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# Amphiphile self-assemblies in supercritical $CO_2$ and ionic liquids

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Supercritical (sc) $CO_2$ and ionic liquids (ILs) are the very attractive green solvents with tunable properties. Using sc$CO_2$ and ILs as alternatives of conventional solvents (water and oil) for forming amphiphile self-assemblies has many advantages. For example, the properties and structures of the amphiphile self-assemblies in these solvents can be easily modulated by tuning the properties of solvents; sc$CO_2$ has excellent solvation power and mass-transfer characteristics; ILs can dissolve both organic and inorganic substances and their properties are designable to satisfy the requirements of various applications. Therefore, the amphiphile self-assemblies in sc$CO_2$ and ILs have attracted considerable attention in recent years. This review describes the advances of using sc$CO_2$ or/and ILs as amphiphile self-assembly media in the last decade. The amphiphile self-assemblies in sc$CO_2$ and ILs are first reviewed, followed by the discussion on combination of sc$CO_2$ and IL in creating microemulsions or emulsions. Some future directions on the amphiphile self-assemblies in sc$CO_2$ and ILs are highlighted.

## 1 Introduction

Surfactants are an important class of molecules. They have wide applications in chemical industry, material science, biology, and enhanced oil recovery. It is well known that surfactants have the ability to self-assemble into various morphologically different structures, such as micelles, reverse micelles, vesicles, liquid crystals, etc. The conventional solvents for forming surfactant assemblies are water and oil (organic solvent). However, the solvency of water and organic solvent to many compounds are limited, and organic solvent suffers from environmental costs. Moreover, the properties and structures of the self-assemblies formed in conventional solvents cannot be well tuned by the solvent itself, and external stimulus (e.g. solid or liquid additives, heat, light, radiation) are needed instead.[1-4] Therefore, although water has many advantages such as abundant, cheap, and non-toxic, and surfactant aqueous solutions have been widely used, creation of surfactant assemblies and controlling their structures in tunable and functional solvents is desirable in many cases.

Supercritical (sc) $CO_2$ and ionic liquids (ILs) are very attractive green solvents with tunable properties. sc$CO_2$ is readily available, inexpensive, nontoxic, nonflammable, has moderate critical temperature and pressure (31.1 ºC and 7.38 MPa), and can be easily recaptured and recycled after use. Moreover, sc$CO_2$ has adjustable solvent power and excellent mass transfer characteristics that offer the opportunity to replace conventional organic solvents in a variety of applications.[5-8] Room-temperature ILs, which are organic salts with melting points below 100 ºC, have received much attention. In comparison with the conventional solvents, ILs have some unique properties. For example, ILs are an interesting class of tunable and designable solvents with essentially zero vapor pressure, wide electrochemical window, nonflammability, high thermal stability, and wide liquid range. Such unique properties confer ILs great potential of applications in different fields such as chemical reactions, extraction and fractionation, and material synthesis.[9-14]

Using sc$CO_2$ and IL as alternatives of conventional solvents for forming amphiphile self-assemblies has many advantages. The most outstanding feature is that the properties and structures of the amphiphile self-assemblies in these tunable solvents can be easily modulated by the solvent properties (e.g. $CO_2$ pressure, IL structure). Besides, sc$CO_2$ has strong solvent power, low viscosity, and fast mass-transfer rates; ILs can dissolve both organic and inorganic substances and their properties are designable to satisfy the requirements of various applications. Therefore, the amphiphile self-assemblies in sc$CO_2$ and ILs have attracted much attention in recent years.

This review focuses on the use of sc$CO_2$ and ILs as amphiphile self-assembly media in the last decade (from 2004 to early 2014). The self-assemblies in sc$CO_2$ and ILs are first reviewed, respectively, including the formation of reverse micelles, micelles, vesicles, liquid crystallines, microemulsions and emulsions with water or oil. Then the combination of sc$CO_2$ and IL for the formation of microemulsions or emulsions is discussed. At the end of this paper, we give an outlook for the future researches on the amphiphile self-assemblies in sc$CO_2$ and ILs.

## 2 Amphiphile self-assemblies in $scCO_2$

The design of surfactants compatible with $CO_2$ is crucial for the amphiphile self-assembly in $scCO_2$. The effective compounds that have been reported to form self-assemblies in $scCO_2$ are the partially or fully fluorinated surfactants, especially the fluorinated sodium bis(2-ethylhexyl)sulfosuccinate (AOT) analogue, the hydrocarbon surfactants, and hybrid fluorocarbon-hydrocarbon surfactants.[15]

### 2.1 Reverse micelles in $scCO_2$

Fluorosurfactants have been designed for compatibility with $scCO_2$.[16-20] The fluorinated chains represent low cohesive energy density groups, thereby promoting low solubility parameters and low polarizability. For example, Sawada[18] prepared reverse micelles of perfluoro 2,5,8,11-tetramethyl-3,6,9,12-tetraoxapentadecanoic acid ammonium salt in $CO_2$, which had a high potential to solubilize conventional acid dyes and to dye wool fabrics. Liu and coworkers[19] synthesized a series of fluorinated AOT analogue surfactants for reverse micelle formation in $scCO_2$. Berkowitz et al.[16] and Senapati et al.[20] used the molecular dynamics simulation to study the formation of reverse micelles of fluorosurfactants in $scCO_2$.

Although high $CO_2$ compatibility can be achieved by fluorinated surfactants, the cost of fluorinated compounds is high and they are toxic. On consideration of the environmental and economical factors, many nonfluorinated $CO_2$-compatible surfactants were designed, including AOT analogue surfactants (sodium dibutyl sulfosuccinate, sodium dipentyl sulfosuccinate, sodium dihexyl sulfosuccinate),[21] Dodecyl polyoxyethylene(4) polyoxypropylene(5) ether (Ls45),[22] triple hydrocarbon chain surfactant, sodium 1,4-bis(neopentyloxy)-3-(neopentyloxycarbonyl)-1,4-dioxobutane-2-sulfonate (TC14),[23] and AOK (aerosol-octyl-ketone, a non-fluorinated hydrocarbon surfactant with two symmetrical carbonyl groups in the tail).[24]

The hybrid fluorocarbon-hydrocarbon surfactants were used for reverse micelle formation in $scCO_2$. Ikushima et al.[25] investigated the micellization of AOT in $scCO_2$ with fluorinated co-surfactant, 2,2,3,3,4,4,5,5-octafluoro-1-pentanol (F-pentanol). It was found that AOT could be solubilized in $scCO_2$ with smaller amount of F-pentanol compared with ethanol and 1-pentanol, because F-pentanol has '$CO_2$-philic' fluorinated alkane chain. Can et al.[26] investigated the phase behaviour of the fluorocarbon-hydrocarbon oligomers in $scCO_2$. Spherical micelles can be generated in the 5%–65% volume fraction range for the oligomer, with the highest number of spherical micelles occurring at 45% surfactant in $CO_2$. When the volume fraction of the surfactant is increased from 70% to 85%, cylindrical micelles are formed.

### 2.2 $scCO_2$-continuous microemulsions

A microemulsion is a thermodynamically stable dispersion of two immiscible fluids stabilized by surfactant. There are roughly three types of conventional microemulsions, including water-in-oil, bicontinuous and oil-in-water microemulsions. Up to now, for most of the $scCO_2$-continuous microemulsions, water is used as the dispersed phase, forming the water-in-$CO_2$ microemulsions. Zhang and Han[27] reviewed the advance in the $scCO_2$-continuous microemulsions up to 2009, including the structures and properties of $scCO_2$-continuous microemulsions,[28-37] and their applications in nanoparticle synthesis[38-47] and chemical reactions.[48-52]

Since 2009, many efforts have been devoted to the formation, properties and applications of $scCO_2$-continuous microemulsions. In 2010, Sagisaka et al.[53] synthesized a novel double-tail anionic surfactant, sodium bis(1*H*,1*H*,2*H*,2*H*-heptadecafluorodecyl)-2-sulfoglutaconate ($8FG(EO)_2$), and formed water-in-$scCO_2$ microemulsion stabilized by $8FG(EO)_2$. $8FG(EO)_2$ was found to microemulsify at least 52 water molecules by one surfactant at 45 $^oC$ and pressures higher than 195 bar. By nearinfrared spectrum study, Takebayashi et al.[54] revealed that in water-in-$scCO_2$ microemulsion, water is distributed mainly in $CO_2$ at water contents smaller than 0.5 wt% and then is introduced into the microemulsion after saturation of water in $CO_2$ and full hydration of the surfactant headgroup. In 2011, Klostermann et al.[55] determined the bending rigidity of surfactant membranes in bicontinuous $CO_2$-microemulsions. It was found that the stiffness of the membrane increases solely by an increase of the pressure. In 2014, Eastoe et al.[56] reported the hydrotrope-induced morphological transitions in water-in-oil and water-in-$CO_2$ microemulsions stabilized by a trichain anionic surfactant TC14. The addition of a small amount of hydrotrope leads to a significant elongation in the microemulsion water droplets. Most recently, Yin's group[57-60] has done a series of work on the water-in-$scCO_2$ microemulsion formation using different surfactants (poly(ethylene glycol) TMN-6,[57] Ls-45,[58] Ls-36,[59] AOT[60]) and ethanol as cosurfactant. 1,3-propanediol was able to be extracted selectively from the dilute aqueous solution by using $scCO_2$ microemulsion.

### 2.3 Emulsions in $CO_2$/water

Emulsions are mixtures of immiscible liquids (usually water and organic solvent) that form two phases: the disperse phase as liquid droplets and the surrounding continuous phase. Different from the thermodynamically stable microemulsion, emulsions are thermodynamically unstable. Emulsions of $CO_2$ and water, the two most plentiful solvents on earth, are of interest. Johnston's group has done many works on this topic.[61-64] They utilized poly(1,1-dihydroperfluorooctyl methacrylate)-*b*-poly(ethylene oxide) (PFOMA-b-PEO) copolymer stabilizers to suppress the flocculation for water-in-$CO_2$ miniemulsions,[61] stabilized the concentrated $CO_2$-in-water emulsions using low-molecular-weight branched hydrocarbon ionic surfactants,[62] formed the $CO_2$-in-water emulsions stabilized with a branched nonionic hydrocarbon surfactant.[63,64] $CO_2$-in-water emulsions have been successfully applied in synthesizing porous polymers. For example, Cooper and coworkers[65-68] synthesized porous polymer materials by polymerization of a concentrated $CO_2$-in-water emulsion. Wang et al.[69] synthesized hollow silica spheres with mesopore wall structures via $CO_2$-in-water emulsion templating in the presence of non-ionic poly(ethylene oxide)-*b*-poly(propylene oxide)-*b*-poly(ethylene oxide (PEO-PPO-PEO) block copolymer surfactants. Barbetta et al.[70] obtained porous biomaterials using a high internal phase $scCO_2$ emulsion templating route.

## 3 Amphiphile self-assemblies in ILs

The use of ILs as solvents for amphiphile self-assembly was reviewed by Baker and Pandey in 2005,[71] Hao and Zemb in 2007,[72] Greaves and Drummond in 2008,[73] Zecha and Kunz in 2011,[74] and Greaves and Drummond in 2013.[75]

### 3.1 Micelles in ILs

Up to now, most of amphiphiles for micelle formation in ILs are nonionic surfactants. For example, Zheng's group has shown that the amphiphilic poly(oxyethylene)-poly(oxypropylene)-poly(oxyethylene) ethers triblock copolymers,[76] polyoxyethylene (20) sorbitan monolaurate (Tween 20)[77] and Triton X-100[78] can aggregate and form micelles in 1-butyl-3-methylimidazolium tetrafluoroborate ($bmimBF_4$) and 1-butyl-3-methylimidazolium hexafluorophosphate ($bmimPF_6$). It was revealed that the dissolution of Triton X-100 in ILs depressed the surface tension in a manner analogous to aqueous solutions, and a relatively higher critical micellar concentration was obtained compared to that of water.[78] Aktin et al.[79] studied the phase behaviour and self-assembled microstructures of a range of oligo(oxyethylene)-n-alkyl ether (CiEj) surfactants in propylammonium nitrate (PAN). Micelles were found to form at alkyl chain lengths from dodecyl to octadecyl. Sarkar et al.[80] have shown the aggregation behaviour of Triton X-100 in a mixture of ILs, ethylammonium nitrate (EAN) and $bmimPF_6$.

Fluorinated surfactants are highly efficient and have some special properties such as chemical inertness, thermal stability, and oleophobicity, which offer some advantages over hydrocarbon surfactants. Zheng et al. studied the aggregation behaviour of a fluorinated surfactant (FC-4) in $bmimBF_4$,[81] $bmimPF_6$,[81] and 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ($bmimTf_2N$).[82] It was found that the surface activity of the fluorinated surfactant in ILs was superior to the activity of other surfactants. Hao et al. reported the micellization of an anionic fluorocarbon surfactant, tetramethylammonium perfluorononanoic carboxylate, in $bmimBF_4$[83] and a fluorinated zwitterionic surfactant, polyfluorinated-2-dodecenyl (3-sulfate) propyl dimethyl ammonium, in EAN.[84]

The surfactant-like ILs have been reported to form micelles in IL. For example, Kunz and coworkers[85] proved that the surfactant-like ILs ($C_{16}mimCl$, $C_{16}mimBF_4$) can form micelles in another IL EAN. Zheng et al.[86] studied the aggregation behaviour of long-chain ILs 1-alkyl-3-methylimidazolium bromide ($C_nmimBr$) in another IL $bmimBF_4$. It is revealed that spherical aggregates are formed by $C_nmimBr$, and the size is much larger than traditional micelles.

### 3.2 Vesicles in ILs

Nakashima and Kimizuka[87] reported the surfactant vesicles in IL medium about a decade ago. They created the bilayer structures of dialkyldimethylammonium surfactants in ether-containing ILs. Hao and coworkers[88] proved that the $Zn^{2+}$ fluorous surfactant or a mixture of $Zn^{2+}$ fluorous surfactant–zwitterionic surfactant could form vesicles in imidazolium based ILs. Viguerie et al.[89] prepared spherical vesicles from 1,2-dipalmitoyl-sn-glycero-3-phosphatidylcholine (DPPC) in different ILs, including $bmimBF_4$, $bmimPF_6$, 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ($emimNTf_2$), and N-benzylpyridinium bis(trifluoromethylsulfonyl)imide ($BnPyNTf_2$). The mean bilayer thickness was found to be ~63 ± 1 Å in a deuterated IL ($BnPyNTf_2$-d), similar to that observed in water. Wagner et al.[90] reported the spontaneous formation of vesicles from a cationic double tail surfactant (didodecyldimethylammonium bromide, DDAB) in EAN. A low density and highly viscous solution containing giant vesicles and a sponge phase coexists with a dilute high density phase containing large vesicles. Abe et al.[91] investigated the surface chemical properties of a phospholipid, dilauroylphosphatidylcholine (DLPC), in $bmimBF_4$ and $bmimPF_6$. It was found that DLPC has finite solubility in the ILs and it can form vesicles spontaneously with size distribution around 400 nm in diameter above the critical vesicular concentration. Kumar et al.[92] reported the formation of unilamellar vesicles and reverse vesicles of L-prolineisopropylester laurylsulfate in a hydrophilic ethanolammonium formate and hydrophobic $bmimTf_2N$ IL medium, respectively, which otherwise forms aggregated micelles in aqueous medium with an individual micellar size of ~5 nm.

### 3.3 Liquid crystallines in ILs

Dozens of ILs have been reported to be able to support the self-assembly of surfactants into various lyotropic liquid crystalline phases. Drummond et al.[93-98] carried out a systematic study on the phase behaviour of ionic surfactants and non-ionic surfactants in a series of protic ILs (PILs). Self-assembled lyotropic liquid crystalline structures including hexagonal, cubic, and lamellar phases were observed. Chen et al. studied the aggregation of poly(ethylene oxide)-b-poly(propylene oxide)-b-poly(ethylene oxide) triblock copolymer,[99] $C_{16}mimCl$,[100] oleyl polyoxyethylene (10) ether, Brij 97[101] in EAN. A series of lyotropic mesophases, including normal micellar cubic, normal hexagonal, lamellar, and reverse bicontinuous cubic, were identified at room temperature. Warr et al.[102,103] reported the formation of a variety of lyotropic liquid crystals (LC) by some polyoxyethylene nonionic surfactants in EAN. The pattern of self-assembly behaviour and mesophase formation is strikingly similar to that observed in water, even including the existence of a lower consolute boundary or cloud point. The main difference from water is that longer alkyl chains are necessary to drive the formation of liquid crystalline mesophases in EAN, suggesting that a rich pattern of "solvophobic" self-assembly should exist in this solvent. Zheng et al.[104] studied the phase behaviour of Nhexadecyl-N-methylpyrrolidinium bromide ($C_{16}$MPB) in EAN. With increasing $C_{16}$MPB concentration, an isotropic solution phase, a hexagonal ($H_1$) phase, and a cubic phase ($V_2$) are all present in either EAN or $H_2O$. The rheological results reveal that the $H_1$ phase constructed by $C_{16}$MPB in EAN displays a typical Maxwell behaviour, whereas the $H_1$ phase formed by $C_{16}$MPB in water shows a gel-like behaviour, unlike traditional cationic surfactants. The lyotropic LC phase in EAN has a higher thermal stability than that formed in $H_2O$, which may be important to extend the applications of the LC phase.

### 3.4 IL-based microemulsions

Microemulsions containing ILs have drawn considerable interest in recent years,[105-107] since the first publication on nonaqueous IL microemulsions reported by Han et al in 2004.[108] Using ILs as dispersed or continuous phase of microemulsions is very attractive owing to their unusual solvent properties. Up to now, various kinds of IL-based microemulsions have been prepared, including IL-in-oil and oil-in-IL microemulsions,[108-133] IL-in-water and water-in-IL microemulsions,[134-138] IL-in-ethylene glycol (EG) and EG-in-IL microemulsions,[139] and IL-in-IL microemulsion.[140] The phase behaviours, microstructures, micropolarities, thermodynamic and dynamic properties of IL-based microemulsions have been investigated. These microemulsions have shown promising applications in different fields, such as material synthesis,[141-146] polymerization,[147-151] biocatalysis,[152-155] drug release,[156,157] etc.

### 3.5 IL-based emulsions

Binks et al. first reported the IL emulsions stabilized by silica nanoparticles.[158,159] Very recently, Zhang and coworkers prepared some IL-based emulsions, including the high internal IL phase emulsion stabilized by PEO-PPO-PEO triblock copolymers,[160] IL-in-IL nanoemulsion formed in the two immiscible ILs (propylammonium formate and 3-methyl-1-octylimidazolium hexafluorophosphate) with the aid of surfactant AOT,[161] and multiple water-in-IL-in-water emulsions stabilized by surfactant TX-100.[162] In comparison with the conventional oil-water emulsions, these IL-based emulsions involve no volatile organic solvent and are more environmentally benign.

## 4 Microemulsions and emulsions in $CO_2$/IL

The simultaneous utilization of $CO_2$ and ILs can combine the advantages of the two fluids. With the aid of surfactant, $CO_2$ and ILs can be dispersed in the form of microemulsions or emulsions.

### 4.1 Microemulsions in $CO_2$/IL

Han and coworkers[163] constructed IL-in-sc$CO_2$ microemulsions using surfactant N-ethyl perfluorooctylsulfonamide (N-EtFOSA) and guanidium-based ILs. The IL domains dispersed in $CO_2$ can solubilize different salts, such as methyl orange, $CoCl_2$, and $HAuCl_4$. Senapati et al.[164] investigated the formation of IL-in-sc$CO_2$ microemulsions *via* a computer simulation. They demonstrated the entire process of self-aggregation and provided direct evidence of the existence of stable IL droplets within a continuous $CO_2$ phase through amphiphilic surfactants at the molecular level.

Zhang and coworkers[165] created the $CO_2$-in-IL microemulsions, with IL as the continuous phase and $CO_2$ as the dispersed phase. For the 3.0 wt% N-EtFOSA/1,1,3,3-tetramethylguanidinium acetate (TMGA) solution, the maximum amount of $CO_2$ solubilized in micelles, characterized by the molar ratio of $CO_2$ to surfactant in micelles ($R_{CO2}$), can reach 12.5 at 5.7 MPa. At $R_{CO2}$=12.5, the micelles have an average size of 47 nm, considerably larger than the $CO_2$-free micelles. The size of the $CO_2$-swollen micelles can be easily tuned by the pressure of $CO_2$.

### 4.2 Emulsions in $CO_2$/IL

Zhang et al.[166] produced $CO_2$-in-IL emulsions in the $CO_2$/N-EtFOSA/1-butyl-3-methylimidazolium nitrate (bmim$NO_3$) system at 25 $^{o}$C. A $CO_2$-in-IL emulsion templating route for the synthesis of porous polymers was proposed. Highly porous polyacrylamide and poly(trimethylolpropane trimethacrylate) having hierarchical macro- and mesoporous structures were obtained, and the porosity properties can be easily tuned by the control of $CO_2$ pressure. Furthermore, the mesoporous metal-organic framework nanospheres (~80 nm) with highly ordered hexagonal pores have been synthesized in IL/sc$CO_2$/surfactant emulsion.[167] The micelles in emulsions are believed to template the formation of mesopores.

## 5 Conclusion and Perspective

Study on the amphiphile self-assemblies in sc$CO_2$ and ILs is an interesting topic from both viewpoints of scientific researches and practical applications. sc$CO_2$ and ILs are tunable solvents and their properties are different from the conventional solvents. Therefore, the amphiphile assemblies in sc$CO_2$ and ILs will show many unique features and can satisfy many different requirements of applications. Despite exciting recent developments, the area of amphiphile self-assemblies in sc$CO_2$ and IL is still in an immature phase. Some future researches on this topic are in the following.

First, more amphiphile self-assemblies in sc$CO_2$ and IL with special compositions, structures and functions should be designed and constructed to satisfy a variety of tasks. Additional work should be carried out especially using low cost surfactants and ILs. Second, the amphiphile self-assemblies in the mixture of sc$CO_2$ and IL have been studied sporadically and should be paid more attention. Since sc$CO_2$/IL mixture combines the advantages of the two fluids, some novel self-assemblies are expected to be formed, which may find applications with special advantages. Third, the process of the self-assemblies is still not clear despite vast researches have been conducted. Theoretical and experimental techniques should be combined to further unravel the special properties and formation mechanism of the amphiphile self-assemblies in sc$CO_2$ and ILs. Fourth, up to now the designable nature of ILs has only been utilized in a limited extend. The designs of functional ILs and surfactants should be combined to create amphiphile systems with special functions. Fifth, taking the advantages of $scCO_2$, IL and amphiphile self-assemblies, many challenging problems in material synthesis, chemical reaction, and separation can be solved, more attention should be paid to explore the applications of these kinds of functional systems.

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## Biography & photograph

**Jianling Zhang** received her Ph.D. degree at Institute of Chemistry, Chinese Academy of Sciences (CAS), in 2003. She is currently a Professor of Institute of Chemistry, CAS. Her research interest concerns the amphiphile assemblies in green solvents including supercritical $CO_2$, ionic liquids, polyethylene glycol, and their mixtures.

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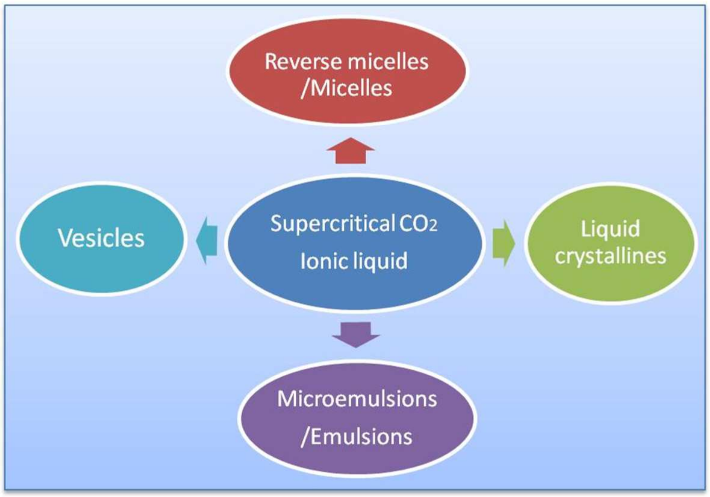

The amphiphile self-assemblies in supercritical $CO_2$ and ionic liquids are reviewed, including micelles, vesicles, liquid crystals, microemulsions and emulsions.