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Preparation of Quaternary Ammonium Gemini Surfactant and Its Application in Insulator Cleaning

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Abstract. In this paper, quaternary ammonium salt type Gemini surfactant G12 was designed and synthesized, and its structure was characterized by infrared and nuclear magnetic resonance. The surface tension, permeability, emulsification ability and cleaning of insulator surface contamination in the composite system were tested, and the test results showed that it had low critical micelle concentration (cmc) and good emulsification performance. Due to the presence of rigid groups, the permeability is average. When quaternary ammonium Gemini surfactant G12 is mixed with permeant JFC-E, and is combined with auxiliary and organic solvent, the cleaning rate of insulators can reach more than 90%.

Key words: Gemini surfactant, insulator cleaning4, mixture, emulsibility, permeability.

1. Introduction

Insulators are important components in the circuit transportation system. Due to long-term exposure to outdoor environment, the surface area of insulators is severely polluted, which may lead to potential safety hazards[1]. These hygroscopic and conductive filth particles such as solid, liquid and gas filth particles adhered to the insulator surface greatly reduce the insulation level of the insulator under the joint action of fog, dew, rain, snow, frost and other weather conditions. As a result, "pollution flashover" malfunction will occur even under the operating voltage, resulting in power outage[2,3]. This can cause serious economic loss to Chinese electric power system[4].

Studies have shown that the main components of insulator pollution are soluble NaCl, CaSO4, CaO, insoluble oil, shellac and SiO2[5,6,7,8,9]. By studying the existing methods of cleaning electric equipment with chemical cleaning agent, it is found that there are still problems of weak cleaning ability and unsatisfactory cleaning effect[10,11,12].

Surfactants are an important component of water-based cleaning agents[13]. Due to the existence of intermolecular repulsion, traditional surfactants tend to have large molecular spacing when they are aligned on the surface of water, which has limited effect on the reduction of surface tension. Gemini surfactant is a new type of surfactant containing two hydrophilic head groups and two hydrophobic tail chains[14]. The strong force of the covalent bond pulls into the distance between the two hydrophilic groups, which makes them more tightly arranged at the liquid level and has better performance[15,16,17,18].

2. Experimental conditions and methods

2.1 Materials and Instruments

Phthalic anhydride, epichlorohydrin, ethylene glycol, dipropylene glycol butyl ether and other reagents were commercially available analytical pure; Surfactant iso-octanol polyoxyethylene ether (JFC-E), BASF (China) Co., LTD. Sodium dodecyl diphenyl ether disulfonate (2A1), DOW Inc.

SFT-D1 surface tension instrument, Beijing Hake Experimental Instrument Factory; HARKE-SPCAX1 Contact Angle Tester, Beijing Hake Experimental Instrument Factory; Nicolet iS50 infrared spectrometer, Thermo Fisher; AVANCE Ⅲ 500 NMR instrument, Bruker, Switzerland.

2.2 Experimental methods

2.2.1 Synthesis of phthalate monoesters

6.21g ethylene glycol (0.10 mole) and 37.03g phthalic anhydride (0.25 mole) were added to a 250 mL round-bottom flask, followed by 0.2 mL concentrated sulfuric acid as catalyst, and 100 mL acetone as solvent. It was refluxed at 65℃ for 4h. Acetone recrystallized three times to obtain phthalate monoester (81% yield).

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2.2.2 Synthesis of phthalate monoester - chloropropane

2.94g phthalate monoester (0.10 mol) and 1.85g epichlorohydrin (0.20 mol) were added to a 250 mL round-bottom flask, and then 100 mL toluene was added as solvent. It was heated to 105℃ for reflux reaction for 6h, and then dried to obtain phthalate monoester chloro-propane (83% yield).

2.2.3 Synthesis process of quaternary ammonium Gemini surfactant G12

0.10 mol phthalate monoester chloro-propane and 0.20 mol dodecyl dimethyl tert-amine were added to a 250 ml round-bottom flask, and the reaction was carried out at 65℃ for 4h to obtain a yellow thick liquid, which was recrystallized with acetone to obtain G12 (78% yield).

2.3 Characterization and testing methods

2.3.1 Infrared absorption spectrum

KBr tablet was used for the test sample, the test resolution was 4 cm^{-1}, the number of scans was 16, and the scan range was 400-4000 cm^{-1}.

2.3.2 Nuclear magnetic resonance hydrogen spectrum

Deuterated chloroform was used as the solvent and the reference was used for calibration. The test frequency was 400 MHz and the temperature was 298.15K.

2.3.3 Surface tension test

A series of aqueous surfactant solutions with different concentrations were prepared and the surface tension was measured by ring method at 25℃. The corresponding γ-lg$C$ curve was plotted, and then the critical micelle concentration (C_{cmc}) and the corresponding surface tension (γ_{cmc}) were obtained. Parallel test three times, take the average as the measurement result.

2.3.4 Penetration capacity test

Using the canvas settling method, the standard canvas was placed on the surface of 500 ml of 0.5% surfactant aqueous solution, and the settling time of the canvas from the surface to the bottom of the beaker was recorded for three consecutive times, and the average value was taken.

2.3.5 Emulsifying ability test

Using the phase separation method. 20 ml 25 g/L aqueous surfactant was put into a 100 mL stopper cylinder, and 20 mL liquid paraffin was added into the cylinder. Then the cylinder was placed in a water bath at 34℃ for 5 minutes. After 10 times of vigorous shaking, it was left to stand, and the time needed to separate 10 ml of aqueous phase was recorded. The operation was repeated for 5 times, and the average value was taken.

2.3.6 Cleaning ability test

Silica, kaolin, 30# transformer oil and sodium chloride were configured into pollution standards according to the mass ratio of 1:10:88:1, weighing 100 mm×100 mm tiles and recording as m1. 3g filth was coated on the surface of the tiles, dried at 60℃ for 12h, and weighed and recorded as m2. The tiles were soaked in the cleanser for 20 minutes, removed and dried for 2 hours, and the weight was recorded as m3. Cleaning rate = (m2-m1-m3) / (m2-m1) ×100%.

3. Results and discussion

3.1 Structural characterization

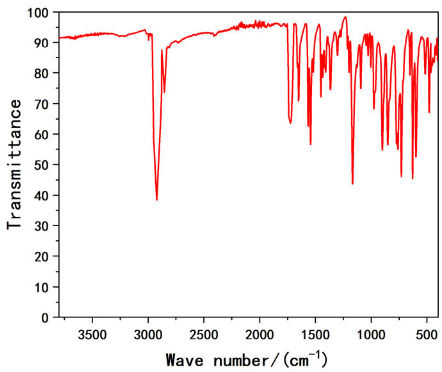

Figure 1. The FTIR spectrum of G12.

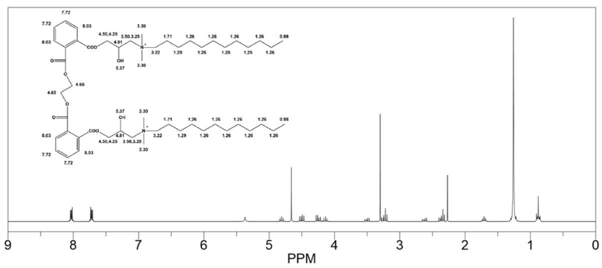

Figure 2. The ^{1}H NMR spectrum of G12.

The FTIR and ^{1}H NMR of G12 are shown in Figure 1 and Figure 2 respectively. The characteristic peak of alkane chain appears near 2922 cm^{-1}2853 cm^{-1}; The stretching vibration peak of C=O bond in ester group appears at 1724 cm^{-1}; C-N bond stretching vibration peak appeared near 1467 cm^{-1}; The characteristic absorption peak of benzene ring appears near 1581 cm^{-1} and 1567 cm^{-1}.
G12(400 MHz, $COCl_3$, TMS): 0.89(t, 6H, $(CH_2)_{13}CH_3$), 3.38(t, 12H,N-CH_3), 1.26(m, 32H, CH_2-CH_2-CH_2-$(CH_2)_{10}$-CH_3), 3.50(t, 12H,N-CH_3), 3.24(d, 4H, N-CH_2-$(H_2)_2$ CH_3), 1.29(d, 4H, CH_2-CH_2-CH_2-$(CH_2)_{10}$-CH_3), 3.50～3.52(m, 4H, N-CH_2-CH), 5.37(m, 2H, OH), 1.71(d, 4H, N-CH_2-CH_2-$(CH_2)_{11}$-CH_3), 4.25～4.50(m, 4H, OH-CH-CH_2-O), 3.22(d, 4H, N-CH_2-(CH_2)12-CH_3), 4.81(m,

2H, 2(CH_2)-CH-OH), 7.72～8.03(d, 8h, C_6H_4), 4.66(d, 4H, O-CH_2-CH_2-O).

According to the results of FTIR and ^{1}H NMR, it can be determined that the product structure is consistent with the target product.

3.2 Surface tension test

The surface tension of surfactant aqueous solutions with different concentrations at 25 ºC was measured by the ring method and the results are summarized in Figure 3. It can be seen from Figure 43 that the C_{cmc} of G12 is 1.6 × 10^{-4} mol/L, γ_{cmc} is 30.3 mN/m. The reason for the surface tension can reduce to 30.3 mN/m is mainly because the rigid group contained in the molecular structure can pull into the space between the two hydrophilic head groups in the form of covalent bond, making the surfactant molecules more closely arranged on the liquid surface.

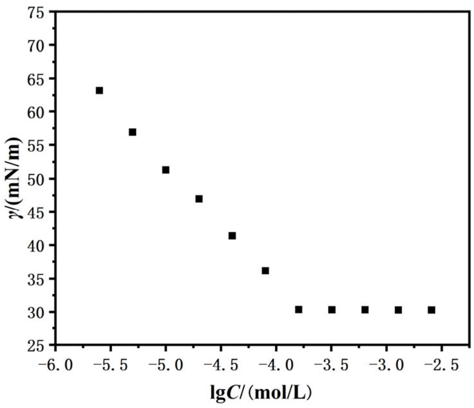

Figure 3. The Surface tension test result of G12.

3.3 Permeability test

The wettability and permeability of G12 and JFC-E were tested by canvas sedimentation method, and the results are summarized in Table 1. It can be seen that the wetting permeability of G12 is general, mainly because the steric hindrance of rigid group is large, which hinders the penetration of surfactant. Therefore, in actual use, G12 needs to be used combine with others to achieve better application effect by using the synergistic effect.

Table 1. Canvas contact time of G12 and JFC-E.

Sample	Test 1	Test 2	Test 3
G12	137"47	135"39	138"51
JFC-E	20"56	22"30	23"19

3.4 Emulsification capacity test

The emulsification ability of G12 and 2A1 were tested by phase separation method, and the experimental results are shown in Table 2. It can be seen that the emulsification performance of G12 is stronger than 2A1, so it is feasible to use G12 in the cleaning field.

Table 2. Time for 10 mL aqueous phase separation of G12 and 2A1.

Sample	Test 1	Test 2	Test 3	Test 4	Test 5
G12	35"09	34"29	35"77	34"26	35"49
2A1	29"28	29"59	29"06	30"44	29"86

3.5 Cleaning capacity test

G12 was applied to the cleaning agent system for insulator surface cleaning, and three cleaning agents were prepared respectively. The composition of detergent is shown in Table 3. The dried porcelain chips were cleaned in three kinds of cleaning agents for a period of time, and the Cleaning efficiency results are shown in Table 4. It shows that the detergent mixed with two surfactants has the best cleaning effect.

We mixed the dirt directly with the cleaning solution, stirred it and placed it for 1 min to observe the dispersion of the system. The stability of No. 1 detergent lotion added with G12 as emulsifier alone is even better than that of No. 3 detergent added with G12 and JFC-E composite system. The main reason is that G12 has better emulsification effect and can better disperse the dirt in the cleaning agent. The reason for the better cleaning effect of the composite system on the porcelain chip is that JFC-E has well permeability, which can play a synergistic effect when used with G12. JFC-E penetrates into the interface of insoluble dirt such as porcelain chips, silica, kaolin, etc. to peel off the dirt. G12 quickly wraps up these dirties to form lotion, which is dispersed in the cleaning liquid system to achieve better cleaning effect.

Table 3. The formulas of different cleaning agents.

Detergent 1		Detergent 2		Detergent 3	
Component	Content	Component	Content	Component	Content
cholamine	4	cholamine	4	cholamine	4
PEG	4	PEG	4	PEG	4
inhibiter	1	inhibiter	1	inhibiter	1
G12	0.5	G12	0	G12	0.5
JFC-E	0	JFC-E	0.5	JFC-E	0.5
water	90.5	water	90.5	water	90

Table 4. Cleaning Results of different formulas.

Sample	Detergent 1	Detergent 2	Detergent 3
Cleaning efficiency(%)	83.56	72.88	95.32

4. Conclusion

The quaternary ammonium salt gemini surfactant was designed and synthesized, and its structure was characterized by IR and NMR. The surfactant has low cmc concentration and can reduce the surface tension of water to 30.3 mN/m. The surfactant mixed with auxiliaries, organic solvents, penetrants, etc. was used to prepared detergent which is used for insulator pollution cleaning. The detergent can achieve good cleaning effect after mixing with penetrant.

Acknowledgements

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