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Synthesis and surface activity study of branched fluorinated cationic (FCS), gemini (FGS) and amphoteric (FAS) surfactants with $CF_3CF_2CF_2C(CF_3)_2$ group

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ABSTRACT

Fluorinated surfactants are usually composed of a perfluorinated chain and a hydrophilic group. Academic surveys have reported that straight chain fluorinated surfactants have the lowest surface tension in a relatively high concentration while the branched fluorinated surfactants show more efficient in a relatively low concentration. Introducing branch is one of effective strategies for synthesis of non-bioaccumulable fluorinated surfactants.

Three novel branched fluorinated surfactants were designed and prepared through a five-step route using perfluoro-2-methyl-2-pentene as starting material. The surface activities of them were investigated and found that they exhibited excellent surface activities. FCS can reduce the surface tension of water to below 20 mN/m, and the cmc value of FAS in water is about 1.04×10^{-4} mol/L at 298 K. All the values of surface properties of FCS, FGS and FAS are lower than that of sodium perfluorooctanoate.

1. Introduction

Fluorinated surfactants are more efficient than traditional surfactants since they display high surface activity at low critical micelle concentration (cmc). Besides, fluorinated surfactants usually have high thermal and chemical stability [1–3]. They are usually composed of a perfluorinated chain and a hydrophilic group [4–6]. The most known fluorinated surfactants are PFOA (perfluorooctanoic acid, $C_7F_{15}CO_2H$) and PFOS (perfluorooctane sulphonate, $C_8F_{17}SO_3X$, with X = K, Na, H). Up to now, fluorinated surfactants have been widely used in more than 200 applications [7] including clothing fabrics, electroplating, fire fighting foams, food packaging, petroleum, textile, etc.

However, compared with hydrocarbon surfactants, fluorinated surfactants are more difficult to synthesize. The methods reported are fluorination by electrolysis [8], telomerization of fluoroolefin [9–11], oligomerization of fluoroolefin [12], etc. Fluorination by electrolysis uses HF as a main raw material which is inexpensive, but the method has many by-products. Telomerization of fluoroolefin usually generates a mixture of products with different chainlength, therefore, strict control of the reaction conditions is required. The most commonly used fluoroolefin in oligomerization of fluoroolefin is hexafluoropropene.

With the forbidden of PFOA and PFOS for persistent, toxic and bioaccumulable pollutants [13–16], it is necessary to develop novel structural fluorinated surfactants. So far, basic research studies in this field revealed that straight chain fluorinated surfactants have the lowest surface tension at a relatively high concentration while the branched fluorinated surfactants show more efficient at a relatively low concentration. For example, Dmowski et al., prepared $CF_3CF_2CF_2C(CF_3)_2CH_2CH_2{-}COONa$ and compared its surface activity with $CF_3(CF_2)_6COONa$ [17]. The result showed that $CF_3CF_2CF_2C(CF_3)_2CH_2CH_2COONa$ has better surface ability than $CF_3(CF_2)_6COONa$ to reduce the surface tension of water at the same concentration. So, introducing branched structure in fluorinated chain part is one of effective strategies for synthesis of non-bioaccumulable alternatives to PFOA [18]. Hexafluoropropylene oligomer is an important starting material which can be used to synthesize many branched fluorinated surfactants and most reported methods are based on hexafluoropropylene trimer. The

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methods for preparing branched fluorinated surfactants using hexafluoropropylene dimmer are extremely scanty [19–25]. Besides, hexafluoropropylene dimmer is easy to get and relatively inexpensive.

In view of these facts mentioned above, we designed an approach for preparation of the title compounds using perfluoro-2-methyl-2-pentene as starting material. In this paper, we would like to report the synthesis and characterization of these novel compounds. Furthermore, evaluation of their surface tension was also reported.

2. Results and discussion

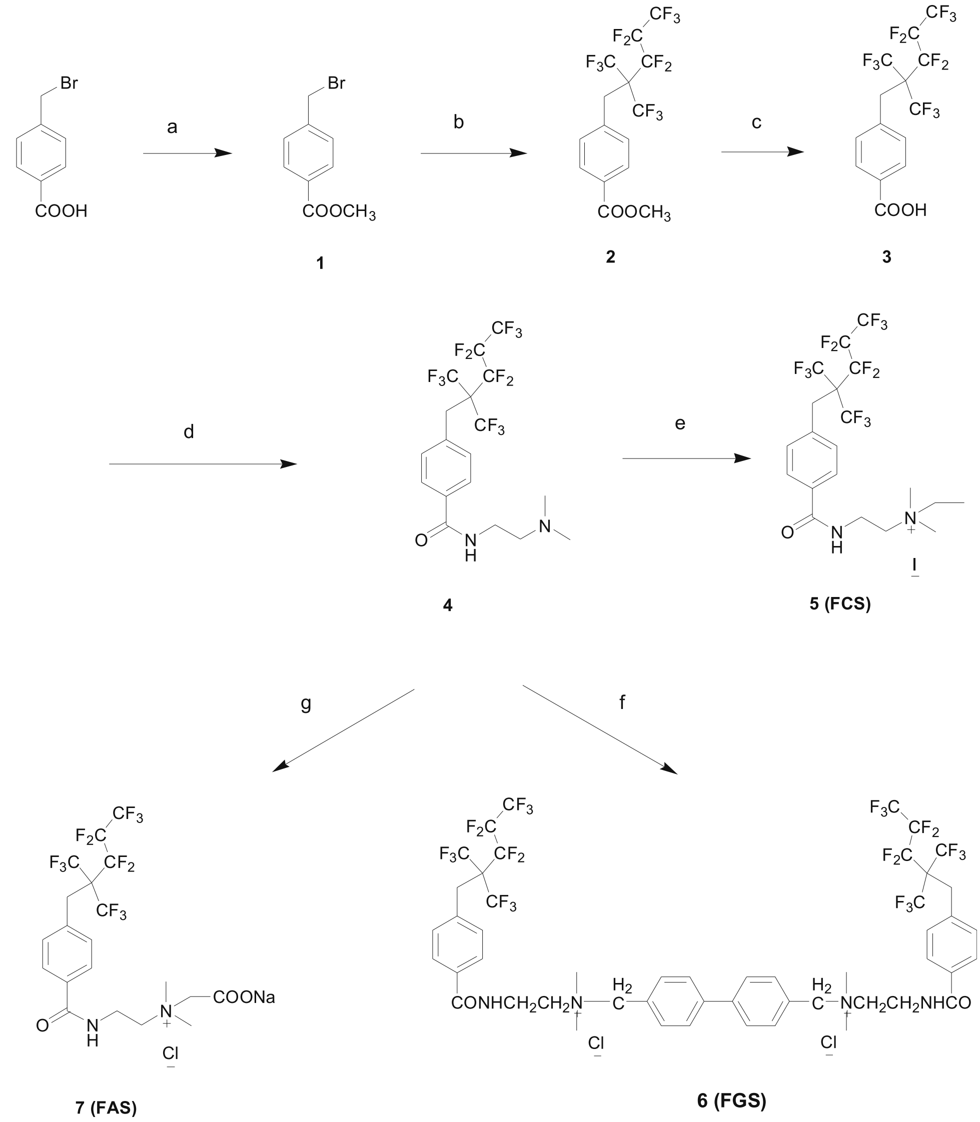
2.1. Synthesis

The synthesis pathway was shown in Scheme 1. Four steps led to the compound **4**. First, 4-(bromomethyl)benzoic acid was esterified with methanol to protect carboxyl. Second, compound **1** was changed to compound **2** by a nucleophilic substitution reaction. Third, saponification of the ester **2** followed by acidification led to the carboxylic acid **3**. Then, conversion of the acid **3** into the amide **4** was carried out by treating with EDCI and N,N-dimethylethylene diamine. Finally, the title branched fluorinated cationic (FCS), gemini (FGS) and amphoteric (FAS) surfactants were obtained in both high yields and high purity by the reactions of the tertiary amine intermediate **4** with appropriate alkyl halides in CH_3CN followed by filtration of the resultant precipitates. In each step, reaction proceeded under mild reaction conditions with high yield of the desired product and easy work-up procedures using low cost reagents. The structures of FCS, FGS, FAS and intermediates were characterized by ^1H NMR, ^{19}F NMR, ^{13}C NMR, FTIR and MS.

2.2. Surface tension

The surface tensions were tested at 25 °C by the Wilhemy plate method using a Kruss K100 tensiometer. A series of different concentrations of aqueous solution were prepared, average value of the surface tension at each concentration was obtained based on the data collection by three times measurement. The tensions were measured followed by concentrations from low to high. Results were shown in Fig. 1.

As is seen in Fig. 1, the cmc value of FCS in water is about 2.5×10^{-3} mol/L at 298 K and the surface tension of the aqueous solution is 19.678 mN/m at the cmc. The cmc value of FGS in water is about 3.4×10^{-4} mol/L at 298 K and the surface tension of the aqueous solution is 22.381 mN/m at the cmc. The cmc value of FAS



Scheme 1. Synthetic route to produce branched fluorinated surfactants.

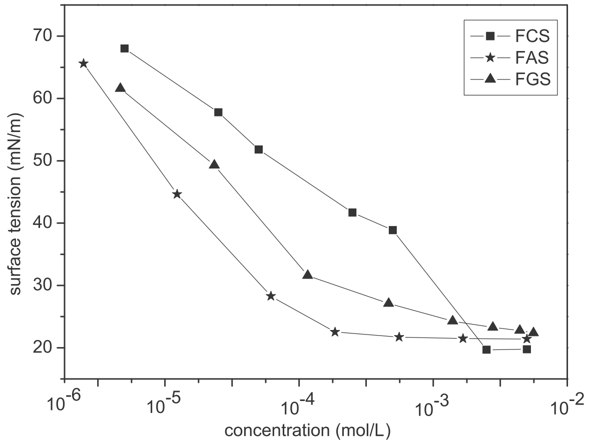


Fig. 1. Surface tension values at 298 K.

in water is about 1.04×10^{-4} mol/L at 298 K and the surface tension of the aqueous solution is 21.390 mN/m at the cmc.

The results showed that FCS exhibited the best ability to reduce the surface tension of water while FAS exhibited the best efficiency to reduce the surface tension of water. Besides, all the values of their surface properties are lower than that of sodium perfluorooctanoate (about 24.7 mN/m at the cmc 3.1×10^{-2} mol/L) [26]. Their low surface tension and cmc at room temperature make them potential surfactants.

Comparing the surface properties of FCS with the surfactant $C_9F_{17}OC_6H_4CONH{-}C_3H_6N^+(CH_3)_3I^-$ (about 20.4 mN/m at the cmc 9.7×10^{-4} mol/L) [27], we see that they exhibit similar ability to reduce the surface tension of water.

3. Conclusions

Novel branched fluorinated cationic (FCS), gemini (FGS) and amphoteric (FAS) surfactants were designed and prepared through a five-step route using perfluoro-2-methyl-2-pentene as starting material. All the steps were easy work-up, mild reaction conditions, low cost and high yields. The surface activities of FCS, FGS and FAS were investigated. FCS exhibited excellent ability to reduce the surface tension of water to below 20 mN/m. FAS exhibited excellent efficiency to reduce the surface tension of water, that is, the cmc value of FAS in water is about 1.04×10^{-4} mol/L at 298 K. All the values of surface activities of the three novel surfactants are lower than that of sodium perfluorooctanoate. Their low surface tension and cmc at room temperature make them potential surfactants. Comparing the surface properties of FCS with the surfactant of similar structure which was synthesized from hexafluoropropylene trimer, we see that hexafluoropropylene dimer can also be used to synthesize branched fluorinated surfactants. On the basis of these encouraging results, we will develop further new surfactants which have the branch-chained group $CF_2CF_2CF_2C(CF_3)_2CH_2C_6H_4{-}$, characterize their behavior in solution and use them in special applications.

4. Experimental

4.1. Materials

Perfluoro-2-methyl-2-pentene was purchased from KALF (SUQIAN) NEW MATERIAL CO., LTD (99% purity). Other chemical reagents were AR grade quality and used without further purification in all cases. Solvents were AR grade quality and dried by the standard methods before use. The surface tensions were tested at 25 °C by the Wilhemy plate method using a Kruss K100 tensiometer. Flash chromatography was performed using silica gel H (10–40 μm). ^{1}H and ^{19}F NMR were collected on Varian Mercury-vx300 spectrometer. ^{13}C NMR was recorded on Varian Mercury-400 spectrometer. IR spectra was measured using Nicolet 380FT-IR instrument and was reported in wave-numbers (cm^{-1}). LRMS was performed on Agilent 5973N mass instrument (EI) or AXIMA Performance MALDI-TOF/TOF MS (MALDI). HRMS was performed on Waters Micromass GTC Premier (EI) or IonSpec 4.7 T FTMS (MALDI). The known compounds were identified by comparison of their physical and spectral data with those reported in the literature.

4.2. Synthesis of the branched fluorinated surfactants

4.2.1. Synthesis of 4-bromomethyl-benzoic acid methyl ester (**1**) [28]

4-(Bromomethyl)benzoic acid (2.140 g, 10.0 mmol, 1.0 equiv), 4-dimethylaminopyridine (DMAP, 0.122 g, 1.0 mmol, 0.1 equiv) and N,N'-dicyclohexylcarbodiimide (DCC, 3.090 g, 15.0 mmol, 1.5 equiv) were combined and cooled to 0 °C. Dry CH_2Cl_2 (40.0 mL) was added and stirred for 10 min. MeOH (4.0 mL) was then added dropwise with stirring. The resulting solution was stirred at room temperature while the course of the reaction was monitored by TLC (petroleum ether: EtOAc = 9:1). TLC showed that the reaction was completed after 5 h. At this point, the solution was filtered and the filtrate was washed with 1 M $KHSO_4$, distilled water, and brine. The organic solution was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The product was purified by column chromatography (EtOAc:petroleum ether = 1:20).

1: white solid, 1.942 g (85% yield); ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.92 (s, CH_3, 3H), 4.50 (s, CH_2, 2H), 7.46 (d, J = 8.4 Hz, Ar-H, 2H), 8.01 (d, J = 8.4 Hz, Ar-H, 2H); LRMS (EI) m/z (%): 149 (100), 228 (4.5), 230 (3.8).

4.2.2. Synthesis of 4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis-trifluoromethyl-pentyl)-benzoic acid methyl ester (**2**)

Perfluoro-2-methyl-2-pentene (15.780 g, 52.6 mmol, 2.0 equiv), compound **1** (6.000 g, 26.3 mmol, 1.0 equiv), CsF (6.000 g, 39.5 mmol, 1.5 equiv) and dry CH_3CN (40.0 mL) were placed in a sealed glass tube and heated at 50 °C for 2 days with stirring. The reaction mixture was poured into ether, washed with portions of water and brine. The organic solution was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The product was purified by column chromatography (EtOAc:petroleum ether = 1:48).

2: white solid, 11.694 g (95% yield); ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.58 (s, CH_2, 2H), 3.91 (s, CH_3, 3H), 7.37 (d, J = 8.4 Hz, Ar-H, 2H), 7.98 (d, J = 8.4 Hz, Ar-H, 2H); ^{19}F NMR ($CDCl_3$, 282 MHz) δ: −62.65 to −62.85 (m, 6F), −80.57 (t, J = 13.7 Hz, 3F), −106.20 to −106.60 (m, 2F), −123.30 to −123.60 (m, 2F); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 32.7, 52.3, 105–127, 129.6, 130.3, 131.7, 136.2, 166.7; IR (cm^{-1}): 2961.9, 1724.9; LRMS (EI) m/z (%): 437 (100), 468 (24.7); HRMS (EI) calcd for $C_{15}H_9F_{13}O_2$ 468.0395, found 468.0396.

4.2.3. Synthesis of 4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis-trifluoromethyl-pentyl)-benzoic acid (**3**) [29]

A solution of compound **2** (2.340 g, 5.0 mmol, 1.0 equiv) in tetrahydrofuran (50.0 mL) was cold to 0 °C, 0.5 N LiOH (0.252 g, 6.0 mmol, 1.2 equiv) in water (12.0 mL) was added dropwise with stirring. The resulting solution was stirred at room temperature for 12 h. Water was then added and the solution was washed with ether. The aqueous layer was collected and acidified with 1 M $KHSO_4$ to PH $\approx$ 3. The aqueous layer was then extracted with ether. The organic layer was combined, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo to give the product **3**.

3: white solid, 2.253 g (99% yield); ^{1}H NMR (DMSO-d_6, 300 MHz) δ: 3.82 (s, CH_2, 2H), 7.45 (d, J = 7.8 Hz, Ar-H, 2H), 7.92 (d, J = 7.8 Hz, Ar-H, 2H); ^{19}F NMR (DMSO-d_6, 282 MHz) δ: −61.95 to −62.20 (m, 6F), −80.04 (t, J = 13.1 Hz, 3F), −106.00 to −106.30 (m, 2F), −122.90 to −123.20 (m, 2F); LRMS (EI) m/z (%): 135 (100), 454 (4.7).

*4.2.4. Synthesis of N-(2-dimethylamino-ethyl)-4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis-trifluoromethyl -pentyl)-benzamide (**4**)*

Acid **3** (5.000 g, 11.0 mmol, 1.0 equiv), DMAP (0.135 g, 1.1 mmol, 0.1 equiv) and 1-ethyl-3-(3-dimethyllaminopropyl)carbodiimide hydrochloride (EDCI, 3.150 g, 16.5 mmol, 1.5 equiv) were combined and cooled to 0 °C. Dry CH_2Cl_2 (50.0 mL) was added and stirred for 10 min. N,N-dimethylethylene diamine (1.8 mL, 16.5 mmol, 1.5 equiv) was then added dropwise with stirring. The resulting solution was stirred at room temperature while the course of the reaction was monitored by TLC (CH_2Cl_2:CH_3OH = 9:1). TLC showed that the reaction was completed after 5 h. At this point, the solution was washed with distilled water and brine. The organic solution was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The product was purified by column chromatography (CH_2Cl_2:CH_3OH:Et_3N = 20:1:trace).

4: white solid, 5.126 g (89% yield); ^{1}H NMR (DMSO-d_6, 300 MHz) δ: 2.16 (s, N(CH_3)$_2$, 6H), 2.38 (t, J = 6.6 Hz, $CH_2CH_2N(CH_3)_2$, 2H), 3.34 (m, $CH_2CH_2N(CH_3)_2$, 2H), 3.80 (s, $C_6F_{13}CH_2C_6H_4$, 2H), 7.41 (d, J = 8.1 Hz, Ar-H, 2H), 7.80 (d, J = 8.1 Hz, Ar-H, 2H), 8.44 (t, J = 5.4 Hz, NH, 1H); ^{19}F NMR ($CDCl_3$, 282 MHz) δ: −62.56 to −62.76 (m, 6F), −80.41 (t, J = 13.7 Hz, 3F), −106.15 to −106.55 (m, 2F), −123.25 to −123.60 (m, 2F); ^{13}C NMR (DMSO-d_6, 100 MHz) δ: 31.3, 37.4, 45.2, 58.1, 126.9, 131.5, 133.7, 134.3, 165.5; IR (cm^{-1}): 3269.3, 1629.5; LRMS (EI) m/z (%): 58 (100), 523 (0.4); HRMS (EI) calcd for $C_{18}H_{16}F_{13}N_2O$ 523.1055, found 523.1056.

*4.2.5. Synthesis of ethyl-{2-[4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis-trifluoromethyl-pentyl)-benzoylamino]-ethyl}-dimethyl-ammonium iodide (**5, FCS**)*

Compound **4** (0.646 g, 1.2 mmol, 1.0 equiv) and iodoethane (0.374 g, 2.4 mmol, 2.0 equiv) were refluxed in MeCN (7.0 mL) under argon atmosphere. The course of the reaction was monitored by TLC (CH_2Cl_2:CH_3OH = 24:1). TLC showed that the reaction was completed after 3 h. Then the mixture was cooled to room temperature. After the solution was evaporated under reduced pressure, the residue was washed with acetone. The resulting white solid was dried under reduced pressure.

5: white solid, 0.812 g (99% yield); ^{1}H NMR (DMSO-d_6, 300 MHz) δ: 1.27 (t, J = 6.3 Hz, $N(CH_3)_2CH_2CH_3$, 3H), 3.09 (s, N(CH_3)$_2$, 6H), 3.38–3.50 (m, $NHCH_2CH_2N(CH_3)_2CH_2CH_3$, 4H), 3.60–3.70 (m, $CH_2CH_2N(CH_3)_2$, 2H), 3.81 (s, $C_6F_{13}CH_2C_6H_4$, 2H), 7.45 (d, J = 8.1 Hz, Ar-H, 2H), 7.82 (d, J = 8.1 Hz, Ar-H, 2H), 8.82 (t, J = 5.0 Hz, NH, 1H); ^{19}F NMR (DMSO-d_6, 282 MHz) δ: −62.00 to −62.22 (m, 6F), −80.09 (t, J = 13.3 Hz, 3F), −105.90 to −106.40 (m, 2F), −122.90 to −123.10 (m, 2F); ^{13}C NMR (CD_3OD, 100 MHz) δ: 8.7, 33.2, 34.9, 51.5, 61.5, 62.5, 128.3, 133.1, 134.6, 136.6, 169.6; IR (cm^{-1}): 3279.5, 3202.2, 1650.7, 1455.2; LRMS (MALDI) m/z 553.1 (M−I); HRMS (MALDI) calcd for $C_{20}H_{22}F_{13}N_2O$ (M−I) 553.1519, found 553.1510.

*4.2.6. Synthesis of [1,1′-biphenyl]-4,4′-dimethanaminium, N^4,N^4,N$^{4'}$,N$^{4'}$-tetramethyl-N^4,N$^{4'}$-bis-{2-[4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis-trifluoromethyl-pentyl)-benzoylamino]-ethyl}-, chloride (1:2) (**6, FGS**)*

Compound **4** (1.569 g, 3.0 mmol, 3.0 equiv) and 4,4′-bis(chloromethyl)-1,1′-biphenyl (0.251 g, 1.0 mmol, 1.0 equiv) were refluxed in MeCN (3.0 mL) under argon atmosphere for 2 days. Then the mixture was cooled to room temperature and filtered. The solid was washed twice with CH_2Cl_2, then dissolved in MeOH. Again the MeOH solution was filtered and the filtrate was concentrated in vacuo leaving a white solid. The solid was washed with acetone and dried giving a white powder.

6: white powder, 1.024 g (79% yield). ^{1}H NMR (CD_3OD, 300 MHz) δ: 3.20 (s, N(CH_3)$_2$, 12H), 3.61 (t, J = 6.5 Hz, $CONHCH_2CH_2$, 4H), 3.77 (s, $(CH_3)_2NCH_2C_6H_4$, 4H), 3.98 (m, $CONHCH_2CH_2$, 4H), 4.71 (s, $C_6H_4CH_2C_6F_{13}$, 4H), 7.49 (d, J = 8.1 Hz, Ar-H, 4H), 7.75 (d, J = 8.1 Hz, Ar-H, 4H), 7.81–7.91 (m, $CH_2C_6H_4C_6H_4CH_2$, 8H); ^{19}F NMR (CD_3OD, 282 MHz) δ: −63.60 to −63.85 (m, 12F), −82.02 (t, J = 13.8 Hz, 6F), −106.80 to −107.20 (m, 4F), −124.00 to −124.30 (m, 4F); ^{13}C NMR (CD_3OD, 100 MHz) δ: 33.2, 35.1, 50.8, 63.6, 69.1, 110–125, 128.4, 128.4, 128.9, 133.1, 134.6, 135.0, 136.6, 143.5, 169.6; IR (cm^{-1}): 3424.8, 3035.7, 1655.7, 1546.5, 1504.3, 1484.4; LRMS (MALDI) m/z 748.1 (M-2Cl-$CH_2CH_2NHCOC_6H_4CH_2C_6F_{13}$), 479.8 ($C_6F_{13}CH_2C_6H_4CONHCH_2CH_2$); HRMS (MALDI) calcd for $C_{34}H_{35}F_{13}N_3O$ (M-2Cl-$CH_2CH_2NHCOC_6H_4CH_2C_6F_{13}$) 748.2562, found. 748.2567.

*4.2.7. Synthesis of ethanaminium, N-(carboxymethyl)-N,N-dimethyl-2-[4-(3,3,4,4,5,5,5-heptafluoro-2,2-bis-trifluoromethyl-pentyl)-benzoylamino]-, chloride, sodium salt (1:1:1) (**7, FAS**)*

Compound **4** (1.151 g, 2.2 mmol, 1.1 equiv), $ClCH_2COONa$ (0.232 g, 2.0 mmol, 1.0 equiv) and MeCN (20.0 mL) were placed in a sealed glass tube and refluxed for 2 days with stirring under argon atmosphere. Then the mixture was cooled to room temperature and filtered. The solid was washed twice with CH_2Cl_2, then dissolved in MeOH. Again the MeOH solution was filtered and the filtrate was concentrated in vacuo leaving a white solid. The solid was washed with acetone and dried giving a white powder.

7: white powder, 1.024 g (80% yield). ^{1}H NMR (CD_3OD, 300 MHz) δ: 3.33 (s, N(CH_3)$_2$, 6H), 3.76 (s, CH_2COONa, 2H), 3.80–3.90 (m, $NHCH_2CH_2N(CH_3)_2CH_2COONa$, 4H), 3.92 (s, $C_6F_{13}CH_2C_6H_4$, 2H), 7.46 (d, J = 7.8 Hz, Ar-H, 2H), 7.83 (d, J = 7.8 Hz, Ar-H, 2H); ^{19}F NMR (CD_3OD, 282 MHz) δ: −63.60 to −63.90 (m, 6F), −82.04 (t, J = 14.0 Hz, 3F), −106.90 to −107.20 (m, 2F), −124.10 to −124.40 (m, 2F); ^{13}C NMR (CD_3OD, 100 MHz) δ: 33.2, 35.2, 52.3, 62.8, 63.4, 65.5, 128.4, 133.1, 134.8, 136.5, 168.8, 169.7; IR (cm^{-1}): 3197.0, 3028.4, 2919.2, 1654.2, 1625.5, 1545.1, 1449.7; LRMS (MALDI) m/z 605.0 (M−Cl). HRMS (MALDI) calcd for $C_{20}H_{19}F_{13}N_2O_3Na$ (M−Cl) 605.1080, found 605.1072.

Supporting information

^{1}H NMR, ^{19}F NMR and ^{13}C NMR for the novel branched fluorinated cationic (FCS), gemini (FGS), amphoteric (FAS) surfactants and all the intermediates are provided.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jfluchem.2014.11.005.

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