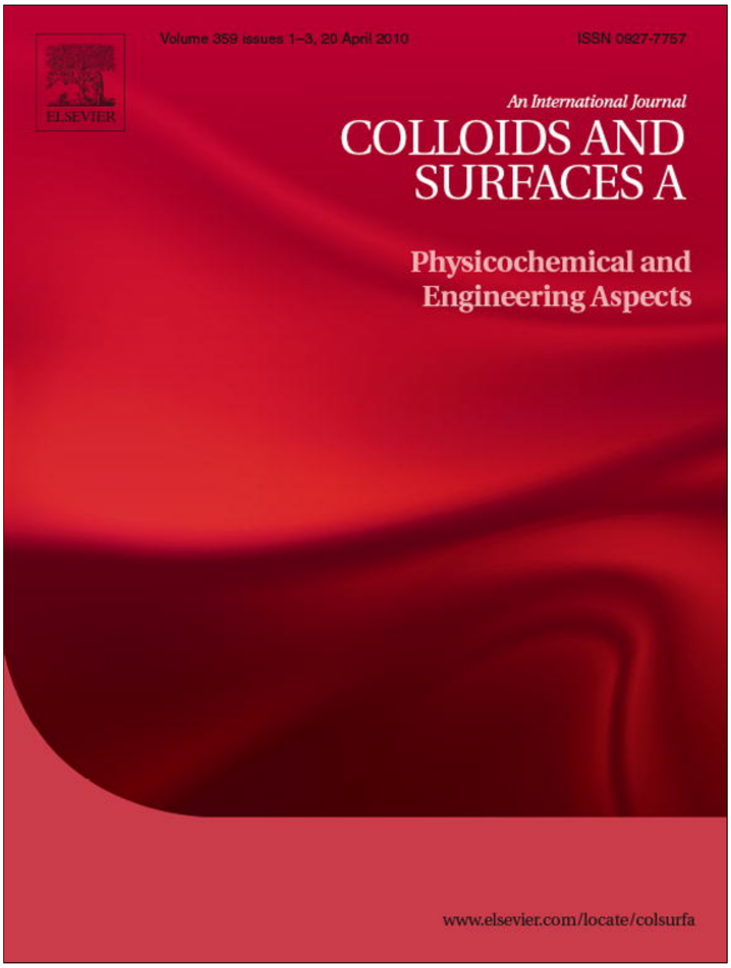
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Dispersion of multiwalled carbon nanotubes by ionic liquid-type Gemini imidazolium surfactants in aqueous solution

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ABSTRACT

Butyl-α,β-bis(dodecylimidazolium bromide) ($[C_{12}$-C_4-$C_{12}im]Br_2$) is a new type ionic liquid-based Gemini surfactant. Multiwalled carbon nanotubes (MWCNTs) can be dispersed effectively in $[C_n$-C_4-$C_nim]Br_2$ aqueous solutions due to its special molecular structure, including two imidazole ring head groups and two hydrophobic chains. The resulted MWCNT suspensions are stable for more than one month and no precipitation is observed. Both UV-vis-NIR and transmission electron microscopy (TEM) studies indicate that the MWCNTs dispersed in solutions are present as individual. The dispersed amount of MWCNTs first increased and then decreased with increasing the concentration of $[C_{12}$-C_4-$C_{12}im]Br_2$. Compared with single-chain ionic liquid-based surfactant 1-butyl-3-alkylimidazolium bromide ($[C_nmim]Br$), $[C_n$-C_4-$C_nim]Br_2$ has stronger ability of dispersing CNTs, which is also ascribed to its molecular structure. It was also found that the $[C_n$-C_4-$C_nim]Br_2$ with a longer hydrocarbon chain demonstrated a stronger dispersion ability. The ζ-potential measurements show that the MWCNTs dispersed in $[C_{12}$-C_4-$C_{12}im]Br_2$ aqueous solution have relatively high positive charges, which can conclude that it is the Coulomb force between CNTs that makes them stable. Based on these, the possible dispersion mechanism has been proposed.

1. Introduction

Carbon nanotubes (CNTs) have attracted great interest of researchers over the past decades because of their unique structural and the resultant electrical, optical and mechanical properties [1–4]. However, it is very difficult to disperse CNTs homogeneously in both organic and aqueous solvents due to their strong van der Waals attraction among tubes, which limits greatly their application in many fields. To solve this problem, many approaches [5] have been attempted, for instance, various surfactants [6–8], polymers [6,9–11], and natural macromolecules [12–14] have been used as dispersing agents.

Ionic liquids (ILs) are a series of organic molten salts. As a new type solvent, they have negligible vapor pressure, non-flammability, and designbable properties. Due to these unique properties, they have been widely used as environmentally benign solvents in many fields, such as chemical reaction, catalysis, electrochemistry and liquid/liquid extraction [15–19]. It is an interesting topic to use ILs to disperse CNTs. Some researches have been carried out in this field. Imidazolium-based ILs, such as 1-butyl-3-methylimidazolium tetrafluoroborate can form gel called "bucky gels" by grinding with single-walled carbon nanotubes (SWCNTs) [20]. Y. Li and co-workers indicated that imidazolium-based ILs interacted with SWCNTs through weak van der Waals interaction [21]. Kocharova et al. reported carbon nanotubes can be effectively dispersed in aqueous solutions by 1-dodecyl-3-methylimidazolium bromide and 1-(12-mercaptododecyl)-3-methylimidazolium bromide [22]. B.X. Han and co-workers found that CNTs could be dispersed stably in water with the aid of very small amounts of the amino organic salts, 1-aminoethyl-3-methylimidazolium bromide and 1-(2-aminoethyl)-pyridinium bromide [23]. Crescenzo et al. discussed in detail the capacity and stability of 1-hexadecyl-3-vinyl-imidazolium, a water-soluble long-chain ionic liquid with properties of surfactants, to disperse SWCNTs [24]. However, the ability of ILs to disperse CNTs in aqueous solutions still needs to be investigated intensively.

Ionic liquid-type Gemini imidazolium surfactants with four-methylene spacer groups ($[C_n$-C_4-$C_nim]Br_2$, n = 12, 14) have higher surface activity than the corresponding single-chain ionic liquid-type surfactants 1-alkyl-3-methylimidazolium bromide [25]. In this work, the capacity of $[C_n$-C_4-$C_nim]Br_2$ dispersing MWCNTs was studied and the interactions between MWCNTs and ionic liquid-type Gemini surfactants were monitored by UV-vis-NIR and TEM. To better clarify the effect of different molecular structures on dispersing MWCNTs, single-chain ionic liquid-based surfactants were

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selected for comparison. The experimental results indicate that ionic liquid-type Gemini imidazolium surfactants demonstrate a superior ability of dispersing and stabilizing MWCNTs in aqueous solutions even at very low concentration. The effect of chain length of $[C_n$-C_4-$C_n im]Br_2$ on the dispersion ability of MWCNTs was also investigated. $[C_n$-C_4-$C_n im]Br_2$ with longer hydrophobic chain has a much better ability to disperse MWCNTs.

2. Materials and methods

2.1. Materials

MWCNTs (purity > 95 wt%, prepared by the chemical vapor deposition method, with a length 5–15 μm, amorphous carbon <3%) were purchased from Shenzhen Nanotech Port Co., Ltd., China, and used as received. Ionic liquid-type Gemini imidazolium surfactants ($[C_n$-C_4-$C_n im]Br_2$, n = 12, 14) and 1-alkyl-3-methylimidazolium bromide were prepared and purified according to the literatures [25,26]. Distilled water was used as dispersed phase throughout the experiments.

2.2. MWCNT suspensions preparation

The suspensions of MWCNTs were prepared by adding 1 mg MWCNTs into 5 mL surfactant aqueous solutions with different concentrations, then the solutions were sonicated for 10 min in water bath at 100 W and 40 kHz. Then the suspensions of MWCNTs were stored at room temperature one day for the sedimentation of large tube bundles before characterizations. The resulting suspensions were then centrifuged for 10 min at 4000 rpm in order to separate the precipitation from the bulk solutions.

2.3. Methods

MWCNT suspensions were characterized by UV-vis-NIR on a computer-controlled spectrophotometer (U-4100, Hitachi, Japan). The absorbance of the solution was measured with a wavelength range from 200 nm to 1300 nm. Before measurement, each sample was diluted by distilled water and its concentration became one-fifth of the initial concentration. Transmission electron microscopy (TEM) observations were performed on a JEOL JEM-100 CXII(Japan) with an accelerating voltage of 80 kV. The samples were prepared by dipping a copper TEM grid into the MWCNT dispersion and subsequent drying. The ζ-potential measurements of surfactant adsorbed CNTs were made on a Malvern Zeta Master apparatus (Zeta Master 3, Malvern Instrument Ltd., Malvern, UK). Each point is an average of the three determinations. All sonication processes were carried out with an ultrasonicator (KQ-100B, Kunshan Ultrasonic Instrument Co., Ltd., China) with a frequency of 40 kHz and a fixed power of 100 W.

3. Results and discussion

3.1. Effect of $[C_{12}$-C_4-$C_{12}im]Br_2$ on the suspension of MWCNTs

Fig. 1a shows that MWCNTs are hardly suspended in aqueous solution in the absence of ionic liquid-type Gemini imidazolium surfactants ($[C_{12}$-C_4-$C_{12}im]Br_2$), even after 10 min of ultrasound. However, under the same condition, after $[C_{12}$-C_4-$C_{12}im]Br_2$ was added into aqueous solution, a macroscopic homogenous black dispersion was obtained. MWCNTs could form stable homogeneous solutions for more than one month. The concentration of $[C_{12}$-C_4-$C_{12}im]Br_2$ required to disperse the MWCNTs is quite low (only 0.1 mg/mL in Fig. 1b), indicating obviously that $[C_{12}$-C_4-$C_{12}im]Br_2$ has a superior dispersion ability.

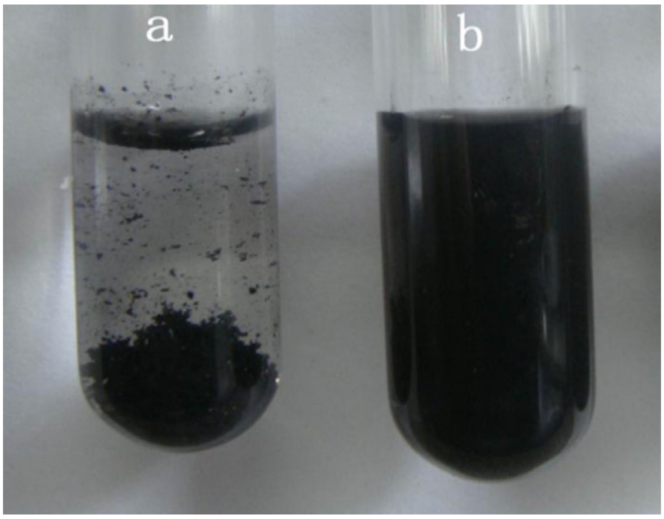

Fig. 1. Vials of MWCNT suspensions (a) without $[C_{12}$-C_4-$C_{12}im]Br_2$, and (b) with 1 mg/mL $[C_{12}$-C_4-$C_{12}im]Br_2$. The left one was imaged after one day, while the right one was imaged after more than one month.

UV-vis-NIR is usually used to evaluate the stability of individual carbon nanotubes. It has been reported that there is almost no absorption band in UV–vis region for bundled carbon nanotubes; however, individual carbon nanotubes are active in this region and strong absorption can be observed [27–31]. It can be seen from Fig. 2a that $[C_{12}$-C_4-$C_{12}im]Br_2$ aqueous solutions display only negligible absorptions in the wavelength range from 200 nm to 1300 nm, so the effect of ILs on the absorptions of MWCNT suspensions in UV-vis-NIR region can be ignored. Fig. 2b shows that MWCNT suspensions have strong UV-vis-NIR absorption, and the maximum absorbance appeared around 260 nm, similar to the previous reports [27–30]. This characteristic band in the UV-vis-NIR region is caused by carbon nanotubes 1D van Hove singularities. The inset is the magnification of curve b, we can see that dispersed carbon nanotubes have obviously absorption in this region.

MWCNTs usually aggregate together owing to strong van der Waals attraction among tubes. Upon storage at room temperature for more than one month, the MWCNTs dispersed by $[C_{12}$-C_4-$C_{12}im]Br_2$ were still stable. In order to visualize the dispersion state of MWCNTs in solution and to support the interpretation of the UV-vis-NIR results, TEM investigations have been performed.

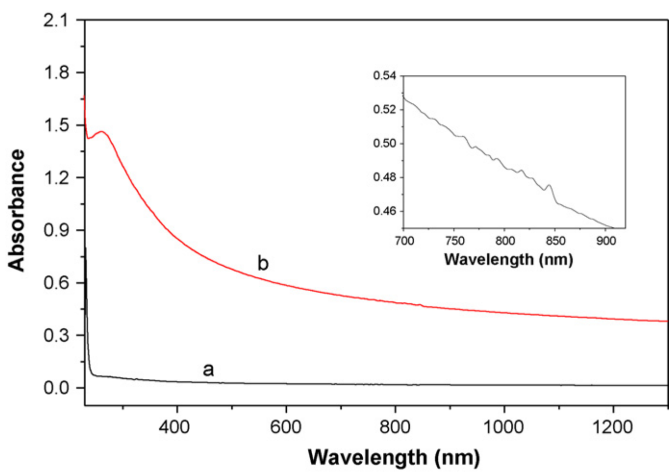

Fig. 2. UV-vis-NIR spectra of MWCNT suspensions (a) 1 mg/mL $[C_{12}$-C_4-$C_{12}im]Br_2$ aqueous solution, and (b) 1.0 mg MWCNTs dispersed in 5 mL of 1 mg/mL $[C_{12}$-C_4-$C_{12}im]Br_2$ aqueous solution. The inset is the magnification of curve (b).

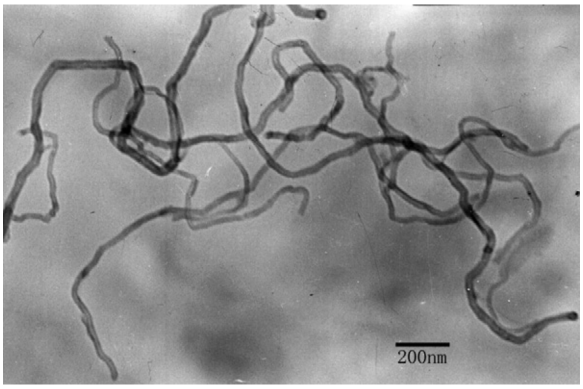

Fig. 3. TEM micrograph of MWCNT suspensions obtained from 1.0 mg MWCNTs dispersed in 5 mL of 1 mg/mL $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ aqueous solution.

The morphology and tubular structure of MWCNTs were observed (Fig. 3), suggesting that the structural integrity of MWCNTs is not deteriorated and MWCNTs can be dispersed effectively in water with the aid of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$. MWCNTs are present as individual carbon nanotube with the various diameters ranging from 10 nm to 20 nm.

3.2. *Effect of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ concentration*

The effect of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ concentration in aqueous solution on MWCNTs dispersions was studied. The UV-vis-NIR adsorptions of MWCNTs in the presence of increasing concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ aqueous solutions were obtained. Each spectrum was recorded on the supernatant of the sample once it had undergone the proper sonication and centrifugation processes. The absorption peaks in the spectra match exactly in wavelength but differ in intensity. According to the Lambert–Beer's law, with increasing the concentration of carbon nanotubes in aqueous solutions, the absorbance of carbon nanotubes at the same wavelength will increase linearly. So the concentration of carbon nanotubes in MWCNT suspensions can be determined by the absorbance of carbon nanotubes. To characterize the dispersion of MWCNTs in surfactant aqueous solutions using UV-vis-NIR spectroscopy, absorbance values were recorded at 600 nm because $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ has no absorption at this wavelength.

Fig. 4 shows the correlation between the absorbance of MWCNT suspensions and the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ at 600 nm. It can be seen that the absorbance intensity of MWCNTs did not increase continuously with increasing the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$. Instead, it passed through a maximum and then decreased. The absorbance curves could be divided into two sections. At the beginning, with increasing the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$, the absorbance sharply increases until the maximum is achieved. This indicates that at low $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ concentration, the amount of surfactant is sufficient to coat the carbon nanotubes evenly. After that, with the subsequent increase in the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$, the absorbance decreases slowly. Similar behavior has been reported previously when using other surfactants as dispersant, such as CTAB, SDS and TX-100 [31–33]. The above-mentioned phenomenon is mainly caused by the formation of micelles. Just like other Gemini surfactants, ionic liquid-type Gemini imidazolium surfactants have very high surface activity and very low cmc (0.5 mg/mL for $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$) [25]. The amount of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ molecules in bulk solutions increases gradually with the accretion of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ at the concentration below its cmc. More and more $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ molecules can adsorb on the surface of carbon nanotubes. So the amount of carbon nanotubes dispersed in aqueous solutions increases until the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ reaches its cmc. When the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ is up to cmc, the molecules of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ in aqueous solutions exist in two formats. Some molecules adsorb on the surface of carbon nanotubes and others self-aggregate to form micelles. These two formats of molecules compete with each other, and reach a balance finally. Furthermore, the amount of micelles increases with increasing concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$. Therefore, although the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ increases, the amount of free $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ molecules in bulk solutions does not increase. At the same time, the reaggregation of separated carbon nanotubes can be led by the formation of micelles around CNTs and the interactions between micelles adsorbing on carbon nanotubes. So the amount of carbon nanotubes dispersed in aqueous solutions decreases slowly with increasing concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$.

3.3. *Effect of hydrophobic chain length*

The effect of hydrophobic chain length of the ionic liquid-type Gemini imidazolium surfactants on their ability of dispersing

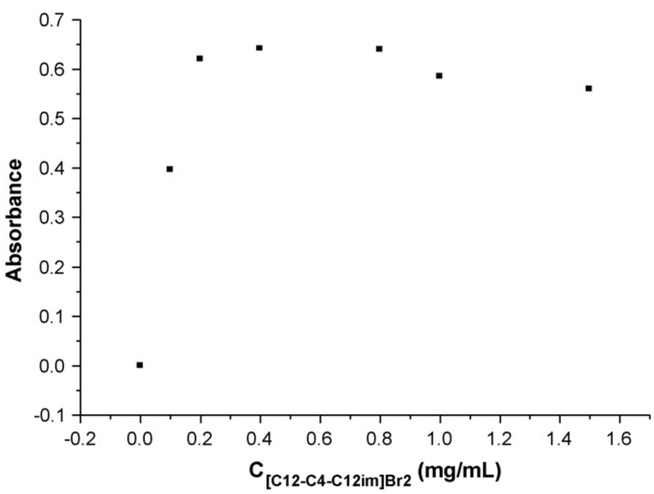

Fig. 4. The variation of the absorbance of MWCNT suspensions at 600 nm vs the concentration of $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$. (1.0 mg MWCNTs dispersed in 5 mL $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$ aqueous solutions.)

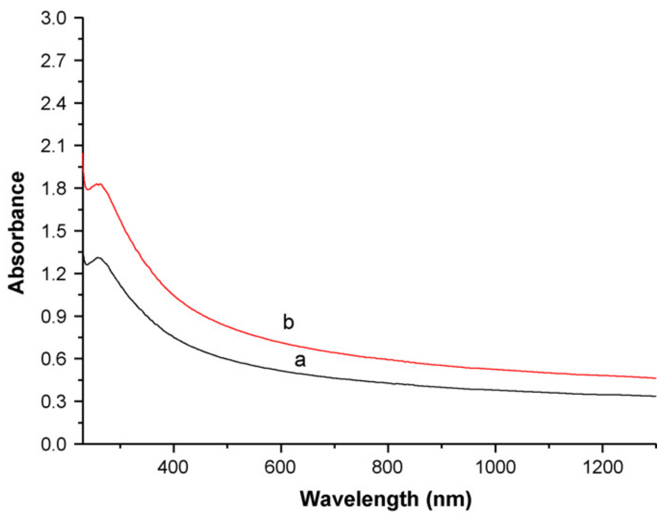

Fig. 5. UV-vis-NIR spectra of MWCNT suspensions for 1.0 mg MWCNTs dispersed in the 5 mL of 1 mM $[C_n\text{-}C_4\text{-}C_nim]Br_2$ aqueous solutions (a) $[C_{12}\text{-}C_4\text{-}C_{12}im]Br_2$, and (b) $[C_{14}\text{-}C_4\text{-}C_{14}im]Br_2$.

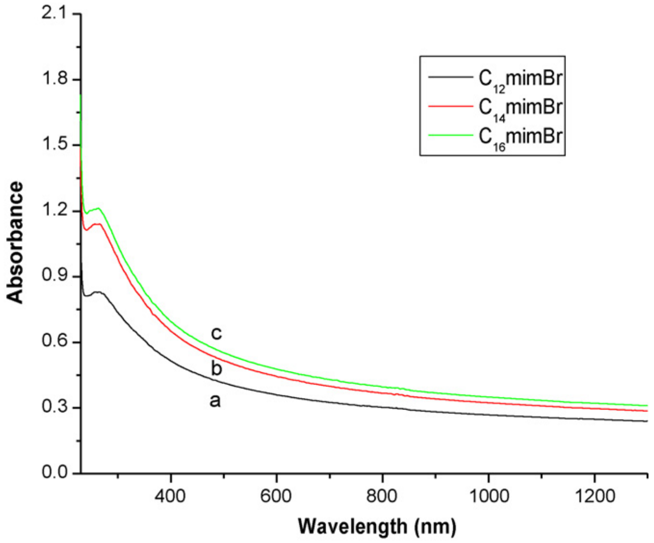

Fig. 6. UV-vis-NIR spectra of MWCNT suspensions for 1.0 mg MWCNTs dispersed in 5 mL of 1 mM [$C_n$mim]Br aqueous solutions (a) [C_{12}mim]Br, (b) [C_{14}mim]Br, and (c) [C_{16}mim]Br.

MWCNTs in aqueous solutions was also investigated. Fig. 5 shows that the amount of MWCNTs dispersed in [C_{14}-C_4-C_{14}im]Br_2 aqueous solution is more than that in [C_{12}-C_4-C_{12}im]Br_2 at the same concentration. Although [C_{14}-C_4-C_{14}im]Br_2 and [C_{12}-C_4-C_{12}im]Br_2 are homologues with only a slight difference in the length of hydrocarbon chain, it is obvious that the dispersing ability of MWCNTs for the former is better than that for the latter. Similar phenomenon was observed for single-chain ionic liquid-based surfactants, 1-alkyl-3-methylimidazolium bromide ([$C_n$mim]Br) in Fig. 6. The ability of [$C_n$mim]Br to disperse MWCNTs also increases with increasing hydrophobic chain length. Generally, hydrophobic tail groups tend to lie flat on the graphitic surface because graphitic unit cells match well with the methylene units of hydrocarbon chain [7,34]. Longer tails mean high spatial volume and more steric hindrance, thus providing greater repulsive forces between individual carbon nanotubes. Meanwhile, the hydrophobic interaction between surfactants and MWCNTs increases with the increase of hydrocarbon chain length.

3.4. Comparison with 1-alkyl-3-methylimidazolium bromide

As known, 1-hexadecyl-3-vinyl-imidazolium bromide has larger ability of dispersing CNTs compared with traditional surfactants, such as SDS, SDBS, and CTAB [24]. Ionic liquid-type Gemini imidazolium surfactants with four-methylene spacer groups ([C_n-C_4-$C_n$im]Br_2, n = 12, 14) have higher surface activity than the corresponding [$C_n$mim]Br [25]. For the sake of comparison, the spectral feature of the MWCNTs/[C_{12}mim]Br dispersion is also shown in Fig. 6. Figs. 5 and 6 exhibit that [C_{12}-C_4-C_{12}im]Br_2 could disperse more carbon nanotubes into the bulk aqueous solutions than [C_{12}mim]Br even at lower concentration. This can be interpreted in terms of the discrepancies in their chemical structures. In order to disperse nanotubes in water, surfactant molecules adsorb on the surface of carbon nanotubes and orient themselves in such a fashion that hydrophobic tail groups face toward the nanotubes surface while hydrophilic head groups extend into the water [31]. The charge repulsion between the hydrophilic head groups of surfactant molecules can prevent the aggregation of carbon nanotubes. Compared with the corresponding [$C_n$mim]Br, the charge repulsion of the cationic imidazole rings between ionic liquid-type Gemini imidazolium surfactant molecules adsorbed on the surface of carbon nanotubes is much stronger, which can be ascribed to its structure with two imidazole ring head groups. As for the surfactant adsorption on MWCNTs, [C_n-C_4-$C_n$im]Br_2 with two hydrophobic long chains has stronger adsorption ability and aggregation ability. So ionic liquid-type Gemini imidazolium surfactants, [C_n-C_4-$C_n$im]Br_2, exhibit much higher properties than [$C_n$mim]Br when dispersing and stabilizing the MWCNTs in aqueous solutions.

3.5. Mechanism of dispersing CNTs

Fig. 7 depicts a schematic illustration of a plausible mechanism of dispersing CNTs by the ionic liquid-type Gemini imidazolium surfactant, [C_{12}-C_4-C_{12}im]Br_2, which has two long hydrophobic chains and imidazole ring head groups. When CNTs are added in [C_{12}-C_4-C_{12}im]Br_2 aqueous solutions, the hydrophobic tail groups tend to lie flat on the graphitic surface by the hydrophobic interaction, while hydrophilic head groups are inclined to extend into the water. Since the surface of CNTs adsorbs some molecules of [C_{12}-C_4-C_{12}im]Br_2, it becomes positive charged. CNTs can be dispersed effectively in aqueous solutions due to the Coulomb force between CNTs.

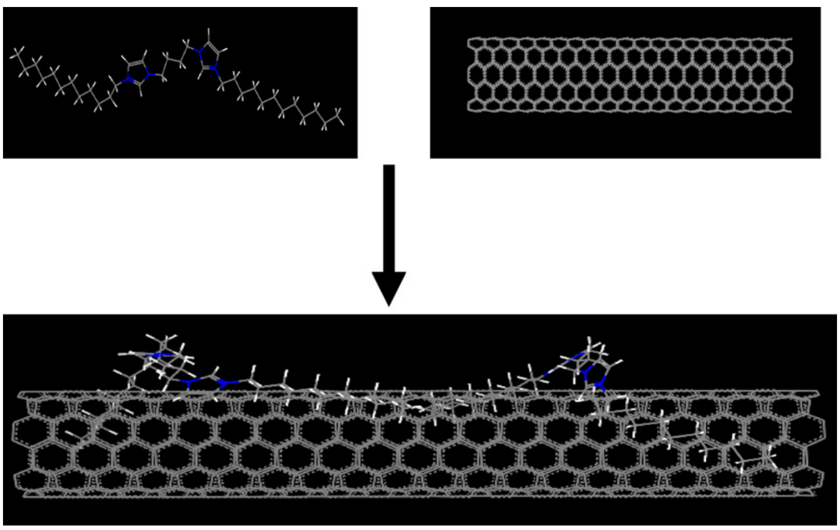

Fig. 7. Schematic representation of how ionic liquid-type Gemini imidazolium surfactant [C_{12}-C_4-C_{12}im]Br_2 molecule adsorb on the CNTs surface.

Table 1
ζ Potential of MWCNTs at different $[C_{12}$-C_4-$C_{12}im]Br_2$ concentrations.

Entry	$C[C_{12}$-C_4-$C_{12}im]Br_2$ (mg/mL)	ζ Potential (mV)
1	0.1	55.9
2	0.2	58.7
3	1.0	61.5

The ζ-potential measurement is an important parameter to understand the stability of colloid particles in aqueous solutions. Table 1 shows the variation of ζ-potential values of MWCNTs with the increasing concentrations of $[C_{12}$-C_4-$C_{12}im]Br_2$. When the concentrations of $[C_{12}$-C_4-$C_{12}im]Br_2$ is only 0.1 mg/mL, ζ-potential for the surface of MWCNTs is up to 55.9 mV, which can be ascribed to strong adsorption ability of $[C_{12}$-C_4-$C_{12}im]Br_2$ on the surface of MWCNTs. With the increase of concentration of $[C_{12}$-C_4-$C_{12}im]Br_2$ in aqueous solutions, the positive value of ζ-potential becomes larger, which shows more surfactant molecules adsorb on the surface of MWCNTs. For MWCNTs dispersed in $[C_{12}$-C_4-$C_{12}im]Br_2$ aqueous solution, the relatively high positive charges of ζ-potential make them stabilize in the aqueous solutions due to Coulomb force between CNTs. This further confirms the stability observed over time for MWCNTs dispersions of $[C_{12}$-C_4-$C_{12}im]Br_2$. Therefore two long hydrophobic chains and imidazole ring head groups make ionic liquid-type Gemini imidazolium surfactant have a superior ability of dispersing CNTs.

4. Conclusions

In summary, we have demonstrated that ionic liquid-type Gemini imidazolium surfactant ($[C_n$-C_4-$C_nim]Br_2$, n = 12, 14) can disperse MWCNTs effectively even at very low concentration. The resulted MWCNT suspensions have quite high stability, and no precipitation was observed after stored at room temperature for more than one month. The dispersed amount of MWCNTs first increased and then decreased with increasing the concentration of $[C_{12}$-C_4-$C_{12}im]Br_2$. Compared with the single-chain ionic liquid-based surfactant, the Gemini ionic liquid-based surfactants have advantages to prepare stable MWCNT suspensions. For $[C_n$-C_4-$C_nim]Br_2$ surfactants with longer hydrocarbon chain, their ability of dispersing MWCNTs is superior. This work will provide guideline to design more suitable molecule structure of MWCNT dispersant.

Acknowledgments

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