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ORIGINAL ARTICLE

Effects of pH, Temperature and Ionic Strength on Aggregation Behavior of Dodecyldiethoxylamine Oxide

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Abstract The isoelectric point of dodecyldiethoxylamine oxide (DDEAO) containing hydroxyethyl groups was determined with the method of acid–base titration. The isoelectric point value of DDEAO is 7.8. The effects of pH, temperature and ionic strength on the aggregation behavior of DDEAO were investigated by surface tension measurements. The results show a good surface activity over a large pH range, especially under weakly acidic conditions. The surface activity of DDEAO increases trend increasing temperature. The thermodynamic parameters were also evaluated and the results show that the micelle formation is entropy-driven within the temperature range investigated. CMC values decrease with increases in ionic strength. The packing parameters (*P*) were evaluated and ensure the formation of vesicles for DDEAO at various pH values, which is expected to have potential applications in some fields, such as in nanomaterials synthesis and the application in drug delivery.

Keywords Amine oxide · Aggregation behavior · Surface activity

Introduction

Amine oxide surfactants show nonionic characteristics in neutral or alkaline solutions, and exhibit a cationic behavior in acid solutions [1, 2]. They are a kind of mixture made up of protonated and nonprotonated species in aqueous media, and their composition depends on the pH of the aqueous solution [3]. These surfactants produce less irritation to the skin, have high foaming properties and an excellent thickening function [4–6]. Among amine oxides, dodecyldimethylamine oxide (DDMAO) is the most widely used surfactant and has been extensively researched [7–9]. Mixtures of DDMAO and sodium dodecylsulfate (SDS) or dodecyltrimethylammonnium bromide (DTAB) have been widely studied. Results reported have shown good synergistic effects of mixed solutions [10–12]. DDMAO has been used in detergents, shampoos, cosmetics and textile auxiliaries based on their characteristics and advantages [5, 6].

Many applications such as sewage treatment [13], preparation of nanostructured materials [14] and the application in drug delivery [15] depend on the aggregation behavior of surfactants. Therefore, there is a lot of research into the aggregation behavior of surfactants in the last few years. The aggregation behavior of amine oxide surfactants have been extensively researched employing surface tension, rheological measurements, small-angle neutron scattering, and static light scattering [10, 16–22]. The results indicate that the aggregation behavior of amine oxide surfactants can be controlled by altering the length of the hydrocarbon chain, the pH of the aqueous solution, ionic type, the media of solution, temperature and ionic strength. In spite of much research into the aggregation behavior of amine oxide surfactants, amine oxide surfactants containing hydroxyethyl groups have been studied less.

In this paper, dodecyldiethoxylamine oxide (DDEAO) containing hydroxyethyl groups was synthesized according to the literature. The synthesis route and chemical structure are shown in Scheme 1. The isoelectric point of DDEAO was measured. The effects of pH, temperature and ionic strength on the aggregation behavior of DDEAO were investigated. The various physicochemical properties and

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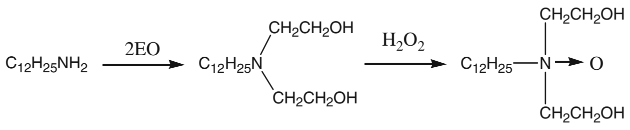

Scheme 1 Synthesis route of dodecyldiethoxylamine oxide

thermodynamic parameters were evaluated. The results were also compared with those of DDMAO.

Materials and Methods

Materials

Dodecyldiethoxylamine oxide (DDEAO) was prepared by the reaction of dodecyldiethoxylamine with hydrogen peroxide in water as the solvent in the laboratory based on the literature [23]. Water was removed by vacuum, and then the crude material was recrystallized three times from acetone to obtain purity. The pH value of DDEAO in aqueous solution was adjusted by NaOH or HCl aqueous solutions. NaCl was used to adjust the ionic strength of the aqueous solutions.

pH Titration

The isoelectric point (pI) of DDEAO was determined by a pHs-3c pH meter from Shanghai Precision and Scientific Instrument Co. using the method of acid–base titration.

Surface Tension Measurement

The surface tensions of DDEAO were measured from a series of aqueous solutions with a platinum ring using a Krüss K12 Tensiometer. The surface tension of double distilled water, 72.0 ± 0.3 mN/m, was used for calibration purposes. Surfactant solutions were prepared with the double-distilled water. The samples were stabilized for 10 min in the instrument before measurements were taken.

Results and Discussion

Effect of pH on the Aggregation Behavior of DDEAO

The pH titration curve of DDEAO is shown in Fig. 1. The isoelectric point (pI) is the titration jump point of the pH titration curve. As can be seen from Fig. 1, the pI value of DDEAO is 7.8. Amine oxide surfactant has been known to be mainly nonprotonated at high pH, and protonated at low pH [24]. DDEAO shows cationic behavior when the pH value is below 7.8, while it exhibits nonionic behavior the pH is greater than 7.8. Moreover, the pH of DDEAO is 6.0 in aqueous media. So DDEAO behaves similarly to cationic surfactants in aqueous solutions. It is expected to form micelles containing both protonated and nonprotonated species.

The surface activity of DDEAO were investigated by surface tension measurements with different pH values at 298 K. The critical micelle concentration (CMC) values and the surface tensions at CMC (γ_{CMC}) of DDEAO at various pH values were determined from the plots of surface tension (γ) versus surfactant concentration in Fig. 2. The results are presented in Table 1. CMC values versus pH plots are depicted in Fig. 3. As can be seen, CMC and γ_{CMC} values of DDEAO decrease with increasing pH down to a minimum and then increase upwards in the range of pH 2–10. The minimum of CMC is approximately at pH = 6 (below the pI value of DDEAO). This is probably due to the synergism between protonated and nonprotonated species in aqueous media. Since the amount of nonprotonated species increases as the pH value (pH < 6) increases in solution, the electrostatic repulsion between the hydrophilic groups is weakened, resulting in a decrease in CMC. However, CMC values have a slight increase in pH > 6. The result is caused by a weak interaction between hydrocarbon chains with a rise in pH. γ_{CMC} values show a similar trend to CMC. This can be explained by the fact that DDEAO shows different characteristic of surfactants at various pH values. It is noteworthy that DDEAO has lower CMC value than DDMAO (1.7 mM) in aqueous solution [10]. Moreover, in the case of DDMAO, CMC values of nonprotonated species are approximately 1 mM [9]. However, CMC values of DDEAO at high pH in this work are lower than those of DDMAO. It is well established that hydroxyethyl group of DDEAO is a dominant factor in the determination of CMC values. This result indicates that the

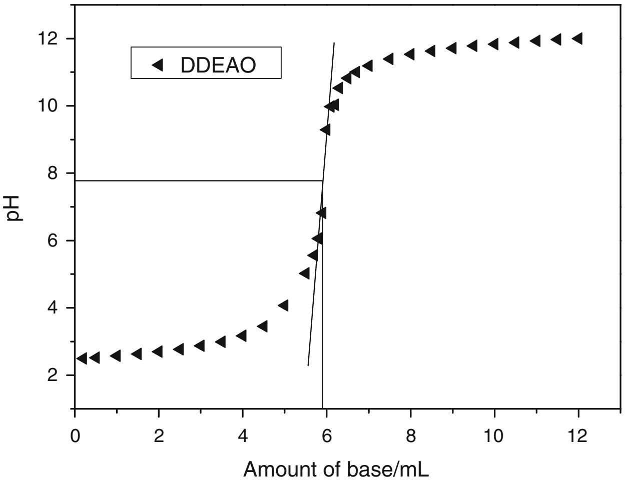

Fig. 1 The relationship between the base amount and pH

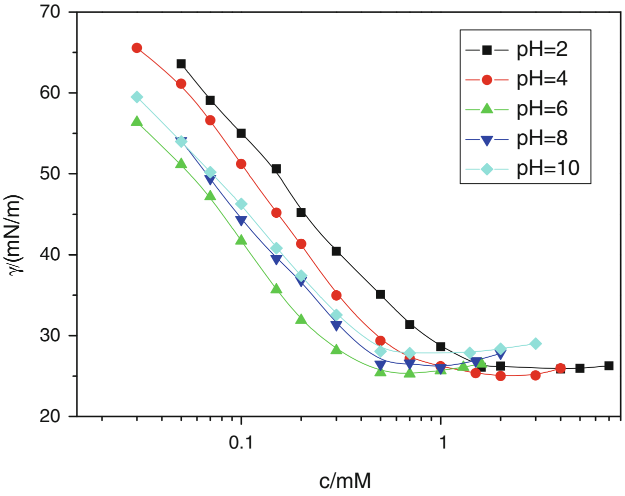

Fig. 2 Surface tension vs. concentration for DDEAO with different pH values at 298 K

Table 1 Surface properties of DDEAO with different pH values at 298 K

pH	CMC (mM)	γ_{CMC} (mN/m)	Γ_{max} (μmol/m^2)	A_{min} (Å^2)	pC$_{20}$	*P*
2	1.20	26.0	4.83	34.4	3.91	0.60
4	0.73	25.7	5.18	32.0	4.02	0.64
6	0.38	25.4	5.20	31.9	4.36	0.65
8	0.45	26.6	5.04	33.0	4.25	0.62
10	0.48	27.8	4.69	35.4	4.25	0.58

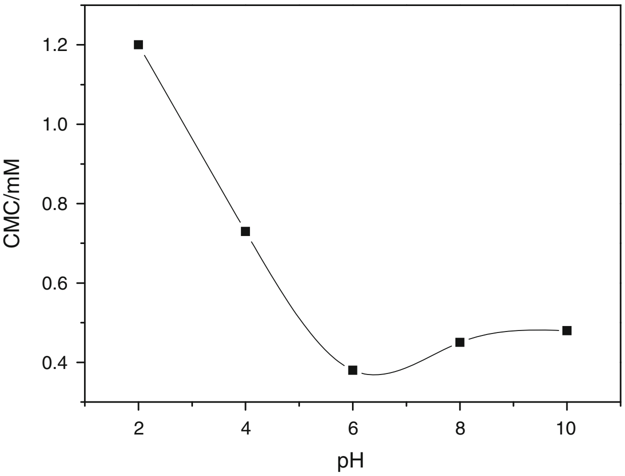

Fig. 3 The CMC values as a function of pH for DDEAO at 298 K

surface activity of DDEAO is somewhat superior to that of DDMAO.

From the surface tension curves, the surface excess concentrations of DDEAO (Γ_{max}) at various pH values were calculated from the Gibbs adsorption Eq. (1) and the minimum surface area per surfactant molecule (A_{min}) was obtained from the Gibbs Eq. (2):

$$\Gamma_{max} = -\frac{1}{n2.303RT}\left(\frac{\partial\gamma}{\partial \log c}\right)_T \quad (1)$$

$$A_{min} = \frac{1}{N_A \Gamma_{max}} \quad (2)$$

where R is the gas constant, T is the absolute temperature, N_A is Avogadro's number and c is the concentration of surfactant in solution. For nonionic and zwitterionic surfactant, $n = 1$ [25–28]. The values of Γ_{max} and A_{min}, which are considered as a sign of packing densities of surfactant molecules at the air/liquid interface, are presented in Table 1.

As shown in Table 1, there are higher Γ_{max} values and lower A_{min} values for DDEAO with the increase of pH with pH < 6. At a lower pH, the strong electrostatic repulsion between the hydrophilic groups leads to a looser compactness of the aggregation at the air/liquid interface. However, the amount of nonprotonated species increases with a rise in pH, and the electrostatic repulsion at the air/liquid interface is weakened. This encourages the DDEAO molecules to arrange more closely. In pH > 6, DDEAO shows a decrease Γ_{max} value and a increase A_{min} value with increasing pH. The effect of interaction between hydrophobic chains becomes a dominant factor in the determination of Γ_{max} and A_{min} values. With increasing pH, the van der Waals interaction between the hydrophobic chains becomes weak resulting in lower packing densities of surfactant molecules in the surface adsorption layer. In addition, in comparison with DDMAO [23], DDEAO has larger Γ_{max} and smaller A_{min} values in aqueous solutions. The hydroxyethyl group of DDEAO can form a hydrogen bond between molecules. The intermolecular hydrogen bonds can conduce the molecules to arrange more compactly at the air/water interface monolayer.

The adsorption of surfactants can be expressed by the pC$_{20}$ value. It is the value of the logarithm of the reciprocal C$_{20}$, i.e., the concentration of surfactants to lead the surface tension of water to decrease by 20 mN/m. The larger the pC$_{20}$ value, the higher is the adsorption efficiency of surfactants [29]. The values of pC$_{20}$ obtained for DDEAO at various pH values are also presented in Table 1. From Table 1, with the increases in pH, pC$_{20}$ values increase up to pH = 6 and then slightly decrease. It is noteworthy that the pC$_{20}$ values are larger in the pH 6–10 range compared with those of pH 2–4. The result indicates that DDEAO is easily adsorbed at high pH. It is considered to be due to the following two points. First, the electrostatic repulsion between the hydrophilic groups is a dominant factor in the determination of pC$_{20}$ values at low pH, while the effect of interaction between hydrophobic chains is dominant factor at high pH. Second, the influence of electrostatic repulsion is stronger than the effect of interaction between hydrophobic chains.

The packing parameter (P) determines the geometry of micelles. The packing parameter of DDEAO at various pH values are obtained by using Eq. (3) [30].

$$P = \frac{V_c}{l_c A_{\min}} \quad (3)$$

where V_c is the volume of hydrophobic group, l_c is the chain length of hydrophobic group. $A_{\min}$ is the minimum surface area per surfactant molecule. When the hydrophobic group is the straight-chain alkane, V_c and l_c were calculated by Tanford's equations [31]:

$$V_c = (27.4 + 26.9n) \times 10^{-3}\ \mathrm{nm}^3 \quad (4)$$

$$l_c = (0.15 + 0.1265n)\ \mathrm{nm} \quad (5)$$

where n is the number of carbon atoms in the hydrocarbon chain. The values of the packing parameter of DDEAO with different pH values at 298 K are given in Table 1. $P \leq 1/3$ indicates spherical micelle formation. $1/3 < P \leq 1/2$ shows symmetrical micelle formation such as spheroidicity, oblate spheroids and rods. The vesicles formation is generally indicated when the packing parameter lies within the range of 0.5–1 [32]. From the P values in Table 1, the various pH values of DDEAO at 298 K have values of the packing parameter within the range of 0.5–1. Based on the molecular structure of DDEAO, the sectional area of the hydrophilic group is compressed because of the attractive interaction between the hydrogen bond of head groups. The value of the sectional area of the hydrophilic group relative to the volume of the hydrophobic group becomes small, which causes the P value to be in the range of 0.5–1.

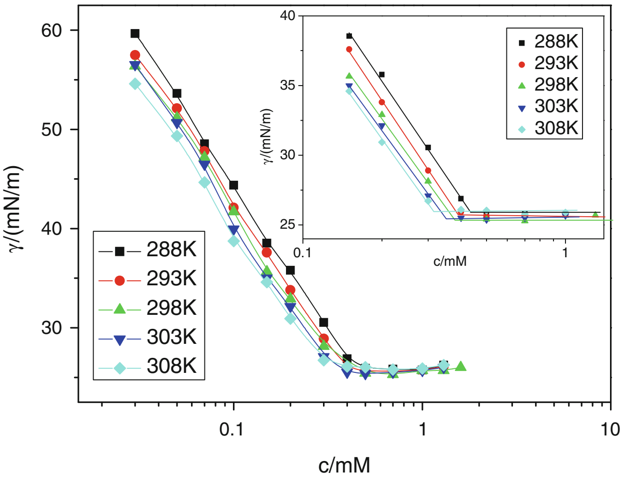

Fig. 4 Surface tension vs. concentration for DDEAO in aqueous solution at different temperatures

Effect of Temperature on the Aggregation Behavior of DDEAO

Surface tension measurements were employed to study the micellar aggregation behavior of DDEAO in aqueous solutions at different temperatures. Figure 4 depicts the surface tension isotherms of DDEAO in the temperature range of 288–308 K. CMC and γ_{CMC} values determined by surface tension measurements are listed in Table 2.

As shown in Table 2, CMC values decrease slightly with the increase of temperature. In the case of DDMAO, CMC values of the nonprotonated species decrease with increasing temperature, while the values of protonated species increase with temperature [9]. Although DDEAO shows cationic characteristics in aqueous solution, the variation of CMC with temperature is similar to the nonprotonated species of DDMAO. There are two opposite processes for the effect of temperature on the CMC values. Increasing temperature weakens the hydration of the hydrophilic groups, which favors micelle formation and results in a lower CMC. However, the orderly structure of the water molecules surrounding the hydrophobic groups is broken with increasing temperature, which is a disadvantageous factor for micelle formation. It is clear from the data that the former is a dominant factor in the determination of the CMC values of DDEAO in this work. There is a slight variation with the value of γ_{CMC} with increasing temperature.

Based on the single-stage equilibrium model of micellar solution, the standard Gibbs free energy change of micellization (ΔG_{mic}), the standard enthalpy change for the micellization process (ΔH_{mic}), and the standard entropy of micelle formation (ΔS_{mic}) for nonionic and zwitterionic surfactants can be estimated from the following equations [33]:

Table 2 Key surface property parameters and thermodynamic parameters of DDEAO at different temperatures

T/K	CMC (mM)	γ_{CMC} (mN/m)	ΔG_{mic} (kJ/mol)	ΔH_{mic} (kJ/mol)	$-T\Delta S_{\mathrm{mic}}$ (kJ/mol)
288	0.42	25.74	−28.23	8.41	−36.64
293	0.40	25.73	−28.84	8.71	−37.55
298	0.38	25.64	−29.46	9.01	−38.47
303	0.35	25.53	−30.16	9.31	−39.47
308	0.33	25.77	−30.81	9.62	−40.43

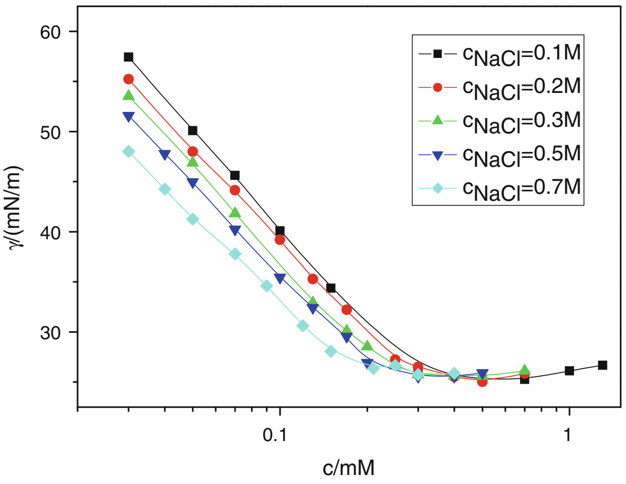

Fig. 5 Surface tension vs. concentration for DDEAO with different concentrations of NaCl at 298 K

$$\Delta G_{\text{mic}} = RT \ln X_{\text{cmc}} \tag{6}$$

$$\Delta H_{\text{mic}} = -RT^2 \frac{d \ln X_{\text{cmc}}}{dT} \tag{7}$$

$$\Delta S_{\text{mic}} = \frac{\Delta H_{\text{mic}} - \Delta G_{\text{mic}}}{T} \tag{8}$$

where R is the gas constant, T is the absolute temperature, X_{CMC} is the CMC expressed as a mole fraction, $X_{\text{CMC}} = \text{CMC}/55.4$. All the calculated thermodynamic parameters of micellization at different temperatures for DDEAO are listed in Table 2.

As it can be seen, the negative value of ΔG_{mic} means that the micelle formation of DDEAO is a spontaneous process in the temperature range investigated. More negative ΔG_{mic} values are observed at high temperature, which indicates that it is easier to form micelle than those at low temperature. The values of ΔH_{mic} are positive, so the micelle formation process is endothermic. The data from Table 2 imply that the negative values of ΔG_{mic} are mainly determined by the large negative values of $-T\Delta S_{\text{mic}}$. Therefore, the micellization process is mainly entropy-driven for DDEAO in aqueous solutions. The driving force of the micellization process is the hydrophobic group of surfactant transfer from solution environment to the interior of the micelle. During this process, the solvated water molecules are released, thus an entropy increase is obtained [34, 35].

Effect of Ionic Strength on the Aggregation Behavior of DDEAO

Surface tension measurements were employed to study the effect of ionic strength on surface activity of DDEAO. Figure 5 depicts the plots of surface tension versus surfactant concentration of DDEAO varied with different concentrations of NaCl at 298 K. The key surface property parameters are summarized in Table 3.

Table 3 Key surface property parameters of DDEAO with different NaCl concentrations at 298 K

c_{NaCl} (M)	CMC (mM)	γ_{CMC} (mN/m)	$\Gamma_{\max}$ (μmol/m^2)	$A_{\min}$ (Å^2)
0.1	0.30	25.61	5.56	29.9
0.15	0.29	25.55	5.47	30.4
0.2	0.28	25.53	5.33	31.2
0.3	0.26	25.63	5.26	31.6
0.4	0.25	25.71	5.25	31.6
0.5	0.22	25.79	5.21	31.9
0.6	0.20	25.60	5.14	32.3
0.7	0.18	25.78	4.95	33.6

As can be seen, the value of γ_{CMC} has little variation, however, CMC values decrease with the increase of NaCl. The decrease of CMC values depends upon the salting-out effect on the hydrocarbon chain and the influence of the hydrophilic group on water structure. In addition, the increase in NaCl decreases the surface charge density and electrical repulsion, which favors micelle formation and decreases the CMC [36, 37]. A decreasing trend in $\Gamma_{\max}$ and an increasing trend in $A_{\min}$ suggesting a lower packing density at the air/liquid interface. This can be ascribed to the increase in hydrophilic group's volume with the addition of NaCl.

Figure 6 shows the relationship between the logarithm of CMC values and the logarithm of the concentration of NaCl in aqueous solutions at 298 K. It is different from that of ionic surfactants, e.g., sodium dodecylsulfate (SDS) which exhibits a linear dependence. It is also incomplete similarity to those of zwitterionic surfactants which show biphasic curves [38, 39]. When the salt concentration is low, log CMC shows a linear dependence on log c_{NaCl} for DDEAO. However, a deviation occurs in the range of c_{NaCl} >0.35 M. The CMC value declines with a sharp slope. Similar, a deviation occurs in the range of $c_{\text{NaCl}} > 1$ M in the case of the protonated species of DDMAO [9]. Compared with DDMAO, DDEAO is more sensitive to salt concentration. The observed dependence can be interpreted in terms of the salting-out effect on the hydrocarbon tail. At the same time, shape/size changes of micelles with the increase in NaCl may be another reason. The result is useful for some applications, such as emulsification of oil spills at sea and enhancement of oil recovery from depleted reservoirs.

Conclusions

The effects of pH, temperature and ionic strength on the aggregation behavior of DDEAO were investigated. The

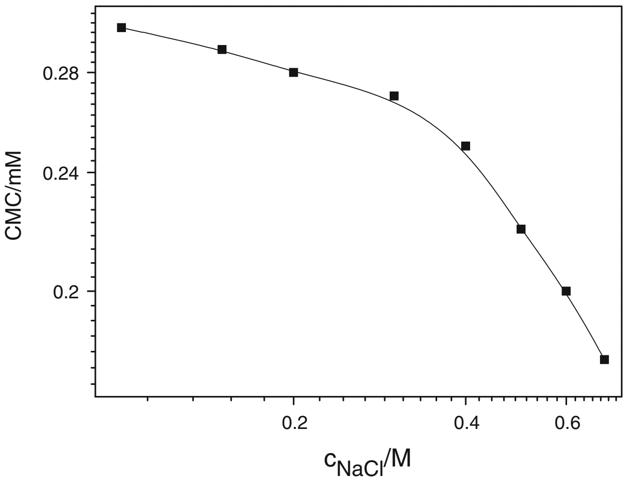

Fig. 6 The relationship between the logarithm of CMC values and the logarithm of concentration of NaCl at 298 K

experimental results demonstrate that DDEAO shows good surface activity over a large pH range, especially under weakly acidic condition. It can be exploited for utilization as surfactants for acidic shampoo. Compared with DDMAO, the surface activity of DDEAO is somewhat superior to that of DDMAO [9, 10, 24]. The packing parameters (*P*) ensure the formation of vesicles for DDEAO at various pH values. The surface activity of DDEAO increases with increasing temperature. This trend is similar to the nonprotonated species of DDMAO [9]. CMC values decrease with increasing ionic strength. Compared with DDMAO, DDEAO is more sensitive to salt concentration [9]. Above all, it has a high surface activity under a wide range of conditions, which makes it a suitable candidate for industrial applications combining the multiple functions of amine oxide surfactants.

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