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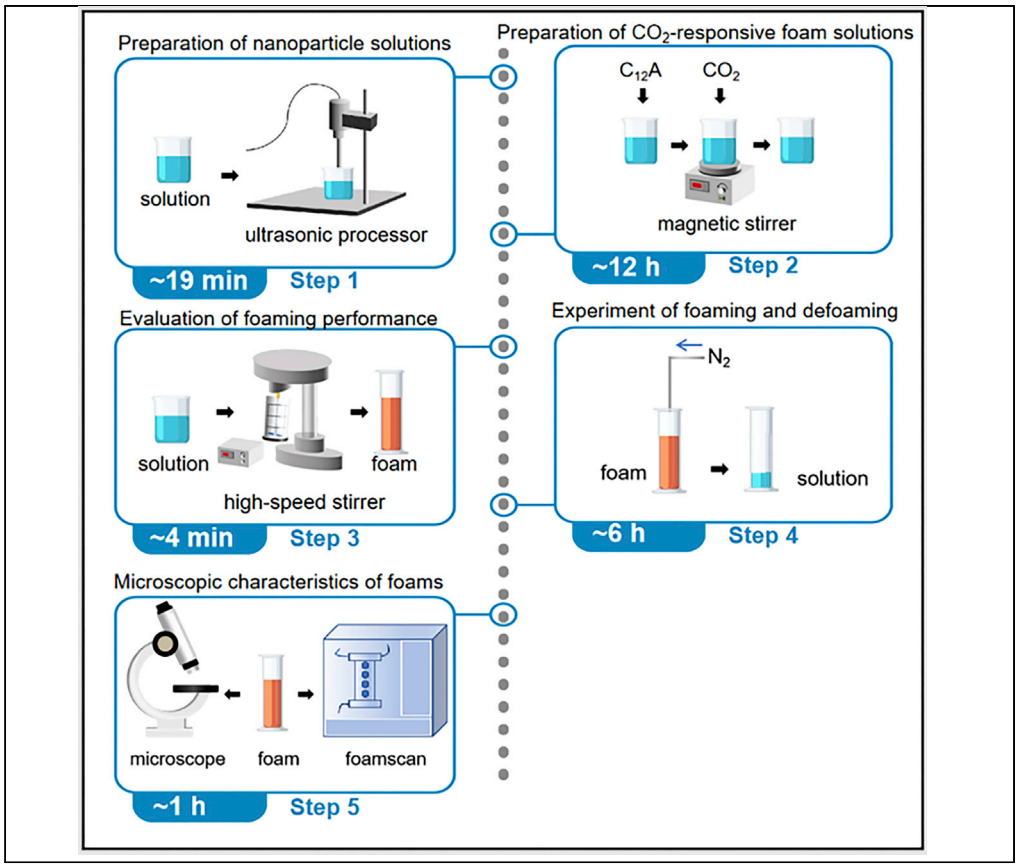
Protocol

Protocol for preparation and characterization of CO_2-responsive foaming



Despite the unique switching characteristics of CO_2-responsive foaming, its stability remains questionable. In this protocol, we describe steps to synthesize a stable CO_2-responsive foam by adding the preferably selected hydrophilic nanoparticle N20 into the surfactant $C_{12}A$. We detail the selection of the most suitable nanoparticles for the surfactant by measuring the foaming volume and half-life of the dispersion. The protocol can be extended to manufacture with other types of responsive foams (e.g., light responsive foams, magnetic responsive foams).

Publisher's note: Undertaking any experimental protocol requires adherence to local institutional guidelines for laboratory safety and ethics.

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Highlights

Detailed protocol for preparation of CO_2-responsive foaming

Preparation of aqueous nanoparticle solutions and aqueous CO_2-responsive foam solutions

Evaluation of foam's response to CO_2 and N_2

Experiment of foaming and defoaming and microscopic characterization of foams

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Protocol for preparation and characterization of CO_2-responsive foaming

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SUMMARY

Despite the unique switching characteristics of CO_2-responsive foaming, its stability remains questionable. In this protocol, we describe steps to synthesize a stable CO_2-responsive foam by adding the preferably selected hydrophilic nanoparticle N20 into the surfactant $C_{12}A$. We detail the selection of the most suitable nanoparticles for the surfactant by measuring the foaming volume and half-life of the dispersion. The protocol can be extended to manufacture with other types of responsive foams (e.g., light responsive foams, magnetic responsive foams).
For complete details on the use and execution of this protocol, please refer to Li et al. (2022).[1]

BEFORE YOU BEGIN

There are many ways to prepare stable CO_2-responsive foams.[2] Several factors are to be considered, such as their applications, large-scale production, stability, production cost, and safety. In this study, we used a simple but effective method to prepare stable CO_2-responsive foams. The ability of the solution to produce foam can be controlled by controlling the electrostatic adsorption between nanoparticles and surfactants.[3] The following protocol describes the specific steps for preparing stable CO_2-responsive foam. The scheme can also be applied to manufacturing other responsive foam (e.g., light responsive foams and magnetic responsive foams).

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
N, N-dimethyldodecylamine ($C_{12}A$)	Macklin Biochemical Co., Ltd.,	CAS: 112-18-5
SiO_2 nanoparticles (V15, N20, T30, T40)	Wacker Chemical Co., Ltd.,	CAS: 112945-52-5
An aqueous solution of SiO_2 nanoparticles (SG07)	Shanghai Zecheng Co., Ltd.,	CAS: 14808-60-7
An aqueous solution of SiO_2 nanoparticles (WT)	Hangzhou Hege Nanotechnology Co., Ltd.,	CAS: 14808-60-7
An aqueous solution of SiO_2 nanoparticles (PT)	Shanghai Zecheng Co., Ltd.,	CAS: 14808-60-7
An aqueous solution of SiO_2 nanoparticles (VK-S01A)	Xuancheng Jingrui new material Co., Ltd.,	CAS: 14808-60-7
Other		
Balance	Mettler-Toledo, Switzerland	N/A
Ultrasonic processor	Hangzhou Success Ultrasonic Equipment Co., Ltd., China	YP-S17

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REAGENT or RESOURCE	SOURCE	IDENTIFIER
High-speed stirrer	Qingdao Senxin, Equipment Co., Ltd., China	Model GJ-3S
FoamScan	Teclis, France	FMS-HTMP
Fourier transform infrared spectrometer	Nexus, USA	NEXUS FT-IR
Stainless-steel needle	Qingdao Wanzhou, Equipment Co., Ltd., China	N/A
Measuring cylinder	Taizhou Aolun Technology Co., Ltd., China	N/A
Microscope	Keyence, Japan	VHX-5000

Note: The particle sizes of V15, N20, T30 and T40 are 14 nm, 10 nm, 7 nm and less than 7 nm, respectively. The particle sizes of SG07, WT, PT and VK-S01A are 7 nm, 10 nm, 15 nm and 12 nm, respectively.

MATERIALS AND EQUIPMENT

Stock solution of saturated nanoparticle solution (storage: 25°C)		
Reagent	Final concentration	Amount
Deionized water	N/A	98.5 g
Nanoparticle	1.5 wt%	1.5 g
Total	N/A	100 g
The solution can be stored for 2–3 days at room temperature.		

Stock solution of saturated CO_2-responsive solution (storage: 25°C)		
Reagent	Final concentration	Amount
Nanoparticle solution	N/A	99.98 g
$C_{12}A$	0.02 wt%	0.02 g
Total	N/A	100 g
The solution can be stored for 2–3 days at room temperature.		

STEP-BY-STEP METHOD DETAILS

Sample configuration for aqueous nanoparticle solutions

Timing: ~19 min

In this section, we describe the preparation of aqueous nanoparticle solutions.

1. Add the 1.0 wt% of SiO_2 nanoparticles to deionized water to form dispersions of different proportions.
2. Disperse all liquids with an ultrasonic processor at 30 kHz for 8 min, let for 3 min, and then disperse again for 8 min while controlling the temperature of the dispersion at 25°C with a water bath.[4]

Sample configuration for aqueous CO_2-responsive foam solutions

Timing: ~12 h

In this section, we describe the preparation of aqueous CO_2-responsive foam solutions.

3. Add 0.02 wt% surfactant $C_{12}A$ to the dispersion, and then inject CO_2 into the solution with a stainless-steel needle at a flow rate of 1 L/min at room temperature of 25°C until the solution reaches saturation.

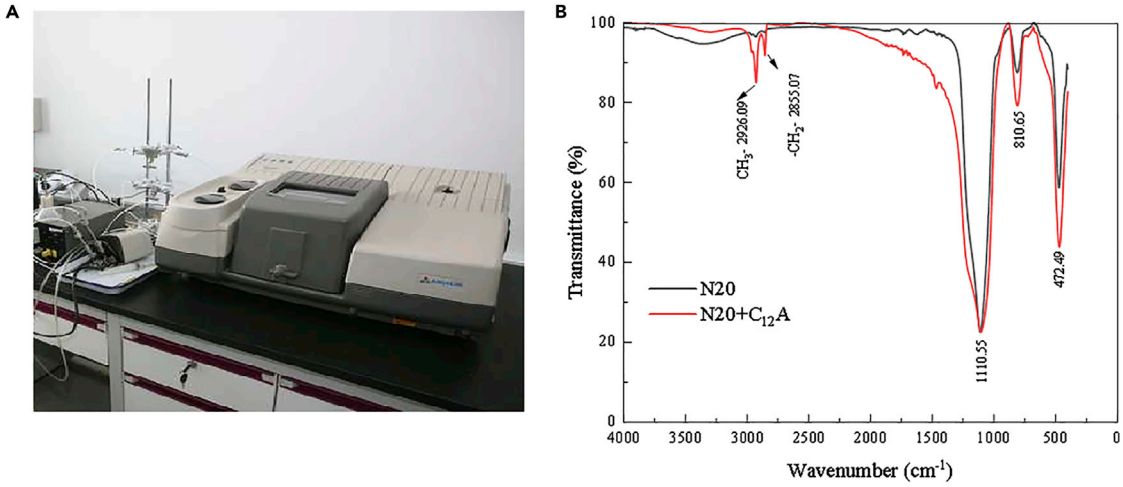

Figure 1. Infrared spectroscopy experiment

(A) Fourier transform infrared spectrometer.

(B) FT-IR spectra of SiO_2 nanoparticles before and after modification with $C_{12}A$. Figure 1 reprinted with permission from Li et al.[1]

4. Leave the dispersions for 1 h in a CO_2 environment at room temperature to stabilize the adsorption of $C_{12}A$ on the surface of SiO_2 nanoparticles.
5. Perform the infrared analysis of SiO_2 nanoparticle adsorbed with $C_{12}A$ and pure SiO_2 nanoparticle.[5]

Note: The FT-IR spectra of SiO_2 nanoparticles before and after modification with $C_{12}A$ was displayed in (Figure 1). It can be seen that the absorption band at 2,926 cm^{-1} corresponds to the telescopic vibration of $-CH_3$, and the absorption band at 2,855 cm^{-1} corresponds to the telescopic vibration of $-CH_2$. The detection of N20 particles and $C_{12}A$-N20 particles showed that $C_{12}A$ was successfully adsorbed on the N20 surface.

Evaluation of foaming performance

Timing: 3 h

In this section, we describe the process of screening out the nanoparticles that best match the surfactant.

6. Pour the configured solution into the mixing cup.
7. Fill the mixing cup with CO_2 for 1 min, replace the air in it with CO_2 and seal the mouth of the mixing cup with plastic wrap to allow it to froth in the CO_2 environment.
8. Use a high-speed stirrer with a stirring time of 3 min and a stirring speed of 8,000 r/min.
9. Transfer the generated foam to the measuring cylinder quickly.
10. Record the time of the initial volume of the foam the time to drain 50 mL of liquid from the foam is the half-life of the drainage.

Note: Because the initial liquid is 100 mL, start timing when the mixer stops, and stop timing when 50 mL of liquid is separated from foam. The time is defined as the half-life of foam drainage, which describes half of the liquid is separated from foam.[6]

11. The foaming volume and half-life of SiO_2 nanoparticles and $C_{12}A$ are shown in (Figure 2). It can be seen from (Figure 2C) that the half-life of $C_{12}A$-N20 foam is 29 min, which is much higher than other foams. Therefore, surfactant $C_{12}A$ and N20 have the best synergistic effect.

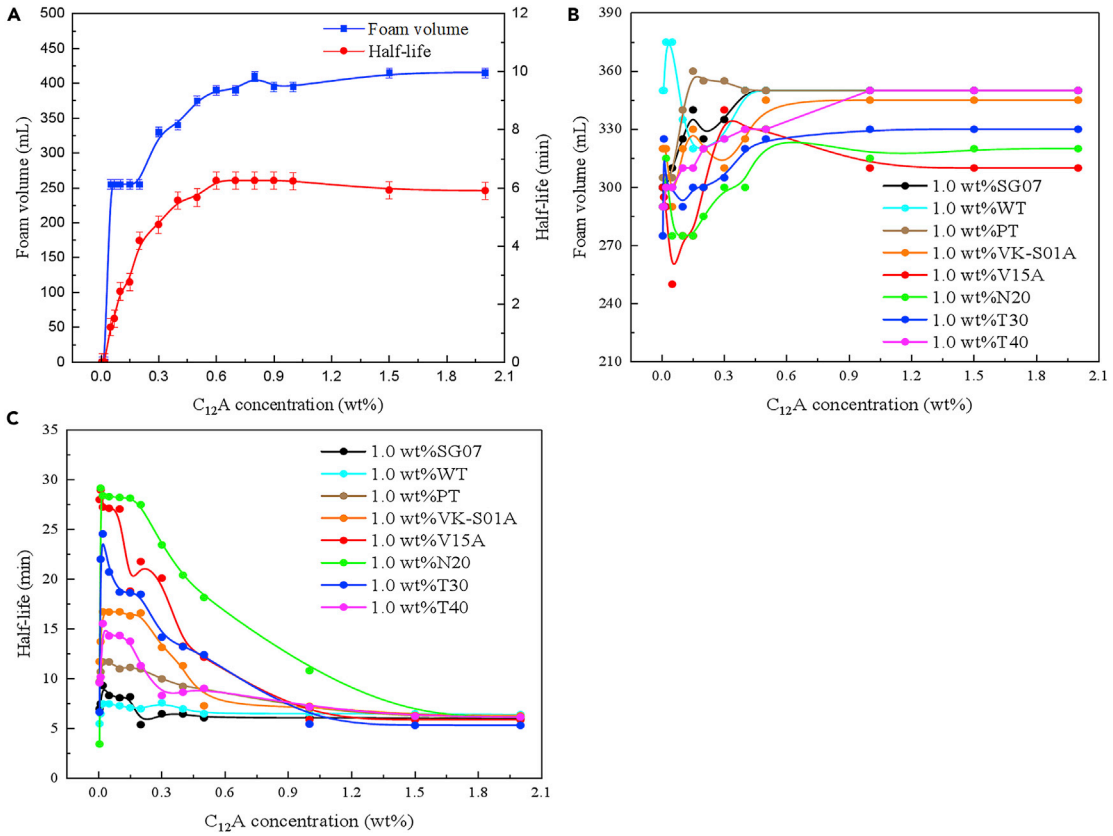

Figure 2. Foam properties of different nanoparticles

(A) Foam properties of $C_{12}A$.

(B) Foam volume of $C_{12}A$-NPS.

(C) Half-life of $C_{12}A$-NPS. Figure 2 reprinted with permission from Li et al.[1]

Note: At present, the most direct and effective way to evaluate the synergy between nanoparticles and surfactants is to measure the foaming volume and half-life of solution. The half-life of foam is used to evaluate the stability of foam. Adding nanoparticles to the solution is mainly to enhance the stability of foam.

Experiment of foaming and defoaming

Timing: ~6 h

In this section, we describe the process of verifying the reproducibility of foaming and defoaming.

12. Use a stainless-steel needle to inject N_2 at a fixed flow rate of 2 L/min into the bottom of the foam in the measuring cylinder and record the defoaming process (Figure 3).
13. Inject CO_2 into the defoaming solution at a flow rate of 1 L/min and allow the solution to react thoroughly with the CO_2. Then foaming with a high-speed mixer in the same way as above.
14. Record foam volume and half-life for three alternating cycles. Perform all experiments at room temperature (25°C).

Microscopic characteristics of foams

Timing: ~1 h

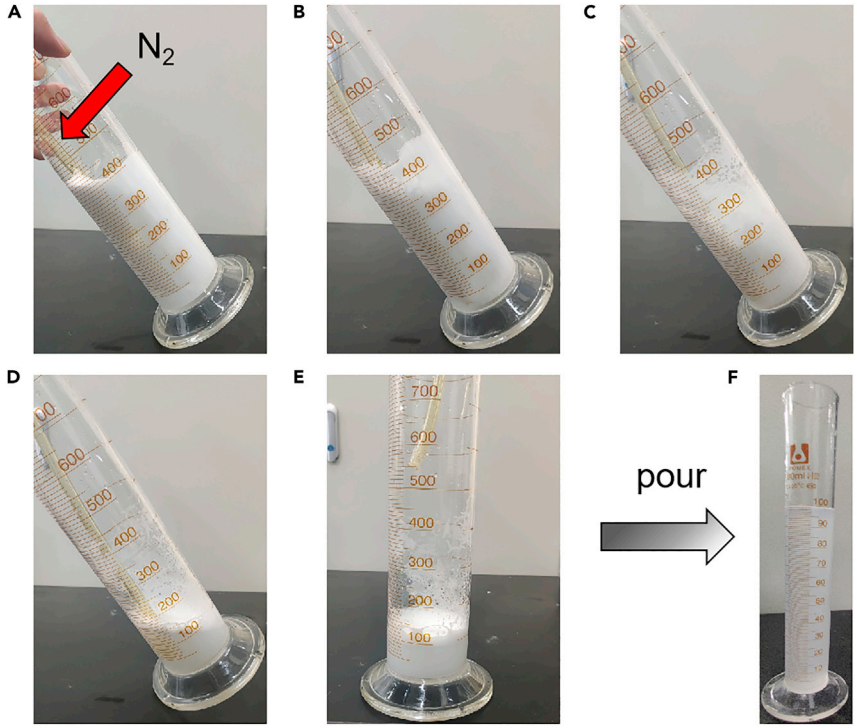

Figure 3. Defoaming experiment by injecting N_2

(A) Inject N_2 for 0 min.
(B) Inject N_2 for 2 min.
(C) Inject N_2 for 4 min.
(D) Inject N_2 for 6 min.
(E) Inject N_2 for 8 min.
(F) Precipitated liquid.

In this section, we describe the process of verifying the microscopic characteristics of the foam.

15. Pour the prepared $C_{12}A$ foam and $C_{12}A$-N20 foam into FoamScan's foam tube separately and observe the foam properties (Figure 4C).
16. Place the prepared $C_{12}A$ foam and $C_{12}A$-N20 foam on separate slides and observe the properties of the foam with a microscope (Figure 4A).

EXPECTED OUTCOMES

This protocol determines the formula of CO_2-sensitive foam 0.02 wt% $C_{12}A$ + 1.0 wt% N20. Using nanoparticles of different particle sizes compounded separately with $C_{12}A$, the performance of C_{12}A-N20 foam is much higher as in (Figure 2). Therefore, the nanoparticle N20 that best matched with $C_{12}A$ is screened. The results of infrared spectroscopy confirm the adsorption of $C_{12}A$ on the surface of nanoparticles N20. The foaming performance of the solution is controlled by CO_2 and N_2, and the foaming volume and half-life of the $C_{12}A$-N20 solution decrease only slightly after three cycles (Lv et al.).[7]

LIMITATIONS

There is a several limitation to this protocol. When surfactant $C_{12}A$ does not react sufficiently with CO_2, it will lead to the instability of foam. Therefore, when injecting CO_2 into the solution, it is better to stir while injecting so that the surfactant can fully react with CO_2.

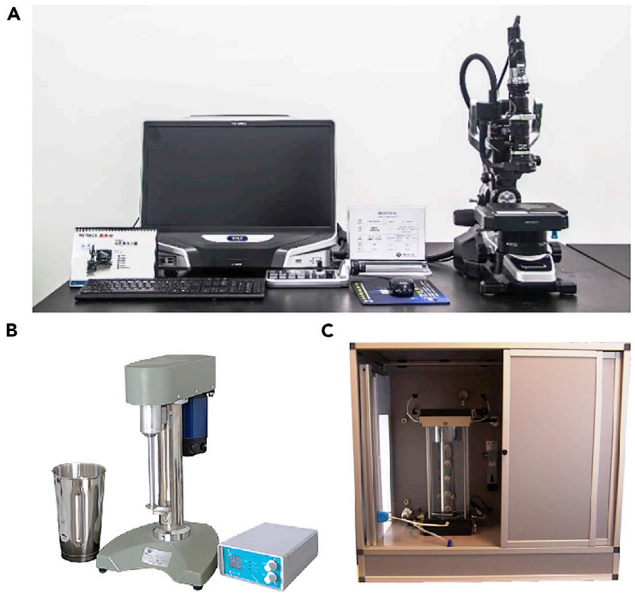

Figure 4. Equipment for evaluating the microscopic properties of foams
(A) Microscope.
(B) High-speed stirrer.
(C) FoamScan.

TROUBLESHOOTING

Problem 1

The foam may overflow from the measuring cylinder during defoaming experiments (step 12).

Potential solution

When injecting N_2 into the foam, it is better to tilt the container at 45° so that the CO_2 in the solution can be better discharged from the container.

Problem 2

In the defoaming experiment, the foam is not eliminated (step 12).

Potential solution

The defoaming process requires the passage of N_2 into the bottom of the foam.

Problem 3

After adding the surfactant to the aqueous solution of nanoparticles, it should not be left for too long. Otherwise, the nanoparticles will agglomerate and sink and the solution will stratify.[8]

Potential solution

We recommend stirring with a magnetic stirrer to make the solution homogeneous.

RESOURCE AVAILABILITY

Lead contact

Further information and requests for resources and reagents should be directed to and will be fulfilled by the lead contact, Kaiqiang Zhang (kaiqiang.zhang@pku.edu.cn).

Materials availability

This study did not generate any unique reagents.

Data and code availability

This study did not generate any datasets and code.

ACKNOWLEDGMENTS

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AUTHOR CONTRIBUTIONS

Conceptualization, S.Y.L.; methodology, S.Y.L., S.P.L.; investigation, S.P.L.; writing—original draft, S.P.L.; resources, S.Y.L.; funding acquisition, S.Y.L.; supervision, S.Y.L., K.Q.Z.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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