Scientific
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Aggregation Behavior of Amphiphilic PAMAM-Based Hyperbranched Polymer in the Presence of Conventional Small Molecular Surfactants

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ABSTRACT

Hyperbranched polymer composed of G1 polyamidoamine (PAMAM) and branched with poly (propylene oxide) (PPO)-*block*-poly (ethylene oxide) (PEO) was investigated to interact with sodium dodecyl sulfate (SDS) and di-dode-cyl dimethyl ammonium bromide (DDAB), respectively, by the methods of turbidity titration and analysis, rheology measurements, dynamic light scattering (DLS) and transmission electron microscopy (TEM). It was noticeable that even at extremely low concentration of SDS (even far from the critical micelle concentration (cmc)), the system exhibits high turbidity, indicating that SDS molecules can insert into cationic amine groups and hydrophobic microenvironment, resulting in the formation of polymer-SDS complexes with large size. At the SDS concentration range of below 0.1 mM, the turbidity and cloud point (CP) temperature of the system keep almost invariable, mostly because of the repulsion between SDS molecules and the complexes. And, therefore, the size of the mixed aggregates retains almost constant. In the case of vesicle system of DDAB, the aggregates are in the size of 100 nm - 200 nm and 500 nm - 3000 nm at the concentrations of 30 mM and 100 mM, respectively. However, in the mixture of hyperbranched polymer with DDAB, by comparison, the size is smaller in a binary system than that of in DDAB system. So it is reasonable to infer that DDAB molecules remove from multilamellar vesicles of DDAB to the hydrophobic microenvironment of hyper-branched polymer aggregates, with the addition of the hyperbranched polymer. It leads to the destruction of the gel-like conformation in DDAB system, leading to the shear thinning of the mixture and, as a result, the viscoelastic character of the system is lost in a large degree.

1. Introduction

Hyperbranched polymers are highly branched and three-dimensional macromolecules with dentritic architecture and long repeat units [1]. Due to their unique physical and chemical properties, hyperbranched polymers have gained significant attention from both academia and industry [2,3]. Various examples of PAMAM-based polymers functionalized with rigid hydrophobic periphery, chromophoric groups, or binding metal ions have been investigated intensively these years [4,5]. Little attention, however, was paid to the aggregation behavior of PAMAM molecules branched with amphiphilic segments [6], especially to understand their solution behavior or with the addition of surfactants [7-11].

Thus far, surfactant-hyperbranched polymer interactions have been concerned, which are important in many potential application fields, such as drug delivery vehicles, cosmetic, food, and paint industries [12,13]. The mixed systems of PAMAM and surfactant in aqueous solution have been investigated by several researchers [14-17]. It showed that the aggregation between the hyperbranched polymer and surfactant is significantly dependent upon the nature of surface groups, or ionic surfactant head groups [18]. Surface activity and hydrophobicity of the surfactants are enhanced upon interaction with PAMAM dendrimers, and the apparent hydrody-

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namic radius largely increases with such interactions [19-22].

In the investigation of salt effect and pH response on the series of polymers with poly(propylene oxide) (PPO)-block-poly(ethylene oxide) (PEO), we found that the hyperbranched amphiphiles are very surface active, of low aggregation concentration, and the aggregation behavior and aggregates conformation are sensitive to pH and salt [23-25]. With the increase of pH value, the hyperbranched polymer solution transformed from turbid to transparent, due to the protonation process of PAMAM; with the addition of inorganic salts, the hydration water molecules preferentially move from coordination shells of hyperbranched polymer molecules to the hydration layer of salt molecules; and the benzene ring of organic salt has a significant effect in increasing the size of complex aggregates, owing to penetrating into the hydrophobic core of aggregates. As a further study, the work has been extended to investigate the molecular interactions in the micellization of amphiphilic polymers and conventional surfactants. The mixtures are characterized by turbidity titration, DLS and TEM methods. The different aggregation properties of it in aqueous solutions are evaluated in absence as well as presence of additives (surfactants). Nowadays, many investigations have been made on the supramolecular structures and interactions of PAMAM molecules, but little has been reported on the relationship between amphiphilic PAMAM-based polymer and surfactant. In order to further understand such interactions, we investigated the interplay between this type of polymer and sodium dodecyl sulfate (SDS) through examining the change in turbidity and aggregation properties, and the transition in rheology and aggregation behavior of the mixed system of the polymer and didodecyldimethylammonium bromide (DDAB).

2. Experimental Section

2.1. Materials

Chemicals were obtained from Beijing Chemical Reagents Company and were of analytical grade. Solutions were prepared with triply distilled water.

2.2. Synthesis and Characterization

The route of synthesis on hyperbranched polymer and DDAB followed the previous work [23,26].

2.3. Measurements

Turbidimetric Titration. The turbidity of hyperbranched polymer solution with titrated SDS was measured at 450 nm using a Brinkman PC920 probe colorimeter equipped with a thermostated water-circulating bath. The cloud point (CP) of the system at different

conditions was obtained from the break of $100-T\%$ (transmittance) vs. temperature curves.

Rheology. The rheological properties of samples were measured at $25.00°C \pm 0.01°C$ with a Thermo Haake Rheo Stress 300 rheometer (cone and plate geometry of 35 mm in diameter with the cone gap equal to 0.105 mm). The range of frequency scanning is $0.1 - 20 \ rad \cdot s^{-1}$.

Dynamic Light Scattering (DLS). DLS measurement was introduced, employing an LLS spectrometer (ALV/ SP-125) with a multi-τ digital time correlator (ALV-5000). Light ($\lambda = 632.8$ nm) from a solid-state He-Ne laser (22 mW) was used as the incident beam. The scattering angle was selected to 90° and the correlation function was analyzed with the Contin method. Solutions were filtrated through 0.45 μm millipore to leach dust, and were laid until stabilization before measurements.

Transmission Electron Microscope (TEM). Negative-staining (with uranyl acetate aqueous solution of 1 wt%) and freeze-fracture (FF) techniques were used for TEM sample preparation. Fracturing and replication were carried out in a high-vacuum freeze-etching system (Balzers BAF-400D). The samples were imaged under a Hitachi H800 electron microscope.

3. Results and Discussion

3.1. Aggregation Behavior of the Mixture of Hyperbranched Polymer and SDS

At the temperature of 15°C, the turbidity curve of hyperbranched polymer (at 1 wt%) with titrated SDS is shown in **Figure 1**, which exhibits that turbidity value ascends with the concentration of SDS increasing firstly, indicating that there may be strong interaction between hyperbranched polymer and SDS molecules. As the concentration of SDS comes to about 0.024 mM, the turbidity value reaches the peak. When it is higher than 0.11 mM
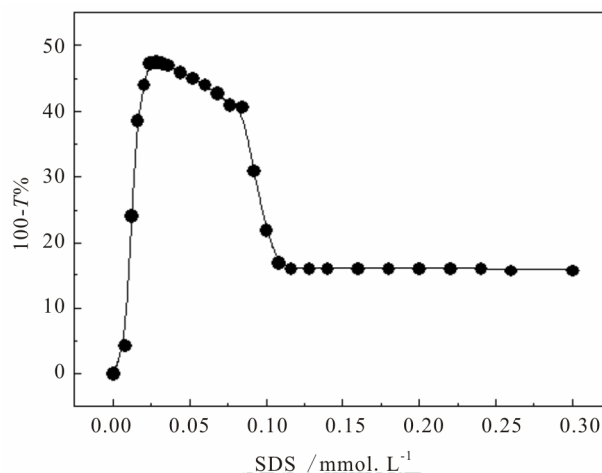

Figure 1. Turbidity curve of hyperbranched polymer solution with the titration of SDS.

(around 1% of cmc of SDS), the turbidity value keeps a constant of around 15%, inclining to an unchangeable state.

Turbidity vs. temperature curves of hyperbranched polymer with SDS at different concentrations and the cloud point (CP) temperatures are shown in **Figure 2** and **Table 1**, separately. As the concentration of SDS increases, CP of the mixture decreases at first (in the concentration range of 0 - 0.05 mM), and then tends to be almost invariable around 24°C (0.1 mM - 0.5 M). The results show that the hyperbranched polymer interacts with SDS even at very low concentration (such as 0.01 and 0.05 mM); while CP varies little with added SDS at higher concentration (≥0.1 mM), which matches well with the deduction from turbidity titration experiments.

With the addition of SDS, R_h of aggregates increases to 220.2 and 396.1 nm at the SDS concentration of 0.01 and 0.05 mM (**Figure 3** and **Table 1**), respectively, indicating that the hyperbranched polymer-SDS complex forms and leads to the formation of bigger aggregates. This result is in well agreement with that of turbidity and CP measurements, in which the turbidity value increases at the same SDS concentration range. In this case, SDS molecules could insert into cationic amine groups [27] and hydrophobic microenvironment [28] as designed and
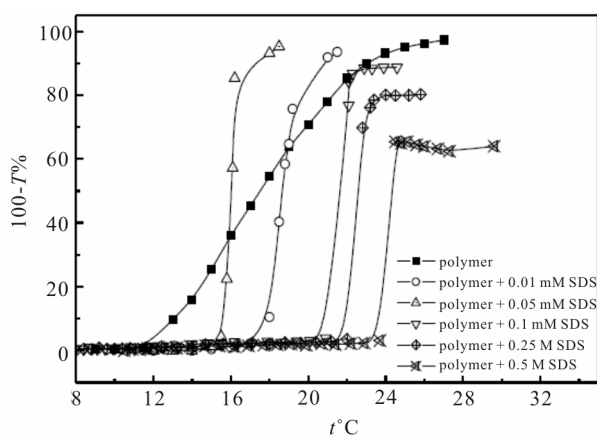
drawn in **Figure 4**, which leads to the enlargement of aggregate size. As the concentration of SDS is higher than 0.1 mM corresponding with the invariable turbidity, the R_h value remains around 370 nm, mainly because of the repulsion between SDS molecules and that in hyperbranched polymer-SDS complexes [29]. As the concentration keeps increasing (up to 0.25 and 0.5 M), there is an R_h distribution of 3 - 5 nm promoted from the self-aggregation of SDS molecules, or the aggregation of multimolecular SDS and monomolecular hyperbranched polymer [30,31].

Through TEM method the molecular aggregation behavior was examined as shown in **Figures 5(a)-(f)**. The aggregates of hyperbranched polymer are around 100 nm
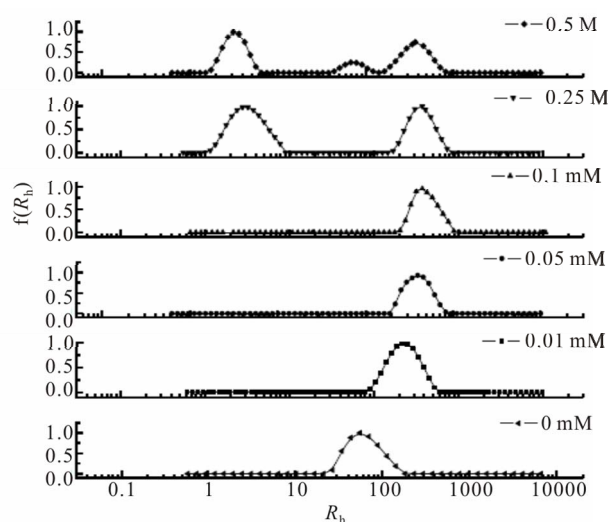
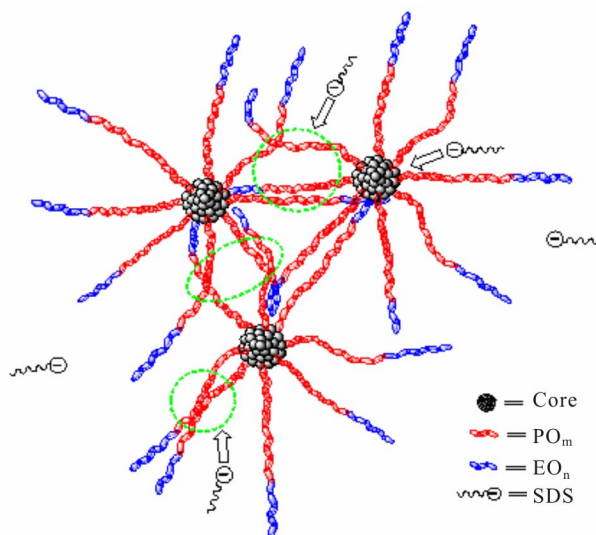**Figure 3. DLS results of the binary system at the SDS concentration range of 0 - 0.5 M.**

Figure 2. Cloud point (CP) curves of mixed system of SDS and hyperbranched polymer.

Table 1. Apparent hydrodynamic radius (R_h) of the aggregates and CP temperature of the mixed system.

Mixed system		CP (°C)	R_h (nm)
$C_{Hyperbranched\ polymer}$	C_{SDS}		
	0	22.5	54.9
1%	0.01 mM	19.2	220.2
	0.05 mM	16.2	396.1
	0.1 mM	22.9	375.5
	0.25 M	23.4	3.1; 379.1
	0.5 M	24.5	4.8; 370.3

Figure 4. Scheme of interaction between hyperbranched polymer and SDS molecules.

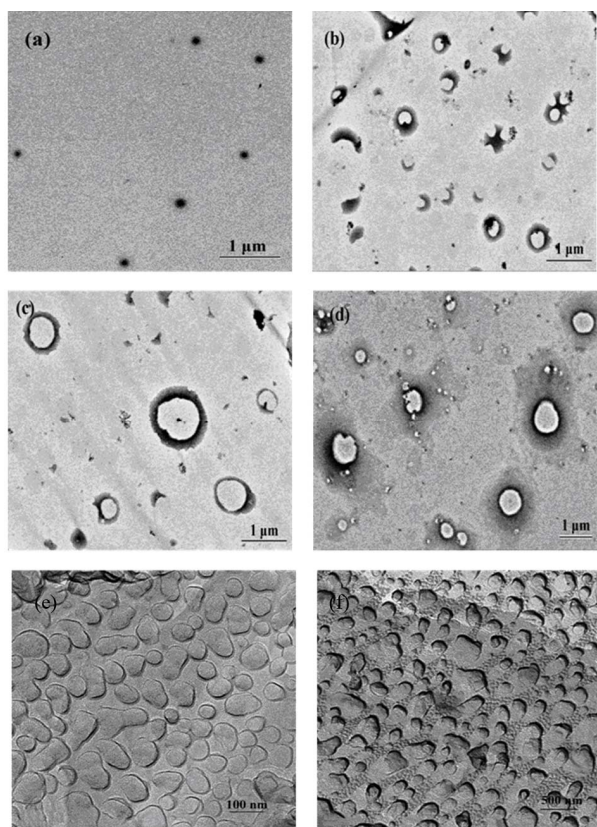**Figure 5. TEM photos of the mixed system of hyperbranched polymer interacting with SDS: (a) 1% hyperbranched polymer (Negative-staining); 0.01 mM SDS + % hyperbranched polymer (Negative-staining); (c) 0.05 mM SDS + 1% hyperbranched polymer (Negative-staining); (d) 0.25 M SDS + 1% hyperbranched polymer (Negative-staining) (e) 1% hyper-branched polymer (FF); (f) 0.01 mM SDS + 1% hyper-branched polymer (FF).**

as observed through negative-staining (**Figure 5(a)**) and freeze-fracture (**Figure 5(e)**) techniques. The size of aggregates grows to 400, 800 and 800 nm with the addition of SDS at 0.01 mM, 0.05 mM, and 0.25 M, respectively, as shown in **Figures 5(b)-(d)** and **(f)**. Moreover, with higher content SDS, there are smaller aggregates in the system, which is in agreement with that from DLS-method.

3.2. Rheological Properties of the Mixed System of Hyperbranched Polymer and DDAB

From **Figures 6(a)** and **(b)**, vivid bilayer vesicle can be observed, and the size becomes bigger ranging from 100 - 200 nm to 500 - 3000 nm with the concentration increase from 30 mM to 100 mM. **Figure 6(c)** shows a collection of unilamellar vesicles with an average size around 100 nm, while **Figures 6(d)-(f)** show typical multilamellar aggregates in the size of 1 - 3 μm, which exhibits that giant multilamellar vesicles coexist with smaller unilamellar ones and some of the multilamellar
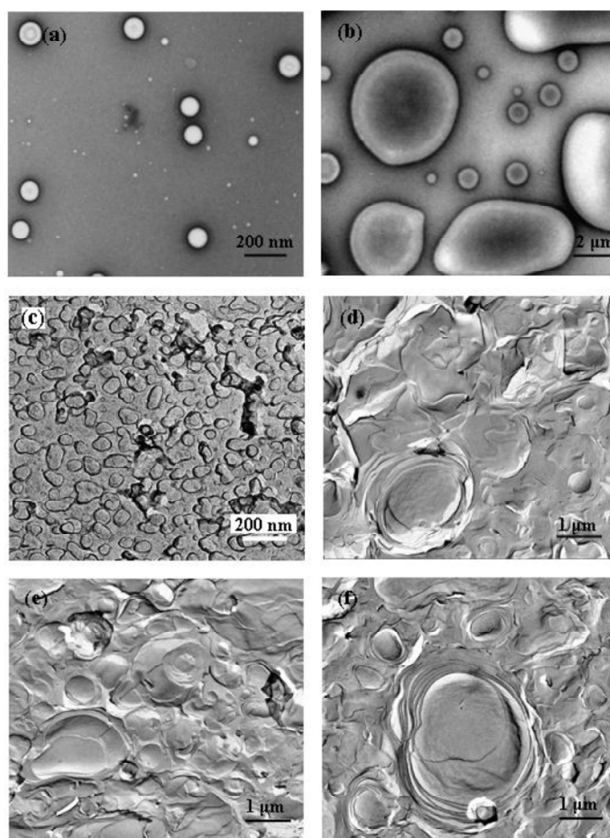

Figure 6. TEM images of DDAB aggregates at different concentrations: (a) 30 mM (Negative-staining); (b) 100 mM (Negative-staining); (c) 30 mM (FF); (d) 60 mM (FF); (e) 80 mM (FF); (f) 100 mM (FF).

aggregates are curved structures like "onion" shells. Almost each vesicle is surrounded by other ones and locates in a cage, which is expected from packing constraints forcing the multilayered structures to occur. From the "cage" it can not escape by a simple diffusion process without deformation of its "shells", and therefore, the system has a viscoelastic property under deformation [32]. The mixed system with hyperbranched polymer and DDAB (at different concentrations) is investigated through TEM measurements shown in **Figures 7(a)-(f)**. The size of aggregates is about 100 nm and 1 μm with the added DDAB at the concentration from 30 mM to 100 mM, which is smaller than that of DDAB system. And the structure transforms from multilamella to unilamella, indicating that the "shell-cage" conformation is mostly destroyed (because there is interaction between DDAB and hyperbranched polymer).

The shear rheology curves of the mixed system are shown in **Figure 8(b)**. The viscosity of the mixture decreases from 1000 to 100 s^{-1}, which is lower than that of DDAB system, because the gel-like conformation of DDAB system transforms to unilamella vesicle system. Furthermore, it comes from the interaction between hy-
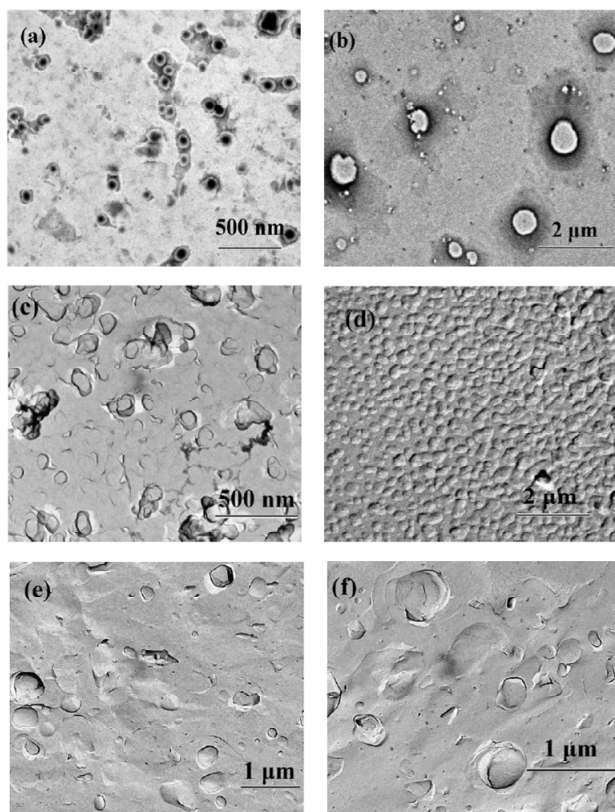
Figure 7. TEM photos of binary system of DDAB and hyperbrabched polymer at differert conditions: (a) 1% hyperbranched polymer + 30 mM DDAB (Negative-staining); (b) 1% hyperbranched polymer + 100 mM DDAB (Negative-staining); (c) 1% hyperbranched polymer + 30 mM DDAB (FF); (d) 1% hyperbranched polymer + 60 mM DDAB (FF); (e) 1% hyper-branched polymer (FF); (f) 0.01 mM SDS + 1% hyper-branched polymer (FF).

Figure 8. Steady shear rheology curves: (a) DDAB; (b) DDAB + 1% hyperbrabched polyer.

perbranched polymer and DDAB, as well as the self-aggregation of DDAB molecules.

Plots of rheological parameters $G'(\omega)$ and $G''(\omega)$ versus angular frequency ω are shown in **Figure 9**. Generally, the storage modulus G' gives a measure of elastic properties of the material and the loss modulus G'' reflects a certain degree of viscosity [33]. Within the studied frequency range, G' and G'' modules exhibit frequency independence at 80 and 100 mM and shows typical gel-like behavior (see the curves a and b). G'' has higher values than G'', indicating that the system has a weak viscoelastic characteristic at these concentrations. However, at lower concentration (lower than 80 mM), the solution is of weaker viscoelastic with lower values of G' and G'' (see the curve c). G'' value is bigger than that of G' at the ω range of 0.1 - 1 s^{-1}, but smaller over 1 - 10 s^{-1}, which indicates that G' and G'' become frequency-dependent. Hence, the colloid solution becomes higher viscoelastic and the relation between G' and G'' changes from frequency dependen-
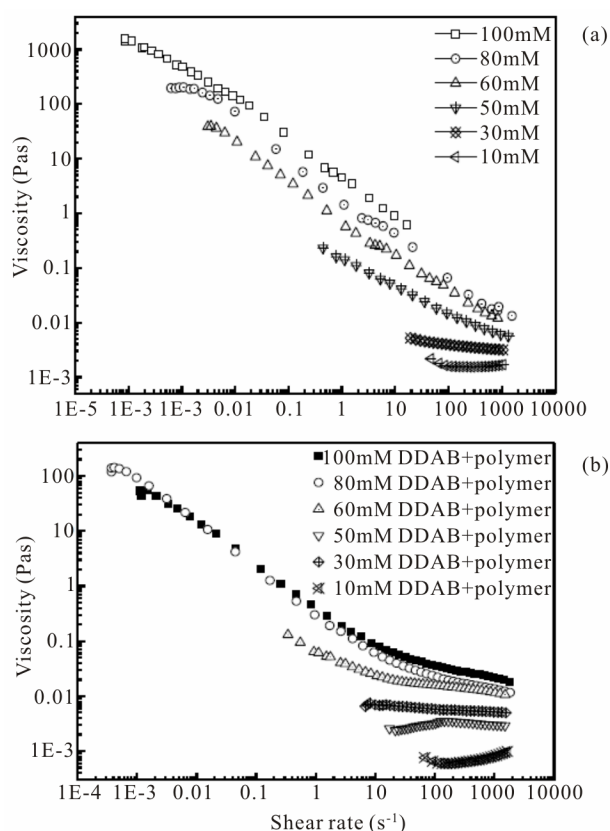
dence to independence, mostly due to the aggregate conformation changing with the increase of concentration. However, with the addition of hyperbranched polymer, the system can not be detected the values of G' and G'' vs. ω, so the binary system does not possess viscoelasticity, indicating that the gel-like configuration is destructed and some DDAB molecules remove from multilamellar vesicles of DDAB to the hydrophobic microenvironment of hyperbranched polymer (**Figure 10**), and therefore the unilamellar vesicles of DDAB and the hyper- branched polymer-DDAB complexes form to a large extent.

4. Conclusions

Through measurements of turbidity analysis, DLS and TEM, the interaction between hyperbranched polymer and SDS, including aggregation behavior and turbidity property of the mixed system, was investigated. With SDS even at low concentration such as far from CMC, there is strong molecular interaction in polymer-SDS system. The aggregates become larger with the addition of SDS, because SDS molecules can insert into amine group and hydrophobic microenvironment. When the concentration of SDS is higher than 0.1 mM, the size of
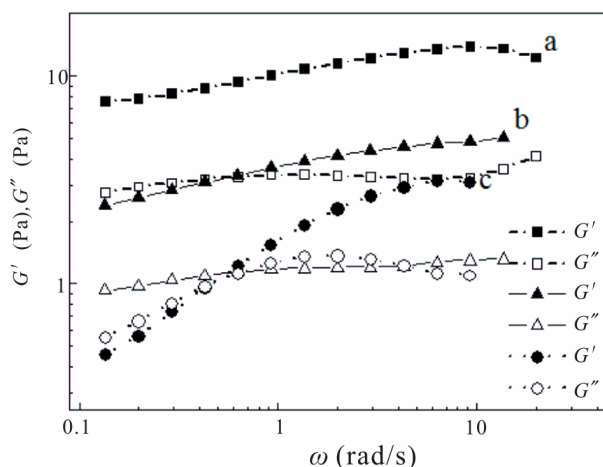
Figure 9. Storage modulus (G') and loss modulus (G'') as a function of angular frequency (ω) at: a 60 mM, b 80 mM and c 100 mM.
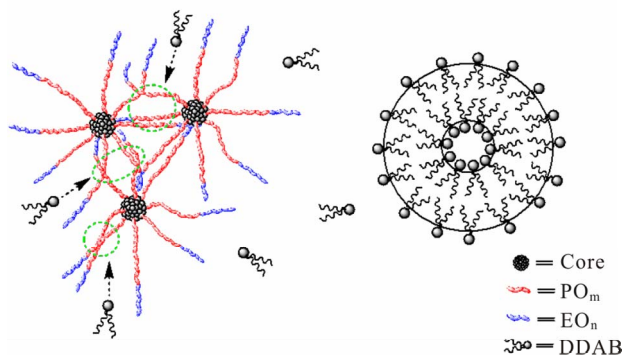

Figure 10. Scheme of interaction between hyperbranched polymer and DDAB molecules.

aggregates keeps constant due to the repulsion among SDS molecules and hyperbranched polymer-SDS complexes. More SDS molecules scarcely lead aggregates continue growing but induce the self aggregates of SDS or multi-molecules of SDS aggregating with monomolecular hyperbranched polymer.

By comparison, the viscoelasticity of DDAB in the presence of hyperbranched polymer was investigated by the methods of steady and dynamic state rheological properties, the detection and analysis of aggregation behavior. The results showed that the gel-like conformation is broken in the presence of hyperbranched polymer. And then, the size of aggregates decreases from 100 - 200 nm and 500 - 3000 nm (in DDAB system at the concentration from 30 mM to 100 mM) to 100 nm and 1 μm (in the mixed system of hyperbranched polymer and DDAB; DDAB at the same concentration range), and the aggregates morphology transforms from multilamella to unilamellar. It induces the viscosity to decrease largely after shearing, and the mixture nearly shows no viscoelastic property.

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