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SHORT COMMUNICATION

Synthesis and Performance Evaluation of CO_2/N_2 Switchable Tertiary Amine Gemini Surfactant

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Abstract A type of switchable tertiary amine Gemini surfactant, *N*,*N′*-di(*N*,*N*-dimethyl propylamine)-*N*,*N′*-didodecyl ethylenediamine, was synthesized by two substitution reactions with 3-chloro-1-(*N*,*N*-dimethyl) propylamine, bromododecane and ethylene diamine as main raw materials. The structure of the product was characterized by FTIR and ^{1}H-NMR. We also investigated the surface tension when CO_2 was bubbled in different concentrations of surfactant solution and the influence of different CO_2 volumes on surface tension under a constant surfactant concentration. Finally the surface tension curve and the related parameters were acquired by surface tension measurements. The experimental results showed that the structure of the synthesized compounds were in conformity with the expected structure of the surfactant, and displayed a better surface activity after bubbling CO_2. The critical micelle concentration (CMC) surface tension at CMC (γ_{cmc}) $p\text{C}_{20}$ (negative logarithm of the surfactant's molar concentration C_{20}, required to reduce the surface tension by 20 mN/m) surface excess (Γ_{max}) at air/solution interface and the minimum area per surfactant molecule at the air/solution interface (A_{min}) were determined. Results indicate that the target product had good surface activity after bubbling CO_2.

Keywords CO_2/N_2 switchable surfactant · Tertiary amine · Performance evaluation

Introduction

Traditional surfactants do not react chemically in use, but they are difficult to separate from the used solution, causing wasted product and potential for environmental damage. In recent years, with the development of green chemistry, traditional surfactants are being replaced by more environmentally friendly surfactants. Switchable surfactants can be reversibly changed between non-surfactant and surfactant behaviors in response to an external trigger such as pH [1, 2], temperature [1, 3], light [4], or a redox reagent [5]. CO_2/N_2 switchable surfactants can transform between non-surfactant and surfactant by controlling the addition and removal of CO_2. It is easy to separate the surfactant after CO_2 has been injected into the used solution. Research nto CO_2 switchable surfactants is becoming more and more popular because CO_2 is a cheap and widely available raw material.

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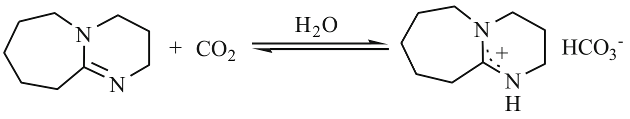
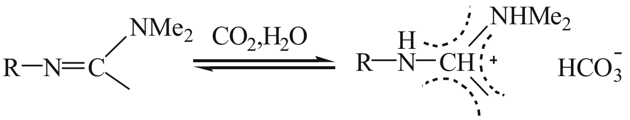


Scheme 1 The reaction of DBU with CO_2 and H_2O

In 1978, Iwatani et al. [6] reported that a white solid could be obtained by DBU (1,8-diazabicyclo [5.4.0] undec-7-ene) reacting with CO_2, but did not identify the chemical nature of the solid. In 2005, Jessop and co-workers [7] proved that a bicarbonate cationic complex could be obtained by the equimolar reaction of DBU, water and CO_2. The reaction is shown in Scheme 1. After the CO_2 was removed, the bicarbonate decomposed into the initial state. The same team [8] also applied the technique to the emulsification and demulsification process of both light crude oil and heavy oil. In 2009, Qinet et al. [9] reported that dodecyl tetramethyl guanidine could form a bicarbonate ionic adduct after being exposed to CO_2. It could be used as a CO_2 switchable surfactant; the principle is shown in Scheme 2. In 2014, Lu et al. [10] investigated the foaming performance of 2-alkyl-1-hydroxyethylimidazolinium bicarbonate cationic surfactants (HEAIBs) for the first time. They found that the surface activity of the surfactants was reversibly switchable; it could be shifted between ionic and neutral states by alternatively bubbling CO_2 and N_2. There are two types of CO_2 switchable surfactant, amidine base and guanidine base. Because of the high cost of amidine base switchable surfactant, its industry synthesis is limited. Because the bicarbonate of guanidine base switchable surfactant needs a higher decomposition temperature, its reversible process is very difficult, so it is rarely applied in industry.

From 2010, the researchers at home and abroad transferred their focus from amidine base and guanidine base switchable surfactants to a more moderate, reversible, efficient tertiary amine structure. In 2012, Zhao Yue [11] discovered that poly(*N*,*N*-dimethylaminoethyl methacrylate) (PDMAEMA) in water could react with carbon dioxide (CO_2), such that the system's lower critical solution temperature (LCST) could be reversibly tuned by passing CO_2 and argon (Ar) through the solution. In the same year, Han Dehui [12] reported CO_2-responsive ABA triblock copolymer hydrogels (the structure is shown in Scheme 3). Through rational block copolymer design, the hydrogels could undergo either CO_2-induced gel-to-sol or the reverse sol-to-gel transition. In 2012, Jessop et al. reported a kind of switchable solvent *N*,*N*-dimethyl cyclohexylamine ($CyMe_2$) [13] for oil-sand separation, and a kind of tertiary amine *N*,*N*-dimethyl ethanolamine (DMEA) [14], which could self-assemble with a traditional surfactant to form a switchable surfactant.



Scheme 2 The "on" and "off" of guanidine switchable surfactants

The studies of tertiary amine structure of polymers or micromolecules in the literature provides an idea for exploring tertiary amine CO_2 switchable surfactant.

In this paper, we introduce a kind of CO_2/N_2 switchable Gemini surfactant, and we describe the use of carbon dioxide (CO_2) as an efficient trigger to control either non-surfactant or surfactant transitions.

Experimental

Materials

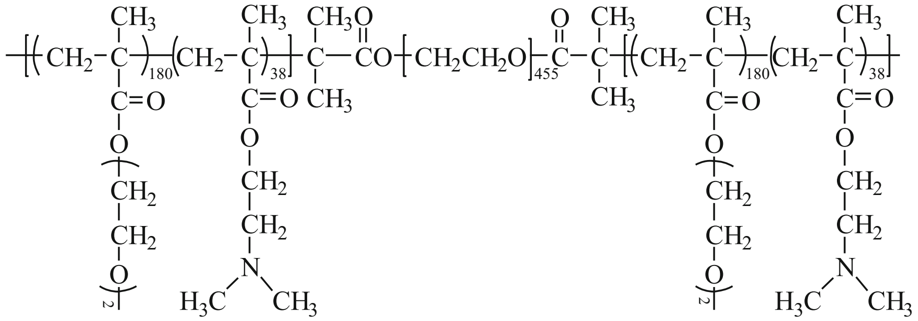
Ethylenediamine, ethanol, bromododecane and potassium iodide were purchased from Kelong Ltd., Chengdu, China. All chemical reagents were analytical grade. CO_2 (g) and N_2 were purchased from Jingli Gas Company, Chengdu, China. 3-Chloro-1-(*N*,*N*-dimethyl) propylamine was prepared in our laboratory. Distilled water was prepared in our laboratory by a model 1810-B quartz automatic dual distilled water apparatus.

Methods

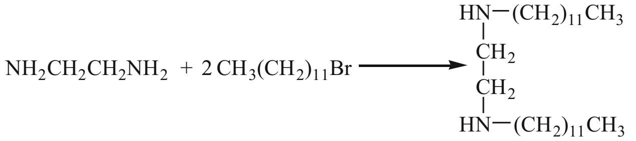
^{1}H-nuclear magnetic resonance (NMR) spectra were recorded on a Bruker (Bruker Corporation, Germany) AVANCE III 400 spectrometer (400 MHz) with D-DMSO solvent. Infrared (IR) spectra were obtained on a Nicolet 6700 spectrometer (Nicolet Corporation, USA). Surface tension was determined by using a JK99B automatic tensiometer at 20 °C in the Du Nöuyring method. Conductivity was obtained by using a DDS-11A conductivity meter (Sile Instrument Reagent Company, Shanghai, China). An OWC-9360 constant speed blender (Shenyang Institute of Aeronautical Engineering) was also used.

Synthesis

Ethylenediamine (0.045 mol) and ethanol (20 mL) were mixed and then added to a 250 mL three-necked flask at 70 °C. 1-bromododecane (0.1 mol) was dissolved in the ethanol solution, then was added slowly into the three-necked flask by constant pressure funnel, and the raw materials were constantly stirred for about 24 h (Scheme 4). Finally, the mixture was filtered while hot and the solid product was washed with ethanol three times. The solid product was dried at 50 °C for 24 h; the yield of *N*,*N*′-didodecyl



Scheme 3 The structure of CO_2-responsive ABA triblock copolymer hydrogels



Scheme 4 The synthesis of *N*,*N*′-didodecyl ethylenediamine

ethylenediamine was 69%. The structures were confirmed by FTIR and ^{1}H-NMR. The characteristic bands were located at 2920.73 cm^{-1} and 2855.93 cm^{-1} ($\upsilon_{C–H}$), and 1416.80 cm^{-1} ($\delta_{C–H}$). ^{1}H-NMR (D-DMSO, TMS, 50 °C): δ 0.865 (t, 6H, CH_3C), 1.240 (m, 40H, $–(CH_2)_{10}–$), 1.574 (m, 2H, NH), 2.496 (m, 4H, CH_2N), 2.919 (m, 4H, NCH_2CH_2N).

In the second step, *N*,*N*′-didodecyl ethylenediamine (0.01 mol) was dissolved in 50 mL ethanol, and then added into a 250 mL three-necked flask at 80 °C. 3-Chloro-1-(*N*,*N*-dimethyl) propylamine (0.022 mol) and potassium iodide (0.022 mol) were dissolved in ethanol. This solution was added slowly into the three-necked flask by a constant pressure funnel, and the mixture was constantly stirred for about 24 h (Scheme 5). After the reaction, the mixture was cooled to room temperature. The solvent was removed by vacuum distillation and the solid product was washed three times with ethanol. Finally, the products were obtained by drying at 50 °C for 24 h. The yield was 54% for the final product *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine. The structures were confirmed by FTIR and ^{1}H-NMR. The characteristic bands were located at 2917.14 cm^{-1} and 2843.59 cm^{-1} ($\upsilon_{C–H}$), 1414.63 cm^{-1} ($\delta_{C–H}$), and 1330.64 cm^{-1} ($\upsilon_{C–N}$). ^{1}H-NMR (D-DMSO, TMS, 50 °C): δ 0.897 (t, 6H, CH_3C), 1.273 (s, 4H, CCH_2C_8CC), 1.25 (d, 32H, $CC(CH_2)_8CCN$), 1.377 (s, 4H, $C_{10}CH_2CN$), 1.482 (s, 4H, $NCCH_2CN$), 2.159 (t, 12H, $C_3N(CH_3)_2$), and 2.341–2.765 ppm (t, 16H, CCH_2N, NCH_2CH_2N, $CH_2N(C)_2$).

Measurements

Surface Tension

Surface Tension Measurements

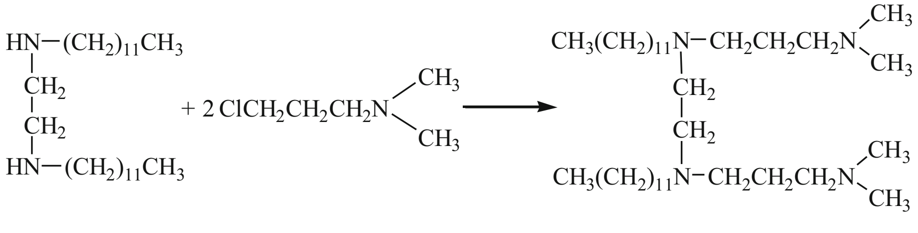
The tensiometer was adjusted until the surface tension of distilled water/air was measured as 71.0 mN/m. The surface tension of the sample solution was determined, in turn, from high to low concentrations.

According to the critical micelle concentration (CMC), γ_{cmc} and γ-logC curves, the other important properties of surfactants, such as pC_{20}, Γ_{max} and A_{min}, can be obtained by calculation or linear fitting.

Γ_{max} is the saturation adsorption of surfactant molecules in mol/1000 m^2 at the gas–liquid interface, which is obtained here by the Gibbs isothermal adsorption equation.

$$\Gamma_{\max} = \frac{-1}{nRT}\frac{d\gamma}{d\ln C}, \tag{1}$$

where γ is the equilibrium surface tension in mN m^{-1}, *R* is the gas constant [8.314 J/(mol K)], *T* is the absolute temperature, *C* is the surfactant concentration, and (dγ/dlnC) is the slope in the surface tension isotherm when the concentration is near the CMC. The value of *n* (the number of species at the interface whose concentrations change with the surfactant concentration) is taken as 1 for an equimolar ratio of surfactant anion and counteraction at interface, in the absence of extra electrolyte. In order to extract the



Scheme 5 The synthesis of *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine

minimum surface area occupied by a surfactant molecule, $A_{\min}$ (nm^2), at the air–water interface when the surface adsorption was saturated [15], Eq. (2) was used,

$$A_{\min} = -\frac{1}{N_A \Gamma_{\max}} \tag{2}$$

where N_A is Avogadro's constant (6.02×10^{23} mol^{-1}). $A_{\min}$ is in nm^2.

pC_{20} is the efficiency of surfactant, which can be characterized by the ogarithm of the surfactant concentration C_{20} at which the surface tension of water is reduced by 20 mN m^{-1}. The pC_{20} ($pC_{20} = -\log C_{20}$) value measures the efficiency of aggregation of surfactant at the air–water interface; the larger the value of pC_{20} , the greater the tendency of the surfactant to adsorb at the air–water interface and to form micelles, and the more efficiently it reduces the surface tension [16]. Equation (3) was used to obtain pC_{20},

$$pC_{20} = \frac{\gamma_0 - \gamma_{\mathrm{cmc}} - 20}{2.303 nRT\Gamma_{\max}} - \log \mathrm{CMC} \tag{3}$$

where γ_0 is surface tension of pure water at 298.15 K, the value is 71.27 mN m^{-1}. γ_{cmc} is the surface tension at the CMC in mN m^{-1}.

Foaming Properties

In our experiment, the blender method was used to test foaming ability of the test material. 200 mL of sample solution was added into a measuring cup. The concentrations of surfactants in the foaming study were 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 wt%. Foam was generated by high-speed (4000 rpm) stirring of the solutions for 60 s using a Waring Blender at 25 °C [16]. The time required for 100 mL liquid phase to form was recorded as $t_{1/2}$, which is the drainage half-life of the bubble.

Conductivity Measurements

In this paper, the conductivities of test material solutions were measured with a DDS-11A conductometer. During the experiment, the temperature of the aqueous solution was kept constant at 25.0 ± 0.1 °C. The concentration of the switchable surfactant was 0.01 mol L^{-1}. CO_2 gas was bubbled through the solution for 80 min, followed by N_2 for 80 min [17].

Results and Discussion

Surface Tension

The curve of surface tension vs concentration (on a log scale) in water/air are illustrated in Fig. 1. The critical micelle concentration (CMC) of the surfactant could be derived from the inflection point of the curve in the graph. The corresponding surface tension of the surfactant was γ_{cmc}.

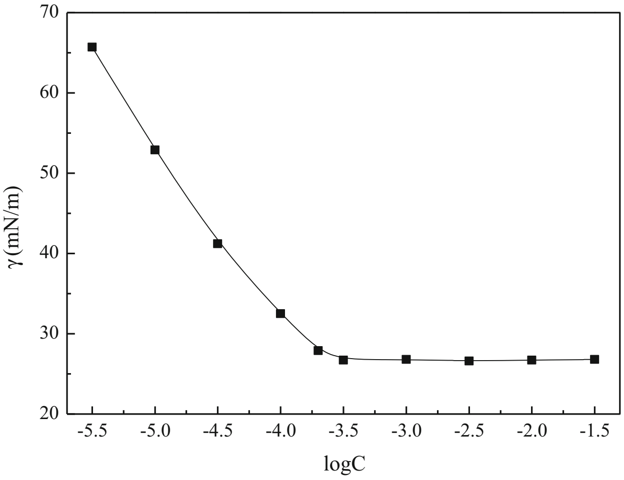


Fig. 1 The *curve* of γ-logC of C_{12}MAEd

Table 1 CMC and γ_{cmc}

Surfactant	CMC (mol/L)	γ_{cmc} (mN/m)
C_{12}MAEd	3.76×10^{-4}	27.8
EBAIBs [18]	1.43×10^{-3}	27.1
$C_{12}H_{25}N(CH_3)_3Br$	1.6×10^{-2}	40
$C_{12}H_{25}SO_3Na$	9.8×10^{-3}	38

Table 2 Surface property parameters of the product

Surfactant	PC_{20}	$A_{\min}$ (nm^2)	$\Gamma_{\max} \times 10^{10}$ (mol/cm^{-2})
$C_{36}H_{78}N_4$	5.01	0.787	2.11

Values of CMC and γ_{cmc} of *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine are listed in Table 1. The γ_{cmc} of *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine is between the traditional single chain cationic surfactant and the traditional Gemini cationic surfactant. It has a similar γ_{cmc} value and a lower CMC value asthe switchable Gemini surfactant, EBAIBs [18].

In Table 2 we see that, compared with a traditional single chain cationic surfactant with the same number of carbon atoms, the synthesized Gemini surfactant had better surface active parameters, pC_{20} value slightly increased, $A_{\min}$ value decreased and $\Gamma_{\max}$ value became larger. This indicates that the synthesized surfactant had strong adsorption on gas–liquid interface and high efficiency in reducing surface tension.

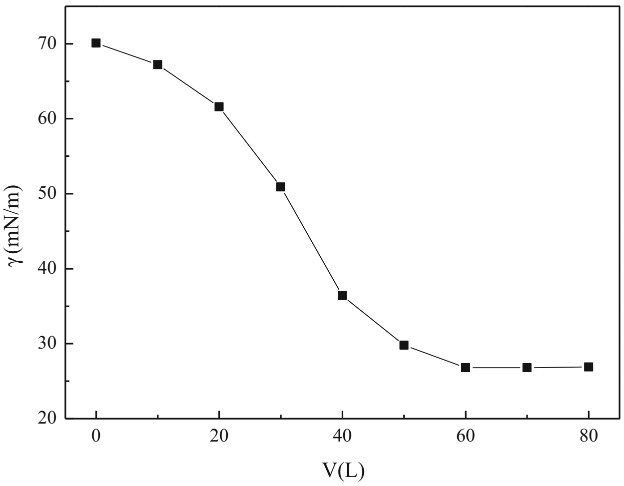


Fig. 2 The *curve* of γ-volume of CO_2

In order to investigate the influence of different CO_2 volumes on surface tension under a constant concentration of surfactant, 10 L of CO_2 gas were bubbled into a 0.01 mol/L concentration of surfactant solution every time for 15 min to make sure a complete reaction of CO_2 and the solution. The curve of surface tension vs the volume of CO_2 is shown in Fig. 2.

Because the switchable surfactant had a very low solubility without bubbling CO_2, the surface tension of the switchable surfactant solution/air is almost the same as that of pure water/air. When the volume of CO_2 bubbled was about 20 L, the surface tension underwent a big change, and after the volume of bubbled CO_2 was about 60 L, surface tension stopped changing. That indicated that the tertiary amine structure transformation to bicarbonate had been completed, and that the transformation of non-surfactant to surfactant had been completed.

CO_2/N_2 Switchable Properties

Reversibility of Conductivity

The initial conductivity of *N*,*N*'-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine was measured in 0.01 mol L^{-1} solution at 25 °C. After that, the CO_2 gas was bubbled at the speed of 1 L/min and the conductivity was recorded every 5 min. When the reaction in the system was in balance and the conductivity value was leveling off, N_2 continued to be bubbled into the solution at the speed of 1 L/min and *r*, the value of conductivity, was recorded until the conductivity reached a minimum value [9]. The experiment was run in triplicate. The curve of conductivity is shown in Fig. 3.

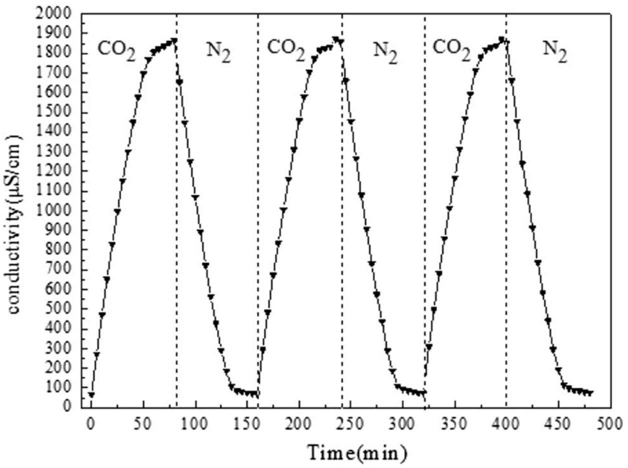


Fig. 3 The conductivity of surfactant solution

From Fig. 3 we see that the initial state of the solution conductivity values was slightly higher than zero. As the CO_2 was continuously bubbled into the solution, *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine reacted with CO_2 to generate *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine bicarbonate (quaternary ammonium cationic surfactant, complete dissociation). The conductivity increased rapidly until the maximum value was attained, but with bubbling N_2 gas into the system, the bicarbonate was decomposed to the initial state and became neutral non-ionic compounds. The conductivity in solution then decreased rapidly and gradually returned to its initial value. The repeated conductivity experiments confirmed that the synthesis of surfactant was CO_2-responsive, that is, the surfactant was CO_2-triggered and switchable.

Reversibility of Foam Property

There was no foam observed before the CO_2 was bubbled into these solutions. When the CO_2 was bubbled into the solutions and rotated for 60 s in the blister apparatus, foam was produced. After the foam disappeared completely, N_2 was introduced into the solutions and rotated for 60 s in the blister apparatus; no foam was observed in the cylinders. Results are shown in Fig. 4.

With the increase of surfactant concentration, foam volume increased. When the concentration reached a certain value, foam volume achieved a maximum value. When we continued to increase the concentration of surfactant, the foam volume no longer increased and gradually stabilized. The maximum foam volume observed was 650 mL. The likely explanation for this phenomenon could be that as the surfactant concentration in the solution was first increased, there was a greater tendency to decrease the

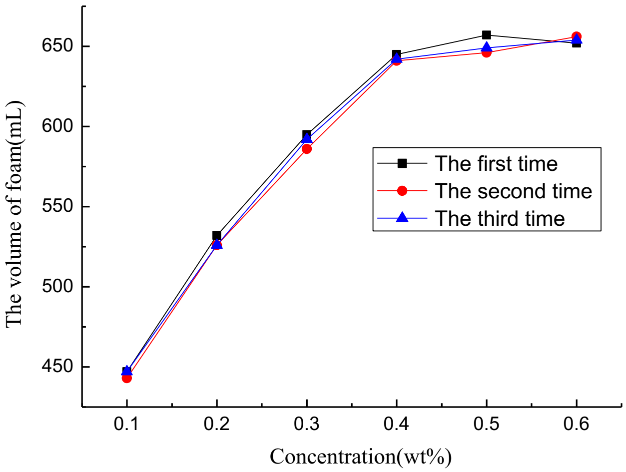


Fig. 4 Variation of foam volume with surfactant concentration after bubbling CO_2

solution surface tension, and the foam volume increased. When the surfactant concentration increased to a certain value, the surface tension of the solution remained stable and the foam volume gradually achieved balance.

Conclusion

By substitution reaction, *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine was prepared with 3-chloro-1-(*N*,*N*-dimethyl) propylamine, bromododecane and ethylenediamine as raw materials. It was confirmed that *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-N,*N*′-didodecyl ethylenediamine and CO_2 could form a bicarbonate complex which possessed the ability to being used as a CO_2/N_2 switchable surfactant. The chemical structures of the prepared compounds were confirmed by FTIR and ^{1}H-NMR. The critical micelle concentrations (CMC) of the bicarbonate complexes in aqueous solution at 20 °C was determined with surface tension methods. This indicated that *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine had reversible surface tension when CO_2 and N_2 were cyclically bubbled into the solution. A conductivity reversibility experiment confirmed that the *N*,*N*′-di(*N*,*N*-dimethyl propylamine)-*N*,*N*′-didodecyl ethylenediamine had CO_2/N_2 switchable performance. After further optimization of the synthesis process, applications for the tertiary amine CO_2/N_2-switchable surfactant will be promising.

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