

# Application and Synthesis of Gemini Surfactant in Heavy Oil Development

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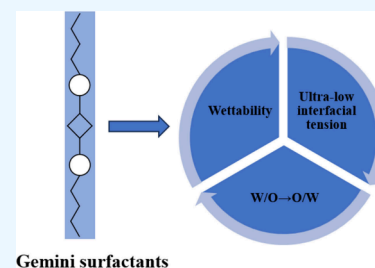
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**ABSTRACT:** Gemini surfactants have many potential applications in the viscosity reduction of heavy oils due to their unique structure, which appears to consist of two single-chain surfactant molecules. This paper introduces the viscosity reduction mechanism of gemini surfactants and the research progress of four types of Gemini surfactants used in the viscosity reduction of heavy oils at home and abroad. The study found that in the formation water containing calcium and magnesium ions with a salinity of more than 30000 mg/L, the added amount of gemini anionic surfactant was 0.4–0.5%, the viscosity reduction rate could reach more than 90%, and ultralow interfacial tension ( $10^{-3}$  orders of magnitude) was achieved. Based on the above advantages, the synthesis and physicochemical properties of gemini anionic surfactants are summarized. Finally, the difficulties in synthesizing anionic gemini surfactants are summarized, and their future development trend is given.



## 1. INTRODUCTION

With increasing energy demands and the serious shortage of conventional oil and gas resources,<sup>1,2</sup> heavy oil as unconventional oil and gas resources has been paid more and more attention by all countries in the world. However, how to extract a heavy oil with high viscosity is a challenge. The existing technologies for reducing the viscosity of heavy oil are mainly divided into physical methods and chemical methods. Chemical viscosity reduction is an economical and effective method, which includes viscosity reduction by adding surfactant, catalytic cracking, and other methods.<sup>3</sup> Because of the advantages of low cost, simple field operation, and high efficiency, adding surfactant to reduce viscosity has become a hot research topic in heavy oil recovery technology.<sup>4</sup>

In the 1960s, Simon<sup>5</sup> and others injected surfactant into the wellbore to disperse heavy oil into an O/W emulsion, which effectively reduced the viscosity of heavy oil and improved the efficiency of exploitation. Since then, surfactants have been widely used in the viscosity reduction of heavy oils. Among the many types of surfactants, gemini surfactants have attracted the attention of researchers. For example, Manisha<sup>6</sup> synthesized two triazine-based gemini surfactants, when mixed with toluene, that can greatly reduce the viscosity of heavy oil containing high asphaltene. As can be seen from the structural diagram of the gemini surfactant in Figure 1, compared with the single-chain surfactant, the gemini surfactant is a surfactant with a special structure of two hydrophobic chains, two hydrophilic groups, and one linking group,<sup>7,8</sup> and its molecules seem to be formed by the connection of two traditional single-chain surfactant molecules.<sup>9</sup> Because of their special structure, gemini surfactants have many unique properties: Under high-concentration surfactants, the adsorption capacity is also very

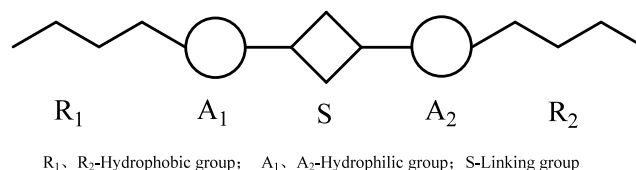


Figure 1. Structure of the gemini surfactant.

small.<sup>10</sup> It has high surface activity and can achieve ultralow interfacial tension ( $10^{-3}$  orders of magnitude),<sup>11,12</sup> which makes the surfactant more closely arranged at the oil–water interface, forming an O/W emulsion, and greatly reducing the viscosity of heavy oil. The cmc is low.<sup>13</sup> With strong temperature and salt resistance, the viscosity reduction rate of a series of nonionic gemini surfactants synthesized by Wang<sup>14</sup> is still above 95% after being treated at 300 °C. The rocks can be transformed to more hydrophilic conditions, thereby improving oil recovery, etc.<sup>15</sup>

Despite the special properties of gemini surfactants, few studies have focused on their practical application, so gemini surfactants have great application potential in the viscosity reduction of heavy oils,<sup>16</sup> which has attracted great interest from many scientists at home and abroad in recent years.<sup>17</sup>

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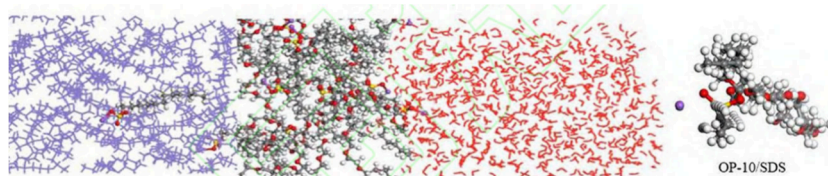


Figure 2. Dynamic simulation between SDS/OP-10 and heavy oil molecules.

In this paper, the structure and viscosity reduction mechanism of gemini surfactants and the application effects of different types of gemini surfactants in the viscosity reduction of heavy oils are analyzed. Meanwhile, the synthesis and performance evaluation of gemini anionic surfactants are reviewed. Finally, the difficulties in the synthesis of anionic gemini surfactants are summarized. The future development trend is also given.

## 2. VISCOSITY REDUCTION MECHANISM OF GEMINI SURFACTANTS AND THEIR APPLICATION IN HEAVY OILS

**2.1. Viscosity Reduction Mechanism.** The viscosity reduction mechanism of a surfactant mainly includes emulsification and adsorption viscosity reduction.<sup>18,19</sup> Regarding emulsification and viscosity reduction, as can be seen from the results of the dynamic simulation between SDS/OP-10 and heavy oil molecules in Figure 2,<sup>20</sup> the nonpolar groups in the surfactant extend to the oil phase, and the polar groups can connect some water droplets through the oil film, thus increasing the contact area between the surfactant and heavy oil and enhancing the substitution effect on the interface, resulting in a decrease in the interfacial tension between oil and water. As can be seen from Figure 3, the emulsification

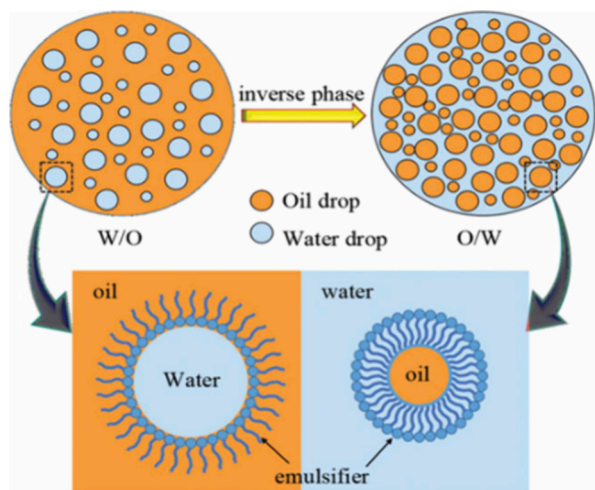


Figure 3. Mechanism diagram of emulsification and viscosity reduction.

viscosity reduction mechanism diagram,<sup>21</sup> a W/O emulsion is transformed into an O/W emulsion with very low viscosity under the action of surfactant. During the flow of crude oil, the friction between oil films is transformed into the friction between water films,<sup>22</sup> which greatly reduces the viscosity and friction resistance.

Regarding viscosity reduction by adsorption, the surfactant can adsorb a water film of the viscosity reducer solution on the

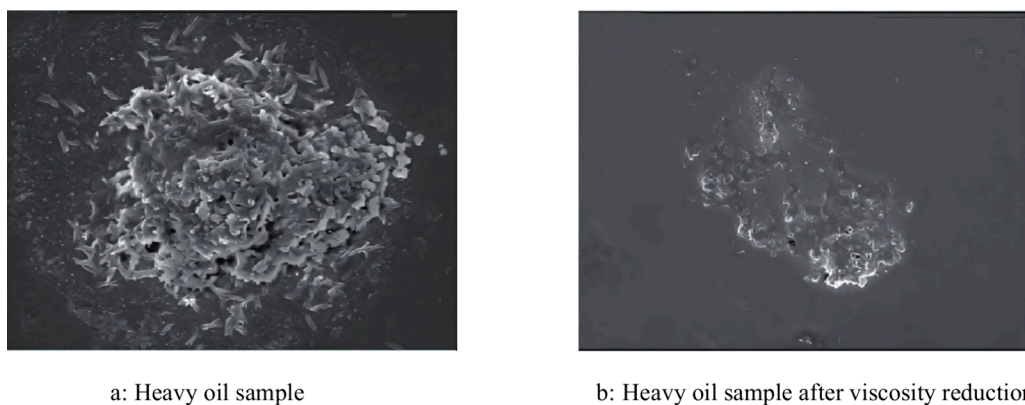
pipe wall so that the friction between the crude oil and the pipe wall becomes the friction between the viscosity reducer solution and the pipe wall,<sup>23,24</sup> and the viscosity is reduced by reducing the flow resistance. As can be seen from the microscopic morphology of heavy oil before and after viscosity reduction<sup>25</sup> in Figure 4, some surfactants can also break up the aggregates formed by the overlapping of colloid and asphaltene by means of hydrogen bonding to form many small aggregates, which reduces the cohesion of crude oil and plays a role in viscosity reduction.<sup>26</sup>

Gemini surfactants can be divided into anionic, cationic, nonionic amphoteric, and nonionic types according to their hydrophilic groups.<sup>27–30</sup> Table 1 summarizes the research data of different types of gemini surfactants in the viscosity reduction of heavy oil and summarizes the source and original viscosity of heavy oil, salinity, surfactant concentration, temperature, oil–water ratio, viscosity reduction rate, and interfacial tension.

**2.2. Anionic Gemini Surfactant.** Liu Peng et al.<sup>31</sup> studied the emulsifying and viscosity-reducing ability of sulfate gemini surfactants GA8-4-8, GA12-4-12, OP-10, and OP-20 on Xianhe heavy oil through solubility, interfacial tension, and viscosity-reducing rate experiments. The results show that the four emulsifying viscosity reducers have good solubility in the simulated formation water with a salinity of 40000 mg/L, the lowest interfacial tension between GA8-4-8 and GA12-4-12 and heavy oil can reach  $10^{-2}$  mN/m, and they have strong emulsifying and dispersing abilities and are therefore suitable for reducing the viscosity of Xianhe heavy oil. When the temperature is 50 °C, the ratio of oil to water is 6:4 and the shear rate is  $7.3 \text{ s}^{-1}$ , and the viscosity reduction rate is above 90% when the amount of GA8-4-8 is 0.3%. When the concentration of GA8-4-8 is 0.4%, the viscosity reduction rate can be more than 99%. When the temperature is 30–70 °C, it is the most suitable emulsion viscosity reducer for Xianhe heavy oil among the four surfactants.

Liao Hui<sup>32,33</sup> synthesized a new type of gemini surface viscosity reducer NP containing benzene rings that is similar to heavy oil components, aiming at the difficulties encountered in the exploitation of deep superheavy oil with high salinity and high asphaltene content. The viscosity–temperature curve was measured with an Anton Paar rheometer at a 1:1 oil–water ratio and constant temperature for 30 min at different temperatures. The experimental results show that the viscosity reduction rate of deep superheavy oil can reach 95% by adding 0.5% NP-4 in high salinity simulated formation water, the viscosity reduction rate can reach more than 99% by adding 2% NP-4, and the oil–water interfacial tension can be reduced to 0.0364 mN/m by adding 0.3% NP-4. In addition, it was found that the length of the carbon chain of the spacer group had an effect on the oil–water interfacial tension.

Gao Jinfeng<sup>34</sup> synthesized gemini anionic surfactant GMS-1 from  $C_{16}$  and  $C_{18}$  fatty alcohols, epoxypromanesulfonic acid, and ethylenediamine according to the component character-



**Figure 4.** Microstructure of heavy oil before and after viscosity reduction.

**Table 1.** Application of the Gemini Surfactant in the Viscosity Reduction of Heavy Oil

Type of surfactants	Heavy oil source	Viscosity of heavy oil mPa·s	Degree of mineralization mg/L	Concentration wt %	Oil–water ratio	Viscosity reduction %	Interfacial tension mN/m	Ref
Anionic	Xianhe oilfield	2500	40000 Ca <sup>2+</sup> :1000	0.4	6:4	99(50 °C)	10 <sup>−3</sup>	31
Anionic	West G oilfield	49900	60000 Ca <sup>2+</sup> +Mg <sup>2+</sup> :40000	0.5	1:1	95.2(60 °C)	0.0364(45 °C)	32, 33
Anionic	Dongxinying oilfield	8769	30000 Ca <sup>2+</sup> :800 Mg <sup>2+</sup> :200	0.1	7:3	96.49(50 °C)	-	34
Anionic	Chenjing oilfield	2100	-	-	7:3	94.3(60 °C)	-	35
Anionic	Shuguang Oilfield	169405	-	0.5	7:3	90.4(70 °C)	-	36
Anionic	Lungu oilfield	17900	-	0.5	6:4	92.1(60 °C)	2.71 × 10 <sup>−3</sup>	37
Cationic	Enping oilfield	339.7	20000 Ca <sup>2+</sup> +Mg <sup>2+</sup> :2500	0.6	3:7	94.04(50 °C)	8.2 × 10 <sup>−3</sup>	38
Cationic	Tahe oilfield	7000	214739.9 Ca <sup>2+</sup> :11598.6 Mg <sup>2+</sup> :1224.5	0.25	7:3	98.54(90 °C)	-	39
Cationic	Offshore oilfield	1287.6	-	-	7:3	93.7(60 °C)	0.065	40
Cationic	-	1265.5	25800	0.2	7:3	92.5(60 °C)	10 <sup>−3</sup>	41
Amphoteric	Shengli Oilfield	5154	12000	0.3	7:3	91.4(50 °C)	0.08	42
Nonionic	Bohai oilfield	3500	82460	1	7:3	96(50 °C)	0.65	43, 44
-	S OilField	440(20 °C)	5680.89	0.3	1:1	97(50 °C)	0.1656	45
-	Lungu oilfield	40700(50 °C)	258900	0.5	1:1	92(60 °C)	-	46
-	Panjin oilfield	820 mm <sup>2</sup> /s <sup>a</sup>	-	0.5	7:3	98.5(50 °C)	-	47
-	Offshore oilfield	400	-	1	1:1	98.68(60 °C)	10 <sup>−3</sup>	48

<sup>a</sup>The density of heavy oil is not marked in the original text and cannot be converted into mPa s.

istics of Dongxinying 27 Guantao Formation heavy oil. The viscosity of heavy oil emulsions at different temperatures was measured with an RV20 rotary viscometer at a shear rate of 17 s<sup>−1</sup>. The experiment shows that GMS-1 aqueous solution with a 1.0–2.0% mass fraction prepared with 30 g/L simulated formation water and heavy oil has a viscosity reduction rate of more than 98% at an oil–water ratio of 7:3. GMS-1 aqueous solution with a mass fraction of 0.1–1.5% can form a low-viscosity emulsion with heavy oil when the oil–water ratio is 7:3 and above and the viscosity reduction rate is more than 96%. The viscosity reduction rate of 1% GMS-1 aqueous solution in Well Ying 27x5 reached 96.97%.

Wang Haibo<sup>35</sup> prepared a weak gel flooding system through the combination of dimethylene-1,2-bis (hexadecyl dimethylammonium bromide) and dimethylene-1,2-bis (hexadecyl methyl sodium taurate). The emulsification and viscosity reduction effect of the weak gel flooding system was measured with a viscometer at 200 r/min. The experiment shows that when the temperature is 60 °C and the oil–water ratio is 7:3,

the viscosity of different heavy oils can be reduced by the system, the viscosity reduction effect is more obvious for the heavy oil with a viscosity greater than 2100 mPa s, and the viscosity reduction rate can reach more than 94%. The displacement experiment results show that the system can increase the displacement efficiency by 11% compared with 60 °C hot water flooding alone.

Hao Jianyu<sup>36</sup> found that sulfonate surfactants had poor performance in field applications and synthesized gemini alkylbenzenesulfonate surfactant with dodecylbenzene, dichloromethane, chlorosulfonic acid, and other raw materials. After being treated at 320 °C, the surface tension of the 0.5% surfactant solution is 24.42 mN/m, which has good temperature resistance. When the oil/water mixture with an oil/water ratio of 7:3 is kept constant at 70 °C, the viscosity reduction rate can still reach 90.5%. The field application shows that the oil production is increased by 257 tons, and the effect is obvious.

Yue Quan et al.<sup>37</sup> developed a series of sulfate gemini surfactants, which have higher surface activity than single-chain surfactants, and the lowest surface tension can reach 26.3 mN/m, aiming at the difficulty of water injection and oil production in the later stage. The interfacial tension between CA-1 solution with a mass concentration of 0.1% and heavy oil can reach  $2.71 \times 10^{-3}$  mN/m. When the temperature is 60 °C and the oil–water ratio is 6:4, the O/W emulsion can be formed by 0.5% CA-4 gemini surfactant solution and Lungu heavy oil; the viscosity is 1420 mPa s, and the viscosity reduction rate is 92.1%.

**2.3. Cationic Gemini Surfactant.** Liu Lang<sup>38</sup> used N,N-dimethylethylenediamine, sodium 2-bromoethylsulfonate, and bromododecane as raw materials to synthesize a gemini surfactant for Enping heavy oil. When the concentrations of gemini surfactant, OP-10, and heavy alkylbenzenesulfonate were 0.6, 0.4, and 0.4%, respectively, by adding 0.5% sodium carbonate to the system, a self-emulsifying viscosity reduction system for heavy oil with ultralow interfacial tension was developed. When the oil/water ratio is 3:7 and the temperature is 50 °C, the viscosity reduction rate of the self-emulsifying viscosity reducer system can reach 94.04%. When the temperature is higher than 50 °C, the oil–water interfacial tension of the self-emulsifying viscosity reducer system can be reduced to less than  $10^{-3}$  mN/m, but when the salinity is higher than 20000 mg/L, the performance of the system begins to deteriorate.

Pei Haihua et al.<sup>39</sup> synthesized a bis-quaternary ammonium surfactant DFA-12 with mixed tertiary amine and dibromoethane as raw materials according to the characteristics of high formation water salinity and high bottomhole temperature in the Tahe oilfield. The viscosity reduction effect was measured by rotating the viscometer at different shear rates for different viscous oils. The experiment shows that in the Tahe oilfield with a salinity of 214739.9 mg/L, under the conditions of a temperature of 90 °C and an oil–water ratio of 7:3, the viscosity reduction rate of the 0.25% DFA-12 + 0.25% HES + 0.1% DFP compound system for different viscosity superheavy oil in the Tahe oilfield is more than 98%.

Xu Dingda et al.<sup>40</sup> developed a compound pressure reduction and injection increase system by using nanosilicon as the main agent and cationic gemini surfactant as an additive to solve the problem of high injection pressure in an offshore heavy oil reservoir. The viscosimeter was used to measure the viscosity reduction effect after stirring at 250 rpm for 2 min. The experiment shows that the viscosity reduction rate can reach 93.7% at a temperature of 60 °C and an oil/water ratio of 7:3. The surface tension of the system is 22 mN/m, and the interfacial tension between oil and water is 0.065 mN/m after 30 days.

Wang Yining et al.<sup>41</sup> used alkyl dimethyl tertiary amine and bromoalkane to synthesize cationic gemini surfactant HXX-1 by a one-step process in order to solve the problem of rapid pressure rise in injection wells of heavy oil reservoirs. After the oil–water mixture with an oil–water ratio of 7:3 was kept constant at 60 °C for 1 h, the viscosity was determined by stirring it with a rotary viscometer at 250 rpm for 2 min. The results show that when the surfactant concentration is 0.05–0.5% the reduction rate is more than 90%, and the instantaneous interfacial tension between the surfactant and neutral kerosene can reach a  $10^{-3}$  mN·m<sup>-1</sup> order of magnitude.

**2.4. Other Types of Gemini Surfactants.** Ma Aiqing<sup>42</sup> developed gemini betaine zwitterionic surfactant GBS to solve

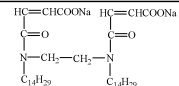
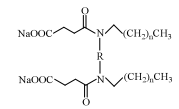
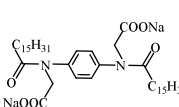
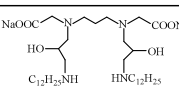
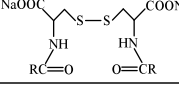

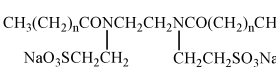
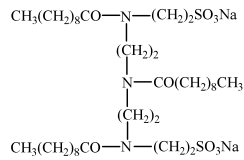
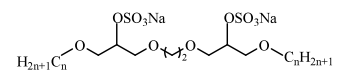
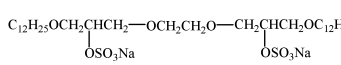
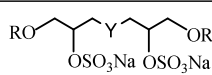
the problems of high water content and low recovery efficiency caused by water flooding of heavy oil. The viscosity of the oil–water mixture with an oil–water ratio of 7:3 was measured with a rotating viscometer at 50 °C for 1 h. The viscosity reduction rate of the surfactant with a mass concentration of 0.3% could reach 91.4%, and the interfacial tension of the oil–water mixture was as low as 0.08 mN/m. Compared with water flooding alone, the oil displacement efficiency can be increased by 8.31%. GBS aqueous solution with a mass concentration of 1% is injected into a water well in the field, and the average daily oil production of the oilfield increases from 2.2 to 4.5 t, which has a good oil displacement effect.

Yanping<sup>43,44</sup> used tetraethylene glycol as a spacer to connect two alkylphenol molecules through an etherification reaction to obtain a gemini skeleton containing an aromatic ring and then introduced a phenolic ether containing a polyoxyethylene chain into the gemini skeleton containing an aromatic structure. A series of nonionic gemini surfactants with aromatic spacers were synthesized. Surfactant E<sub>4</sub>A<sub>10</sub> has good surface activity, and its surface tension is 28.79 mN/m. After the oil–water mixture with an oil–water ratio of 7:3 was kept constant for 30 min at 50 °C, the viscosity of the mixture was determined by stirring it with a rotating viscometer at a rotational speed of 2000 rpm for 5 min. The experiment shows that the viscosity reduction rate of the two surfactants with a mass concentration of 1% was greater than 96.0% and the emulsification viscosity reduction effect was good. Surfactant E<sub>4</sub>A<sub>10</sub> has good salt tolerance, and its viscosity reduction rate is 98.4% when the salinity is 82460 mg/L. The interfacial tension between the heavy oil and the surfactant E<sub>4</sub>A<sub>10</sub> solution can still be maintained at 0.65 mN/m after the surfactant E<sub>4</sub>A<sub>10</sub> is treated at 300 °C. In addition, it is found that the longer the alkyl chain, the stronger the oil–water interfacial film. The increase in the chain length of the surfactant molecule polyether is beneficial to the formation of a relatively stable emulsion. The emulsifying effect of the system is more obvious, the system has higher shear resistance, and the emulsifying stability is greatly improved. However, with the increase in the number of spacer groups, the dispersion of molecules and droplets of the emulsified system decreased, and the emulsifying effect also decreased slightly.

In addition, some literature has not published the types of surfactants. For example, Wang Dawei et al.<sup>45</sup> used gemini surfactant RB107 to reduce the viscosity of heavy oil in an offshore S oilfield, aiming at the problems of high contents of resin and asphaltene and high viscosity in heavy oil in the offshore S oilfield. At 50 °C, the oil–water mixture with a 1:1 oil–water ratio was stirred with a rotary viscometer for 3 min to determine the viscosity. The experiment showed that when the mass concentration of the surfactant was 0.3%, the viscosity of the emulsion system decreased to 13.6 mPa·s and the viscosity reduction rate reached 97%. The results of the wettability test show that when the mass fraction of RB107 solution is 0.5%, the contact angle with crude oil is 10.8° and the interfacial tension between the mass fraction of 0.75% RB107 solution and crude oil is 0.1656 mN/m, which is higher than that of common surfactants.

Wu Ruidong et al.<sup>46</sup> screened more than 20 kinds of viscosity reducers to solve the problem of increasing water cut in Lungu deep heavy oil wells and conducted viscosity reduction experiments on the selected salt-tolerant and temperature-tolerant viscosity reducers. The viscosity of the mixture of oil and water with a 1:1 ratio of oil to water was

Table 2. Synthesis of Anionic Gemini Surfactants

Hydrophilic base type	Structural formula	Reaction time h	Reaction temperature °C	Charge ratio	Productivity %	Surface tension mN/m	Interfacial tension mN/m
carboxylate		22	60	3:1	86.6	27.5	-
		20	65	3:1	Over 50	Minimum 27.6	-
		40	10	1:2	76.8	31.36	0.00852
		90	14	1:2.2	60	16.81	0.0076
		10-15	1	1:2.1	80.5	-	0.032
Sulfonate		4	40	1:1	86.3	-	10 <sup>-5</sup>
		7	30	1:2.3	61.8	29.7	-
		12	50	1:3.2	75.2	27.49	0.3435
Sulfate		8	25	1:4	Over 60	30	0.604
		20	2	1:2.5	97	29.9	-
		35	2	1:2.5	Over 80	30	-

measured with a rheometer at different temperatures. The experimental results show that the viscosity reduction rate of LG15-2 heavy oil can reach 92% when the newly developed gemini surfactant DC848 is formulated with 258900 mg/L Lungu formation water and the dosage is 0.5%. When the dosage is 1.5%, the viscosity of the oil–water mixture is less than 200 mPa s in the temperature range of 40–70 °C, and the viscosity reduction rate is more than 99%, which has a good effect on the emulsification and viscosity reduction of Lungu heavy oil.

Zhang Yuna<sup>47</sup> used a self-made gemini surfactant to investigate the effect of temperature, emulsifier concentration, oil–water ratio, and stirring time on the viscosity reduction rate of heavy oil in the Panjin oilfield. The experimental results

show that the viscosity of Panjin heavy oil can be reduced from more than 820 to 12 mm<sup>2</sup>/s and the viscosity reduction rate can reach 98.5% when the oil–water ratio is 7:3, the temperature is 50 °C, the dosage is 0.5%, and the stirring time is 30 min.

When the concentration of CO<sub>2</sub> was low, Jing Bo et al.<sup>48</sup> used hexadecyl trimethylammonium hydroxide to react with acetic acid to generate a pseudo-gemini surfactant with CO<sub>3</sub><sup>2-</sup> combined with two CTA<sup>+</sup> hydrophobic chains. The oil–water interfacial tension of 1% surfactant solution reached the order of magnitude of 10<sup>-3</sup>. The oil–water mixture with a 1:1 oil–water ratio was manually shaken 50 times at 60 °C, and the viscosity was measured with a rotary viscometer at a shear rate of 6 r/min. The viscosity reduction rate was 98.68%.

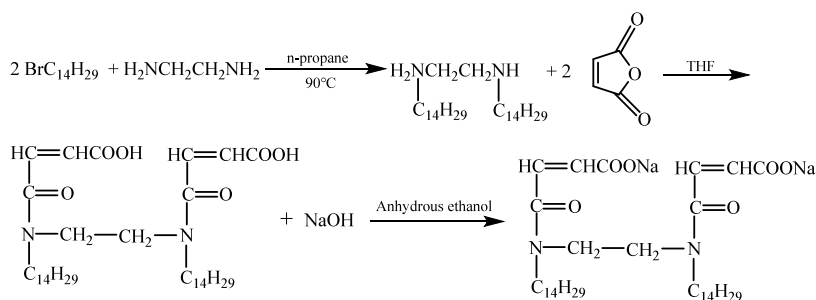


Figure 5. Synthesis route of gemini surfactant GMAS-14.

Liu Zhongyun et al.<sup>49</sup> used the synthesized gemini surfactant LZY-1 to investigate the influence of the viscosity reducer dosage, oil–water ratio, temperature, and rate on the viscosity reduction effect. The results showed that when the dosage of LZY-1 was 0.5%, the oil–water ratio was 3:7, the temperature was 50–60 °C, and the rate was 100–300 rpm. It can make the heavy oil in the LG9-1 well block form a low-viscosity emulsion.

An anionic gemini surfactant has good emulsification and wettability, strong interfacial activity, good compatibility with crude oil, good temperature resistance, less adsorption on sandstone surfaces, a simple production process, and low cost, and it is the earliest and most widely used in oilfield development.<sup>50,51</sup> According to the research data, compared with cationic gemini surfactants and other types of gemini surfactants, anionic gemini surfactants have stronger interfacial activity in high salinity (salinity >30000 mg/L) formation water containing calcium and magnesium ions, and the dosage of most surfactants is 0.4–0.5%. The viscosity reduction rate is more than 90%, and the interfacial tension can reach a  $10^{-3}$  order of magnitude. The interfacial tension of cationic gemini surfactants could reach a  $10^{-3}$  order of magnitude only after adding 0.4% OP-10, 0.5% alkali, and other additives, and the salt resistance of the viscosity reduction system was improved. In this paper, the synthesis and properties of anionic gemini surfactants were introduced.

### 3. SYNTHESIS OF ANIONIC GEMINI SURFACTANTS

Anions can be classified into carboxylate type, sulfonate type, and sulfate type according to different hydrophilic head-groups.<sup>52</sup> Table 2 summarizes the synthesis of anionic gemini surfactants with different hydrophilic groups in terms of the reaction temperature, reaction time, feed ratio, yield, and performance.

**3.1. Carboxylate Gemini Surfactant.** Carboxylate anionic gemini surfactant molecules are formed by connecting two carboxylic acid surfactant monomers through covalent bonds, and the main connecting groups are the ether bond, amide bond, carbon–nitrogen bond, and sulfur–sulfur bond.<sup>53</sup>

Ma Xiping<sup>54</sup> used ethylenediamine, bromotetradecane, and maleic anhydride as the main raw materials to synthesize anionic gemini surfactant GMAS-14 through a three-step reaction, and the resultant route is shown in Figure 5. The optimal reaction conditions of GMAS-14 were as follows: feed ratio (maleic anhydride, molar ratio of N,N'-didecyl ethylenediamine) 3:1, reaction temperature 60 °C, reaction time 22 h, and 86.6% yield of target product GMAS-14 under optimal synthesis conditions. The experimental results showed that the CMC of GMAS-14 was 0.048 mmol/L and the surface tension was 27.5 mN/m at 25 °C.

Yoshimura et al.<sup>55</sup> used fatty acyl chloride and ethylenediamine as raw materials to prepare N,N'-dialkyl dicarboxypropionyl ethylenediamine through acylation, LiAlH<sub>4</sub> reduction, and condensation, and the resultant route is shown in Figure 6.

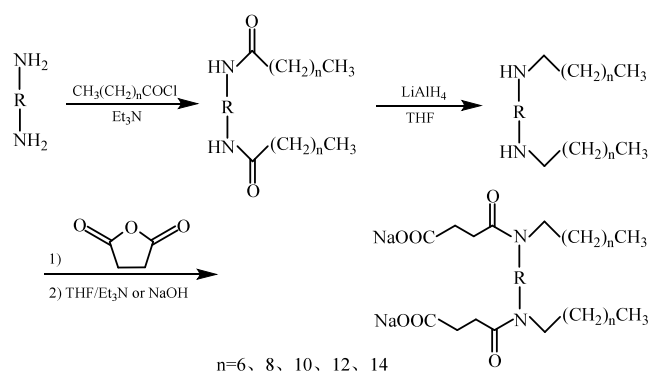


Figure 6. Synthesis route of N,N'-dialkyl bicarboxylpropionyl ethylenediamine.

Under the conditions of reaction temperature 65 °C, reaction time 20 h, and molar ratio of reactant as intermediate:succinic anhydride = 1:3, the synthetic yield is more than 50%. All five surfactants can effectively reduce the surface tension, and the minimum surface tension is 27.6 mN/m when  $n = 8$ .

Jin Lijun<sup>56</sup> used p-1,4-phenylenediamine, palmitoyl chloride, and sodium chloroacetate as raw materials to synthesize gemini carboxylate surfactant DC16-P-16 with an amide group through a two-step reaction, and the resultant route is shown in Figure 7. When the reaction temperature of the second step is 40 °C, the pH is 8, and the reaction time is 10 h, the yield is 76.8%. The results show that the surface tension of DC16-P-16 is 31.36 mN/m, and 0.5% DC16-P-16 can reduce the oil/water interfacial tension to  $8.52 \times 10^{-3}$  mN/m.

Wang Rui<sup>57</sup> used sodium chloroacetate, 1,3-propanediamine, dodecylamine, etc. as raw materials to synthesize a carboxylate anionic gemini surfactant through the substitution-ring-opening reaction, and the resultant route is shown in Figure 8. The optimal synthesis conditions were obtained by the control variable method: the ratio of propylene diamine to sodium chloroacetate was 1:2.2. The yield of N,N-bis (3-chloro-2-hydroxy-propane) sodium propylenediamine diacetate and dodecylamine was more than 60% when the feeding ratio was 1:2.2, the reaction temperature was 90 °C, and the reaction time was 14 h. The results show that the surface tension is 16.81 mN/m, and the oil–water interfacial tension can be reduced to  $7.6 \times 10^{-3}$  mN/m by 0.5% GAC-312 solution at 45 °C.

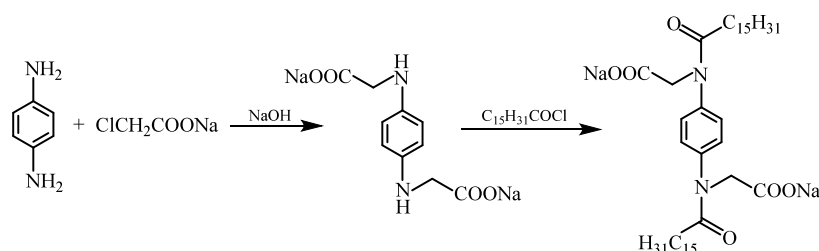


Figure 7. Synthesis route of gemini surfactant DC16-P-16.

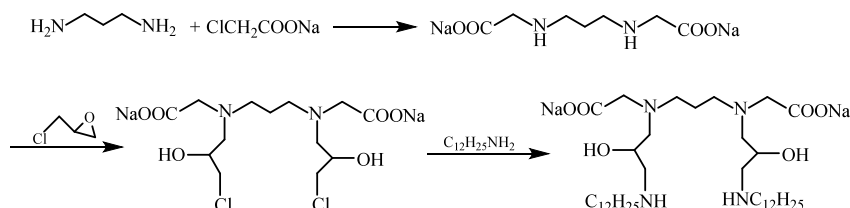


Figure 8. Synthesis route of gemini surfactant GAC-312.

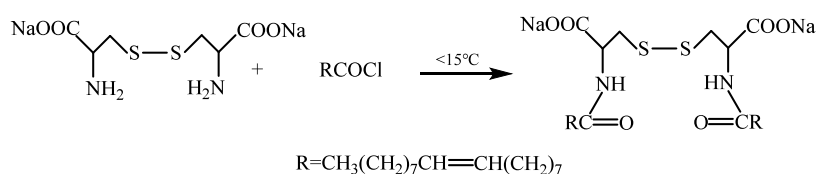


Figure 9. Synthesis route of sodium dioleamido cystine SDOLC.

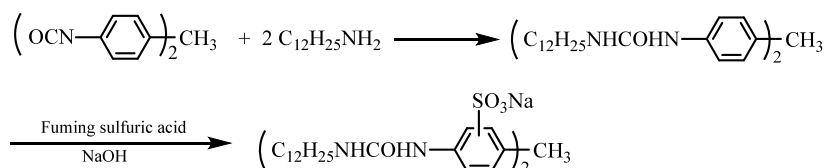


Figure 10. Synthesis route of gemini surfactant UAGS-12.

Fan Haiming<sup>58</sup> used sodium cystine and oleoyl chloride as raw materials to synthesize a new anionic gemini surfactant dioleamido sodium cystine (SDOLC) through a one-step reaction, and the resultant route is shown in Figure 9. When the pH was 8–10, the temperature was 10–15 °C, and the reaction time was 1 h, the yield was 80.5% after recrystallization with ethanol/ethyl acetate 2 times. The results showed that the SDOLC solution with a mass fraction of 0.10% was prepared by using formation water. Can the interfacial tension of 10<sup>−2</sup> mN/m order of magnitude with the crude oil of the Daqing oilfield under the condition of low alkali concentration be reached.

**3.2. Sulfonate Gemini Surfactant.** Sulfonate anionic gemini surfactants can be divided into an ester bond, ether bond, amide bond, and carbon–nitrogen bond according to the different bond groups in the structural formula during the synthesis process.<sup>59</sup>

Li Xiaoyang<sup>60</sup> synthesized anionic gemini surfactant (UAGS-12) with 4,4'-diphenylmethane diisocyanate and dodecylamine as raw materials, and the resultant route is shown in Figure 10. UAGS-12 reacted at 40 °C for 4 h with a yield of 86.3% and formed a mixed surfactant with nonionic surfactant laurate diethanolamide (6501). The experimental results show that when  $m(\text{UAGS-12}):m(6501) = 1:2$ , the oil–water interfacial

tension can reach below 10<sup>−5</sup> mN/m, and it still has excellent ability to reduce the interfacial tension under high salinity.

Wang Ping<sup>61</sup> synthesized the intermediate ethylenediamine N,N'-diethyl sodium sulfonate from ethylenediamine and 2-bromoethyl sodium sulfonate and then reacted it with fatty acyl chloride to synthesize a series of sulfonate gemini surfactants. The resultant route is shown in Figure 11. The

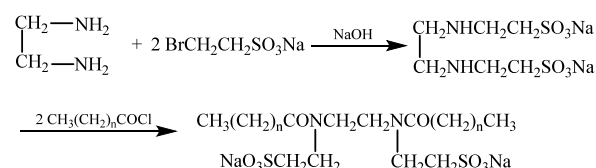


Figure 11. Synthesis route of the sulfonate gemini surfactant.

optimum reaction conditions were as follows: mixed solvent (V(water):V(acetone) = 2:1), reaction temperature 30 °C, and reaction time 7 h; the yield was 61.8%. The results show that the surface tension of DTM-12 is 29.7 mN/m.

Tian Lan<sup>62</sup> used diethylenetriamine, sodium 2-bromoethyl-sulfonate, and decanoyl chloride as the main raw materials to synthesize a new type of sulfonate gemini surfactant N,N,N',N'-amido diethylenetriamine diethylsulfonate sodium (TADS-10), and the resultant route is shown in Figure 12. Under the

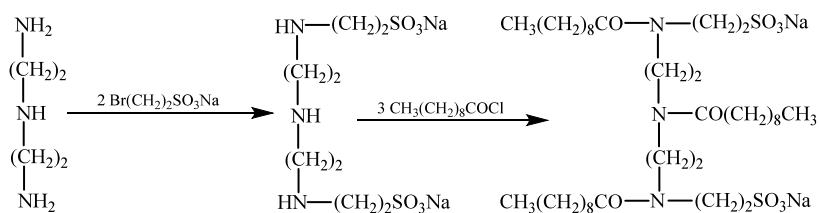


Figure 12. Synthesis route of gemini surfactant TADS-10.

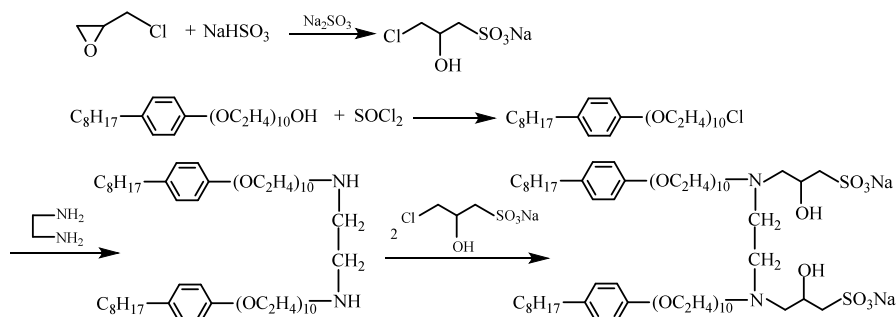


Figure 13. Synthesis route of disulfonate polyoxyethylene ether surfactant (DSEO).

conditions of reaction temperature 50 °C, reaction time 12 h, and molar ratio of reactant as intermediate:acyl chloride = 1:3.2, the synthesis yield was 75.2%. The results show that the surface tension is 27.49 mN/m, and the oil–water interface between TADS-10 with a concentration of 400 mg/L and crude oil is  $10^{-1}$  order of magnitude at 45 °C. The combination of TADS-10 with petroleum sulfonate and OP-10 has an obvious synergistic effect and can achieve ultralow interfacial tension ( $10^{-3}$  mN/m).

Xia Jijia<sup>63</sup> used ethylenediamine, epichlorohydrin, alkylphenol polyoxyethylene ether, and thionyl chloride as the main raw materials to synthesize a disulfonate polyoxyethylene ether surfactant (DSEO) through chlorination, alkylation, and four other steps of reaction, and the resultant route is shown in Figure 13. The experimental results showed that the surface tension was 31.7 mN/m. The complex system of DSEO with anionic surfactant SA and nonionic surfactant NB1214 has a good synergistic effect, and the oil–water interfacial tension can be reduced to  $10^{-3}$  and  $10^{-2}$  mN/m, respectively.

Mpelwa et al.<sup>64</sup> used fatty amine, epichlorohydrin, and sodium bisulfite as the main raw materials to synthesize the gemini surfactant m-3-m through a three-step reaction; the synthesis route is shown in Figure 14. The experimental results show that the gemini surfactant has good surface activity and can reduce the oil–water interfacial tension to  $10^{-3}$  mN/m within 30 min.

**3.3. Sulfate Gemini Surfactant.** Liu Jinhua<sup>65</sup> used long-chain fatty alcohol and epichlorohydrin as raw materials to prepare the corresponding alkyl glycidyl ether by an ether-

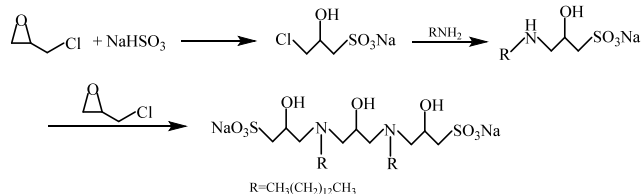


Figure 14. Synthesis route of the m-3-m surfactant.

ification reaction under alkaline conditions with TBAB as the catalyst and then carried out a coupling reaction with ethylene glycol to obtain the corresponding diol intermediate. Finally, in the presence of an excess of base, a sulfonation reaction was carried out to obtain four symmetric disulfonate sodium salt anionic gemini surfactants with different hydrophobic chain lengths; the resultant route is shown in Figure 15. When the

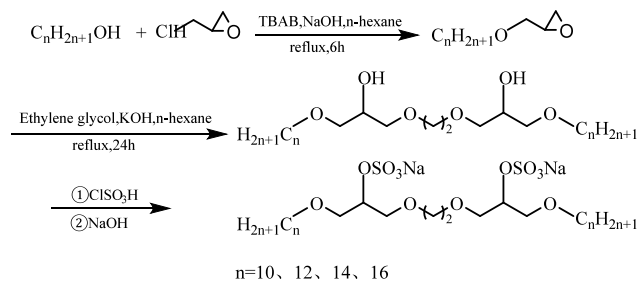
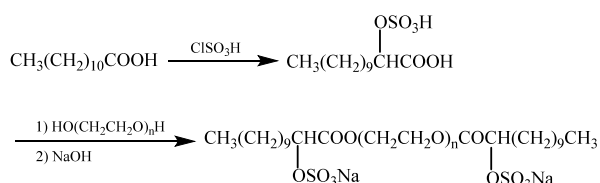


Figure 15. Synthesis route of the symmetric sodium disulfonate anionic gemini surfactant.

pH is 9–10, the temperature is 25 °C, and the reaction time is 8 h, the yield of extraction with petroleum ether is more than 60% after two times. The results show that the surface tension of LJH6 and LJH7 is generally below 30 mN/m in the mass concentration range of 0.4–1.5%, and the lowest interfacial tension of LJH8 is  $10^{-1}$ .

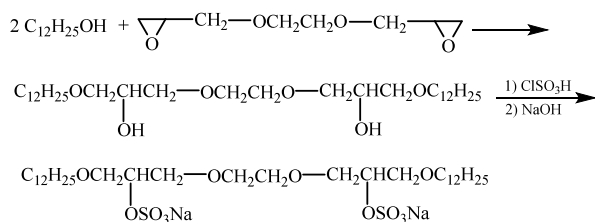
Sun Dong<sup>66</sup> used lauric acid and polyethylene glycol as the main raw materials to synthesize oligomeric glycol ( $\alpha$ -sodium sulfonate) lauric acid diester anionic gemini surfactant (GSS), and the resultant route is shown in Figure 16. The results show that the surface tension of the 1140 mg/L surfactant solution is 28.1 mN/m, the oil–water interfacial tension of the 500 mg/L GSS solution is 0.014 mN/m at 50 °C, and the surface tension is stable when the ratio of nonionic surfactant H and GSS is 3: it can reduce the oil–water interfacial tension in the Wuliwan area to ultralow ( $10^{-3}$  mN/m).

Guo Limei et al.<sup>67</sup> used dodecanol and ethylene glycol diglycidyl ether as raw materials and boron trifluoride etherate



**Figure 16.** Synthesis route of gemini surfactant GSS.

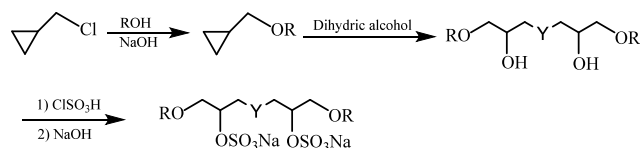
as a catalyst to prepare anionic gemini surfactant sodium didodecyl ethoxy disulfate through a three-step method, and the resultant route is shown in Figure 17. When the molar ratio



**Figure 17.** Synthesis route of sodium didodecyl ethoxy disulfate.

of reactant is the intermediate:chlorosulfonic acid = 1:2.5 with a 20 °C reaction for 2 h, the sulfonation rate reached 97%. The experimental results showed that the surface tension is 29.9 mN/m and the emulsifying ability is better than that of SDS.

Liu Xinjian et al.<sup>68</sup> used fatty alcohol, epichlorohydrin, and diol with different chain lengths as raw materials to synthesize four kinds of anionic gemini surfactants with nonionic spacer groups through a three-step reaction, and the resultant route is shown in Figure 18. When the pH was 10,  $n(\text{alkyl})$



**Figure 18.** Synthesis route of dialkyl ethoxy sodium sulfate surfactant.

oligodiol): $n(\text{chlorosulfonic acid}) = 1:2.5$ , the reaction was carried out at 35 °C for 2 h, and the yield was more than 80% after three extractions with mixed solvent of *n*-butanol–water. The results show that the surface tension of some products is lower than 30 mN/m.

#### 4. CONCLUSIONS AND PROSPECTS

Most anionic gemini surfactants have a good viscosity reduction effect when the dosage is 0.4–0.5% and have good salt tolerance in the range of salinity of 30000–60000 mg/L, which can achieve  $10^{-3}$  ultralow interfacial tension. In recent years, scientists have begun to study a large number of gemini surfactants and have made great progress. However, the synthesis steps of some anionic gemini surfactants are complicated and the synthesis conditions are harsh, which is not conducive to industrial production. At the same time, there are difficulties in separation and purification and low yield. The key research directions of gemini surfactants in the future are as follows. First, the synthesis route of most anionic gemini surfactants is 2 to 3 steps, and the synthesis steps should be simplified as far as possible. Second, cheaper raw materials should be selected to reduce the synthesis cost so as to realize

industrial mass production. Third, the synergistic effect of different types of surfactants should be studied in order to achieve strong matching with oil fields.

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#### Notes

The authors declare no competing financial interest.

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