positive effective spreading coefficient ($S_{\text{eff}}>0$) means that the spreading of secondary fluid around gas bubbles with the help of particles is thermodynamically favorable. The thermodynamically “preferred interface” for interfacial adsorption of particles used in this study and the corresponding calculated spreading coefficients are indicated in Table S2 below.

For some of the larger particles used in this study, SEM images suggest that particles may simultaneously penetrate both interfaces of the oil film around the droplets. A thermodynamic model for this scenario, which would have to take into account the film thickness and thus the fraction of oil not spread at the droplet surfaces, is beyond the scope of the present publication.

**Comparison between the experimentally observed particle location at the interface and theoretical expectations**

For PVC (Vinnolit SA 1062/7) particles in water with added TMPTMA and air bubbles, the model predicts a free energy change per unit area of $-S_{\text{eff,1}} = -11.05 \text{ mJ/m}^2$ for spreading of an oil film around them if the particles adsorb to the oil-water interface at an assumed area coverage of 70%; by contrast, if the particles instead adsorb to the oil-air interface, the predicted energy change would be $-S_{\text{eff,2}} = +4.55 \text{ mJ/m}^2$. From this, one would expect the particles to adsorb preferentially at the oil-water interface. Experimentally, a layer of TMPTMA, spread at a macroscopic air-water interface with the help of the same PVC particles, was solidified by photo-polymerization. Figure S6a shows an SEM image of the solidified oil layer with the particles adsorbed in the oil-water interface with a three-phase contact angle near 90°, favoring strong particle adsorption. Hence the SEM images of Figure S6 are consistent with the spreading
and stabilization of an oil film at the bubble surface by PVC particles (Vinnolit SA 1062/7) preferentially adsorbing at the oil-water interface.

![Image](image1.png)

**Figure S6. Experimental observation of the particle location at the interface.** a) SEM image of the mixed layers of TMPTMA and particles formed on an air-water interface. b) SEM image of PVC particles trapped on the PDMS replica of oil-water interface using the gel trapping technique. The visible part of particle has been immersed in the water phase.

**Preparation details for the mixed layer of secondary fluid and particles at a macroscopic air-water interface**

0.01 wt% particles were dispersed in water by sonication for 30 minutes and then delivered to the air/water interface by homogenization with an IKA UltraTurrax T10 homogenizer in a Petri dish. Air bubbles were removed by exposure to a mild vacuum (100 mbar). Next, TMPTMA (with 5 wt% photoinitiator benzoinisobutylether) was added to the air-water interface slowly and carefully. A mixed wetting layer of oil and particles formed and was solidified by photo-polymerization (after 48 hours, using irradiation with UV light of 365 nm wavelength for 5 hours) and transferred to solid substrates such as gold electron microscope grids for subsequent imaging analysis.
Figure S7. Experimental observations of foam stability where a variety of particles and fluid combinations were tested. The particles tested cover a range of particle size, shapes and wettability.

Table S2. Comparison between experimental observations and theoretical estimates for foams from a variety of particle and fluids combinations. $\theta_{awp}$ and $\theta_{owp}$ are particle contact angles at air-water interface and oil-water interface respectively. $S_{eff}$ is the effective spreading coefficient. The cellulose and aluminum oxide particles are not included here because it is challenging to measure contact angles for these irregularly shaped particles.
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<th>$\theta_{\text{exp}}^{(\circ)}$</th>
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* Only a small amount foam is formed, and most particles settle at the container bottom.
7. References


