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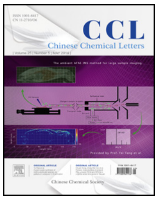

Original article

Phase-transfer catalysis of a new cationic gemini surfactant with ester groups for nucleophilic substitution reaction

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ABSTRACT

A highly effective phase transfer of a quaternary ammonium gemini surfactant with ester groups ((diethylhexanedioate) diyl-α,ω-bis(dimethyl dodecyl ammonium bromide) referred to as **12-10-12**) was synthesized with high yield and characterized by infrared spectroscopy, elemental analysis and ^{1}H-NMR. Then, **12-10-12** was used as a phase transfer catalyst to study the catalytic effect on the reaction of anhydrous sodium acetate and 4-methylbenzyl chloride. The possible catalytic mechanism and the influence of surfactant concentration, temperature and type are also discussed. The experimental results showed that the catalysis efficiency was more active than the traditional, single-chained surfactant, tetrabutyl ammonium bromide. It also revealed that the reaction was first-order with respect to the concentration of 4-methylbenzyl chloride. The concentration of 4-methylbenzyl chloride grew linearly with the concentration of **12-10-12** and as the reaction temperature increased. The optimum reaction time was 7 h.

1. Introduction

Rapid development in the field of catalysis has directed increasing attention on the catalyst. Phase-transfer catalysis and micellar phase-transfer catalysis are two well-known methods of promoting organic reactions, but surfactants are among the most important catalysts. Traditional single-chained surfactants have been widely studied [1–3]. In 1991, Menger *et al.* synthesized, for the first time, reported on a series of three, two-chained surfactants with lower critical micell concentration (CMC) than traditional, single-chained surfactants and named them gemini surfactants. In recent years, more and more investigators are studying these new structural surfactants and their phase-transfer catalytic effects [4–11]. However, a few researchers have studied the micellar catalysis of gemini surfactants [12–17]. For example, Qiu *et al.* [13] studied micellar-catalyzed alkaline hydrolysis of 2,4-dinitrochlorobenzene (DNCB) in the presence of the cationic gemini surfactant, 1,2-ethane bis(dimethyldodecyl ammonium bromide), and found that this system had a relatively low second-order rate constant in micellar pseudophase and a great deviation from the pseudophase ion-exchange model at high concentration. Also some studies examined aqueous/organic two-phase reactions [18,19] or focused on studying their elemental characteristics, for example, since they can be more easily adsorbed in the interface between water and air at lower critical micelle concentrations [4], provide greater efficiency in reducing the surface tension and lowering the Krafft temperature, and better solubilization in comparison with conventional surfactants [20–24].

Until now, it was reported [1,25–28] that extensive micellar catalytic reactions were generally carried out in solution with conventional monomeric surfactants. However, still few studies involved liquid-solid catalysis effects on inorganic/organic reactions using monomeric surfactants, let alone gemini surfactants.

In this paper, a new gemini surfactant connecting two alkyldimethylammonium bromide moieties, namely **12-10-12**, was synthesized and mainly applied to catalysis reactions. Two synthetic steps were designed to form the final product which had a flexible spacer containing ten carbon atoms and two alkyl chains with twelve carbon atoms (Scheme 1). Moreover, the catalysis effect on the reaction of anhydrous sodium acetate and 4-methylbenzyl chloride was also investigated, in contrast to the traditional monomeric surfactants. The results show that **12-10-12** possesses better catalysis ability. Based on the related knowledge,

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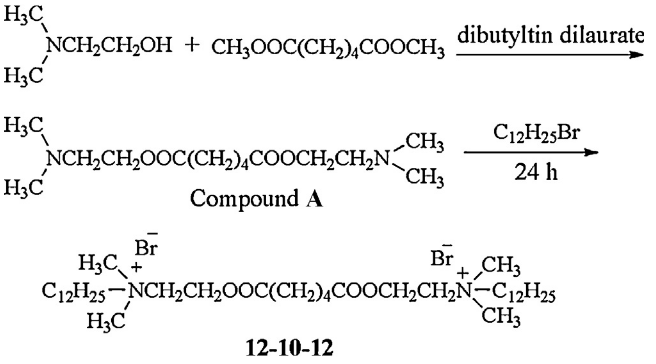

Scheme 1. Synthetic route for the gemini surfactant **12-10-12**.

a theoretical model was presented to explain why this gemini surfactant could promote liquid-solid reactions.

2. Experimental

2.1. Materials and methods

Chemicals, reagents and solvents from commercial sources are of analytical or spectroscopy grade and used as received without further purification. ^{1}H NMR spectra were recorded on an AVANCE-400 NMR spectrometer. Elemental analyses were carried out on an Elementar Vario EL-•. FT-IR spectra were measured on a Nicolet NEXUS 870 spectrometer (KBr discs). Thermogravimetry analyses (TGA) were carried out using a Pyris1 TGA (PE Corp., USA) at a heating rate of 10 °C/min from 40 °C to 600 °C under a nitrogen atmosphere.

2.2. Synthesis and characterization

Synthesis of compound A: Compound A was obtained by refluxing dimethyl hexanedioate (8.70 g, 50.00 mmol) with dimethylaminoethanol (26.70 g, 300.00 mmol) in the presence of 1.50 g dibutyltin dilaurate catalyst at 100 °C for 6 h (Scheme 1). The by-products of methanol and residual dimethylaminoethanol were removed by vacuum from the reaction mixture and the crude product was purified by column chromatography. Yield: 9.00 g (62.50%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.66–1.73 (m, 4H, CCH_2CH_2C), 2.33–2.56 (m, 4H, $2 \times COCH_2$), 3.48 (s, 12H, $4 \times CH_3$), 4.14–4.17 (t, 4H, $J = 7.2$ Hz, $2 \times NCH_2$), 4.46–4.72 (m, 4H, OCH_2). IR (KBr, cm^{-1}): 2947, 2822, 1736, 1460, 1383, 1172, 1041 [15].

Synthesis of **12-12-12**: A solution of compound A (5.00 g, 17.40 mmol) and n-$C_{12}H_{25}Br$ (13.00 g, 52.50 mmol) in acetone was refluxed for 24 h. The precipitate was recrystallized from acetone to afford white crystals 12.50 g. Yield: 91.60%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.86–0.88 (t, 6H, $J = 6.8$ Hz, $2 \times CH_3$), 1.25–1.36 (m, 36H, $2 \times (CH_2)_9$), 1.71–1.77 (m, 8H, $2 \times CH_2$, CCH_2CH_2C), 2.33–2.56 (m, 4H, $2 \times CH_2COO$), 3.48 (s, 12H, $2 \times (CH_3)_2$), 3.62–3.66 (t, 4H, $J = 7.6$ Hz, $2 \times 2CH_2$), 4.14–4.17 (t, 4H, $J = 7.2$ Hz, $2 \times CH_2N^+$), 4.47–4.73 (m, 4H, $2 \times CH_2$) [29]. Anal. calcd. for $C_{38}H_{78}Br_2N_2O_4$: C, 57.98; H, 9.92; N, 3.56. Found: C, 57.37; H, 9.94; N, 3.53. IR (KBr, cm^{-1}): 2921, 2851, 1723, 1472, 1382, 1274, 1153.

2.3. Kinetic measurements

Anhydrous sodium acetate, 3.50 g (42.70 mmol, 0.9163 mmol/L), 4-methylbenzyl chloride 5.00 g (35.60 mmol, 0.764 mol/L) and a certain amount of **12-10-12** were added to 40 mL DMF. The mixture was stirred at required speed and desired temperature. The reaction continued for 7 h. The samples were collected at regular intervals for analysis using Agilent 7820 Gas-Chromatograph equipped with a FID detector, and orthodichlorobenzene served as an internal standard. The 4-methylbenzyl acetate was extracted by benzene.

The kinetic equations are expressed as follows:

$$\begin{aligned} \text{rate} &= -\frac{d[C_r]}{dt} = k_{\text{obsd}}[C_r] = [C_r](k_1 k_2 [N^+] + k_0) \\ &= [C_r](2k_1 k_2 [C_{12}] + k_0) \end{aligned}$$

$$[N^+] = 2[C_{12}].k_{\text{obsd}} = 2k_1 k_2 [C_{12}] + k_0$$

where k_{obsd} is the apparent first-order rate constant for the reaction, $[C_r]$ is the concentration of 4-methylbenzyl chloride, $[C_{12}]$ is the concentration of **12-10-12**, k_0 is the first-order rate constant without phase transfer catalyst, k_1 is the equilibrium constant and k_2 is the second-order-rate constant with the phase transfer catalyst, respectively. The above equations suggest: (1) The reaction is first-order with respect to the concentration of 4-methylbenzyl chloride and the plots of ln$[C_r]$ *vs.* time should be linear. (2) k_{obsd} increases with the concentration of phase transfer catalyst and the plots of k_{obsd} *vs.* $[C_{12}]$ are linear lines.

3. Results and discussion

3.1. Thermal analysis

The TGA measurement of **12-10-12** was determined in the range of 20–600 °C under nitrogen (Fig. 1). In order to study the thermal stability of this compound, thermogravimetric analysis (TGA) was performed on single crystal samples. TGA data show that **12–10-12** is stable up to 205 °C, loses weight from 205 °C to 320 °C corresponding to decomposition of double alkyl chains, and finally loses weight from 320 °C to 410 °C corresponding to the losses of the flexible spacer.

3.2. The catalytic mechanisms of gemini surfactant

According to previous knowledge [13,24], a model to explain the liquid–solid phase transfer catalysis reaction mechanism has been proposed for the nucleophilic substitution reaction of anhydrous sodium acetate and 4-methylbenzyl chloride with the phase transfer catalyst of **12-10-12** (Scheme 2). It is noted that the solubility of anhydrous sodium acetate in DMF is poor at 60 °C.

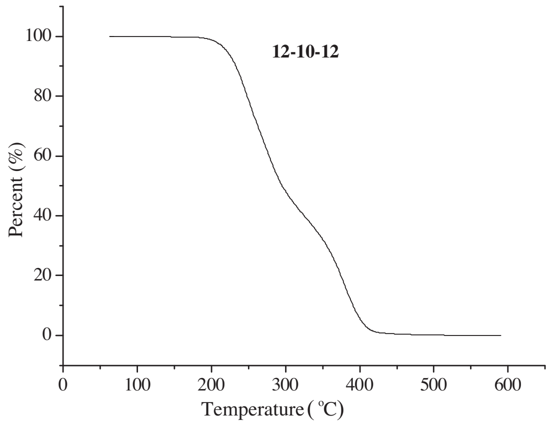

Fig. 1. TGA curve of **12-10-12**.

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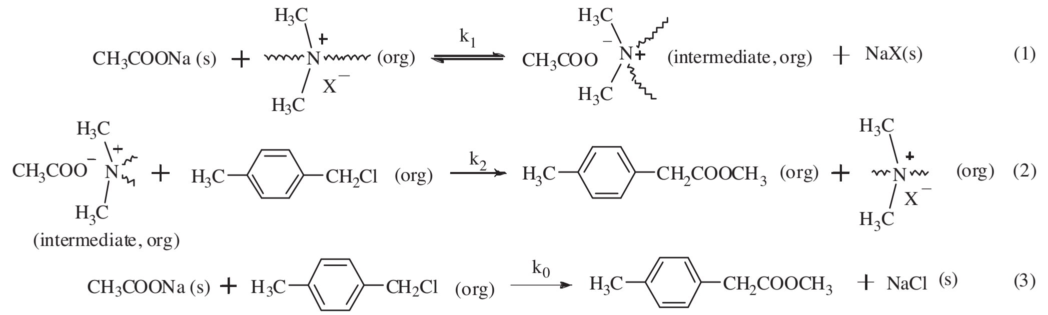

Scheme 2. Model of solid–liquid phase transfer catalysis reaction mechanism (k_0 is the first-order rate constant without phase transfer catalyst, k_1 is the equilibrium constant and k_2 is the second-order-rate constant with the phase transfer catalyst, respectively).

Some kinetic and thermodynamic parameters to better understand the catalytic mechanism were obtained. The reaction of 4-methylbenzyl chloride and anhydrous sodium acetate with the participation of **12-10-12** can be supposed to take place in the liquid–solid interface.

As represented by the reaction mechanism of the micellar catalysis in Scheme 2, the hydrophilic group of gemini surfactant initially unites with CH_3COO^- group, and then reacts with 4-methylbenzyl chloride through the nucleophilic substitution reaction. While the increasing of aggregation number and the micelle size may significantly influence the reaction rate between the reactants at the micelle surface region. So when the concentration of **12-10-12** approaches the value of CMC, the molecules of the gemini surfactant began to form micelles (molecular agglomerates), and the number of micelles will increase with the concentration of **12-10-12**. By this stage, the cationic gemini surfactants can bind with more and more CH_3COO^- groups at the micelle surface region. This is to the benefit of the process of the nucleophilic substitution reaction, and thus leads to the increasing of the reaction rate.

3.3. Influence of surfactant concentration

Firstly, in order to investigate the effect of surfactant concentration for the apparent-first-order rate constants (k_{obsd}), a series of nucleophilic reactions was carried out in the presence of **12-10-12** with various concentrations at 50 °C. As shown by the $-\ln C_r$ *vs.* time characteristics in Fig. 2, all the kinetic results fit the first-order rate equation very well: $\ln(1/C_r) = k_{obsd}\ t + A$.

Table 1
Catalysis parameters of gemini surfactant at various concentrations at 50 °C.

C_{12} (mol/L)	K_{obsd}	Linear correlation coefficients
0	0.071	0.999
0.004	0.14	0.999
0.008	0.17	0.999
0.012	0.21	0.999
0.016	0.24	0.999
0.020	0.30	0.999

The apparent first-order rate constants (k_{obsd}) and linear correlation coefficients were evaluated from the linear plots of $\ln(1/C_r)$ *vs.* time. The data are summarized in Table 1. Clearly, the values of linear correlation coefficients were close to one, which indicated that the association between $\ln(1/C_r)$ and reaction time is coincident with a linear relationship and the experimental results were reliable. Then, Fig. 3 was obtained by using the values of the concentrations of **12-10-12** (C_{12}) and the apparent first-order rate constants (k_{obsd}) in Table 1 as the X and Y axes, respectively. The relationship between the apparent first-order rate constants (k_{obsd}) and the concentrations of **12-10-12** (C_{12}) was also close to a linear relationship, and its slope and intercept were estimated to be 10.6 and 0.071, respectively. Finally, the linear equation between the surfactant concentration (C_{12}) and the apparent-first-order rate constants (k_{obsd}) is deduced as follows:

$$k_{obsd} = 10.6[C_{12}] + 0.071$$

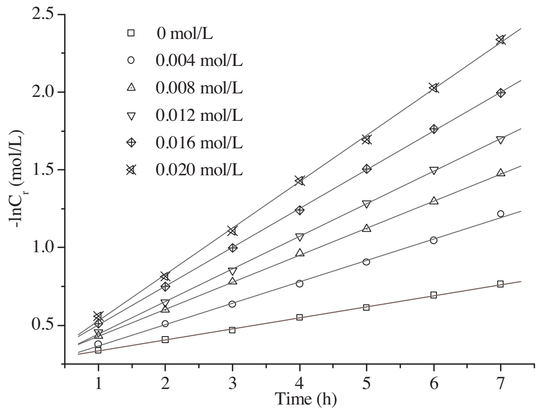

Fig. 2. Plot of $-\ln C_r$ *vs.* time for the phase transfer catalysis by **12-10-12** at 50 °C. [4-Methylbenzyl chloride] = 0.764 mol/L, [sodium acetate] = 0.9163 mol/L.

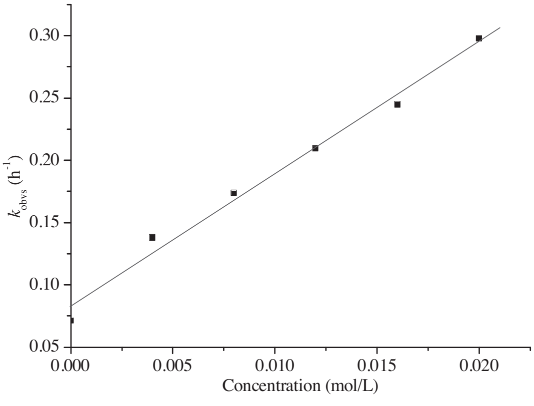

Fig. 3. Plot of k_{obsd} *vs.* concentration of **12-10-12** for the phase transfer catalysis by **12-10-12** at 50 °C. [4-Methylbenzyl chloride] = 0.764 mol/L, [sodium acetate] = 0.9163 mol/L.

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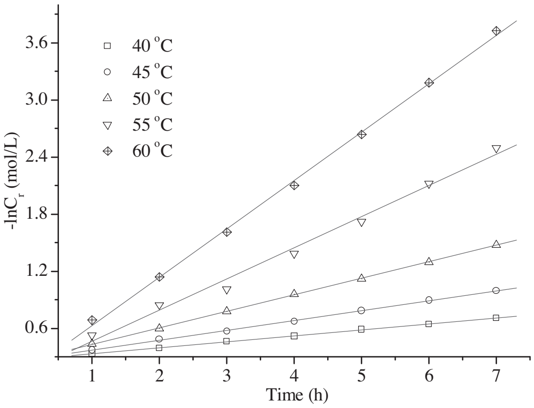

Fig. 4. Plot of $-\ln C_r$ *vs.* time for phase transfer catalysis by **12-10-12** at different temperatures. [4-Methylbenzyl chloride] = 0.764 mol/L, [sodium acetate] = 0.9163 mol/L.

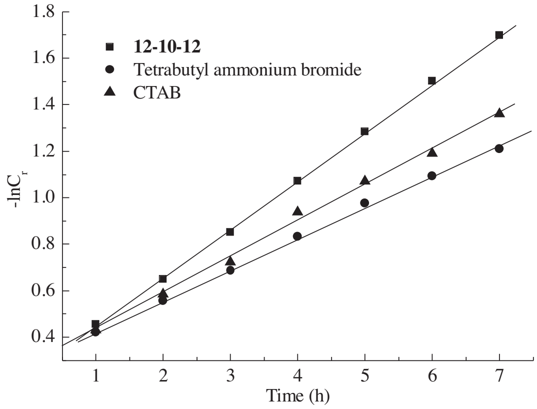

Fig. 6. Plot of $-\ln C_r$ *vs.* time for phase transfer catalysis by **12-10-12**, tetrabutyl ammonium bromide and CTAB, respectively. [4-Methylbenzyl chloride] = 0.764 mol/L, [sodium acetate] = 0.9163 mol/L, [**12-10-12**] = 0.012 mol/L, [tetrabutyl ammonium bromide] = 0.012 mol/L, [CTAB] = 0.012 mol/L.

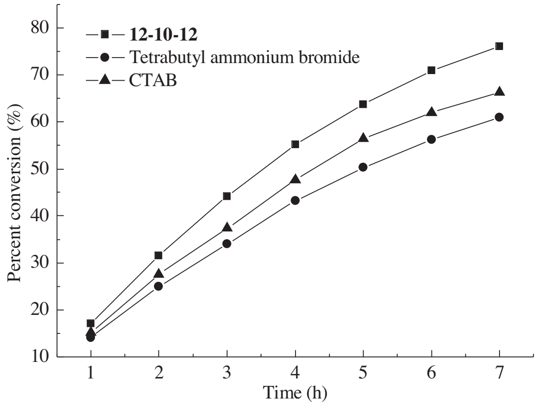

Fig. 7. Plot of percent conversion *vs.* time for phase transfer catalysis by **12-10-12** and tetra butyl ammonium bromide, respectively. [4-Methylbenzyl chloride] = 0.764 mol/L, [sodium acetate] = 0.9163 mol/L, [**12-10-12**] = 0.012 mol/L, [tetrabutyl ammonium bromide] = 0.012 mol/L, [CTAB] = 0.012 mol/L.

In addition, the effect of the concentration of 4-methylbenzyl chloride on the reaction rate was also studied, and a series of nucleophilic reactions was carried out in the presence of **12-10-12** at fixed concentrations at 50 °C. As shown in Fig. S1 (Supporting information), although the concentration of 4-methylbenzyl chloride is different, the ratios between the values of $-\ln C_r$ and reaction time (h) are almost same, which indicates the concentration of 4-methylbenzyl chloride can influence the reaction rate, and it does not impact the apparent first-order rate constants (k_{obsd}).

3.4. Influence of reaction temperature

The nucleophilic substitution reaction was carried out at various temperatures (at 40, 45, 50, 55 and 60 °C) when the concentration of **12-10-12** was 8.00 mmol/L (Fig. 4). The influence of temperature on $-\ln C_r$ was studied according to the Arrhenius equation: $\ln k_{\text{obsd}} = -E_a/RT + \ln A$. Fig. 5 gave the plot of $\ln k_{\text{obsd}}$ *vs.* 1/*T*. The apparent activation energy was 93.11 kJ/mol obtained by the slope.

3.5. Influence of different surfactants

The nucleophilic substitution reaction was studied at 50 °C when the concentration of surfactants was 12.00 mmol/L. The surfactants were **12-10-12**, tetrabutyl ammonium bromide and hexadecyl trimethyl ammonium bromide (CTAB). It is evident from Figs. 6 and 7 that the catalysis kinetic model of **12-10-12** was similar to that of conventional quaternary ammonium salt catalyst and the catalytic activity of **12-10-12** was more efficient than tetrabutyl ammonium bromide and CTAB at the same concentration. The reaction rate with **12-10-12** was greater than those of tetrabutyl ammonium bromide and CTAB, which can be explained in view of unique molecular structure of the gemini surfactant with two catalyst active sites (active site is meant to be the catalyst cation N^+) in one molecule.

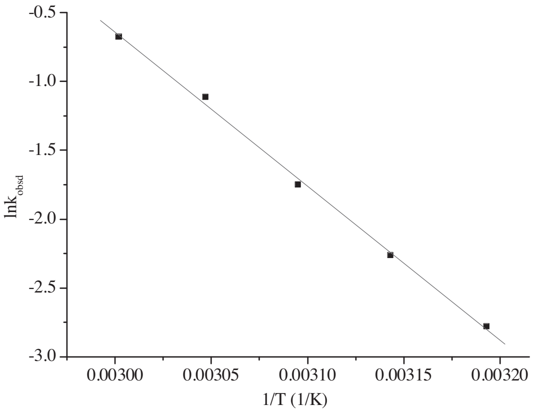

Fig. 5. Plot of $\ln k_{\text{obsd}}$ *vs.* 1/T for phase transfer catalysis by **12-10-12**. [4-Methylbenzyl chloride] = 0.764 mol/L, [sodium acetate] = 0.9163 mol/L.

4. Conclusion

In summary, a gemini surfactant (**12-10-12)** with ester groups was conveniently synthesized and an investigation of the liquid-solid phase transfer reaction of anhydrous sodium acetate and 4-methylbenzyl chloride in the presence of **12-10-12** has been studied. It was found that the phase transfer catalyst of gemini surfactant (**12-10-12**) was more effective than the traditional surfactant phase transfer catalyst (such as tetrabutyl ammonium bromide). A suitable kinetic model was proposed to explain the observed experimental results and it was determined that the experimental results fit the model very well.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at http://dx.doi.org/10.1016/j.cclet.2014.04.006.

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