

Electrochemistry of Methanofullerene Films: Simultaneous Cyclic Voltammetry and Electrochemical Quartz Crystal Microbalance Studies

H. Zhang,¹ X. Huang,¹ L. Z. Fan,¹ B. Nin,¹ Y. Q. Wu,¹ L. P. Zheng,² Y. Cao²

¹Department of Chemistry, Beijing Normal University, Beijing 100875, People's Republic of China

²Institute of Polymer Optoelectronic Materials and Devices, South China University of Technology, Guangzhou 510640, People's Republic of China

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ABSTRACT: Electrochemical properties of eight methanofullerene films, prepared on electrodes by casting, were examined by means of cyclic voltammetry and electrochemical quartz crystal microbalance (EQCM) technique in acetonitrile solution. Compared with C₆₀ film, the film stability with respect to dissolution is associated with the length of the alkyl chains introduced into C₆₀ cage and is strongly dependent on the nature of the cation of the supporting electrolyte. TBA⁺, Li⁺, K⁺, and Na⁺ were selected to observe the effect of counteranions on the electrochemical behavior of methanofullerene films. In TBAPF₆ solution, when short chains, such as formic ether or butyl butyrate groups, were introduced into C₆₀ cage, the film stability was less than that of C₆₀. When the length of the alkyl chain was extended to butyl 12 alkanooate group, the film would not

dissolve until the third reduction process because the long alkyl chain could inflect and encase the cations into the film. In KPF₆ or NaClO₄ solution, however, the solubility of the salt formed by K⁺ or Na⁺ with methanofullerene anion was higher than that formed by TBA⁺ with methanofullerene anion. Li⁺ (alkali metal) affected the behavior of the films in different ways. The possible electron-transfer mechanisms of methanofullerene films in various supporting electrolyte solutions were presented. Furthermore, the film images observed by atomic force microscopy indicated that TBA⁺ and Li⁺ affected the behavior of the methanofullerene films in different ways. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 3634–3640, 2006

Key words: methanofullerenes; films; electrochemistry; EQCM

INTRODUCTION

The number of synthetic fullerene derivatives continues to increase at a relative fast pace¹. In view of their prospective applications in constructing nanostructured materials², it is necessary to investigate the properties of their thin solid films on the electrode surface, considering the best understanding on the stability of their anions on the film, counter ion incorporation into the film, and the resulting change of the film structure during the electrochemical processes³. Films of pristine fullerene, C₆₀ or C₇₀, have been widely studied since their discoveries^{3–24}. As opposed to the well-defined consecutive one-electron reductions observed on the voltammograms of C₆₀ or C₇₀ solution^{25,26}, the electrochemical behavior of their films is rather complicated, depending considerably on the nature of the supporting electrolyte cation and

the electroreductions in the films^{3–24}. In contrast, the behavior in thin solid films of fullerene derivatives has not been much known so far, although their electrochemical properties dissolved in organic solution have been widely studied, which is mainly because of the difficulty associated with synthesizing them in bulk quantities^{27,28}.

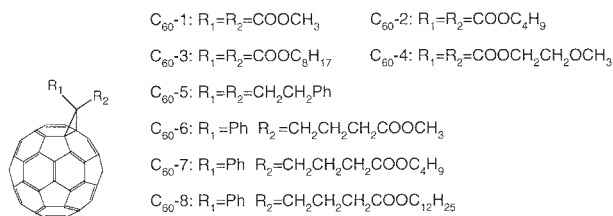
Electrochemical quartz crystal microbalance (EQCM) is very useful and informative in probing electrode surface reactions involving the electrogeneration, electro-doping or undoping of the film with counteractions and film electro-dissolution^{29–31}; Bard and coworkers, Kadish and coworkers, and Oyama and coworkers have applied this technique to study fullerene films in acetonitrile solvents^{5–7}. The measurements of mass-transport processes within fullerene films during electrochemical reactions showed the ingress/egress of TBA⁺ into/out from the film after the first and second peaks and the dissolution/deposition of fullerene films after the third peak³.

We have synthesized a series of methanofullerenes bearing different chain lengths as shown in Scheme 1. Their electrochemical and photoelectrochemical behavior when dissolved in organic solution have been studied^{32,33}. Here, we report the electrochemical be-

Correspondence to: L. Z. Fan (lzfan@bnu.edu.cn).

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Scheme 1. Structures of the methanofullerene C_{60} -1 – C_{60} -8.

havior of their solid films in acetonitrile solution by simultaneous cyclic voltammetry (CV) and EQCM. Comparing with C_{60} film, we will analyze the impacts and mechanisms of different supporting salts to electrochemical behavior of the thin solid film of these methanofullerenes during redox processes. The influence of the structure and chain length of the these methanofullerenes and the nature of the cation of supporting electrolyte on electrochemical properties of the films and the stability with respect to films are reported. Moreover, by atomic force microscopy (AFM), the surface images of these films are also presented.

EXPERIMENTAL

Chemicals

C_{60} (99.9%) purchased from Perkin University was directly used without further purification. Methanofullerene C_{60} -1 – C_{60} -8 were synthesized and characterized by UV-vis, FTIR(KBr) FABMS, and ^{13}C NMR, which have been reported in literature³³. TBAPF₆, KPF₆, NaClO₄, and LiClO₄ purchased from Aldrich were dried for 6 h in vacuum prior to use. Acetonitrile (CH₃CN) of a high performance liquid chromatography grade from Labscan Asia Co. (Thailand) was used as received. Toluene (C₆H₅CH₃) (Park Co. Dublin, Ireland) was dried with sodium and refluxed for 6 h before distillation and then stored in the presence of sodium.

Instrumentation

Simultaneous CV and EQCM were carried out with a model CHI 420 electrochemical workstation (CH Instrument Inc., Austin, TX). An 8-MHZ atom (AT)-cut quartz crystal resonator was used. Geometric area of the electrode was 0.196 cm². Mass sensitivity of this quartz crystal based on the Sauerbrey Equation was 1.4 ng Hz⁻¹. The diameter of the quartz crystal was 13.7 mm. A central 5-mm diameter circular gold spot served as the working electrode. A Pt wire and a KCl saturated Ag/AgCl electrodes were used as auxiliary and reference electrode, respectively. Each experiment was performed with a new Au quartz electrode. The images of films were obtained by AFM with the use of

a HL-II style computerized scanning probe microscope.

Preparation of the novel class C_{60} derivatives solid films

In our experiments, the thin solid films of these methanofullerenes were prepared by drop-coating. Because of its simplicity, this method has become the most common technique for the preparation of fullerene films for electrochemical studies³. Concretely, the thin drop-coated solid films were cast directly onto the Au/quartz electrode from the casting solution. That is, a 6-mL aliquot of toluene containing methanofullerene (~1 mM) was spread and evaporated repeatedly onto the Au electrode surface until a film covering the entire Au surface was obtained. The solvent was dried up rapidly to form relatively fine smooth solid films (note that the thickness of film should be moderate, otherwise the oscillator of quartz will not work.). The surface coverage was about 5.1×10^{-9} mol cm⁻².

All experiments were performed under high-purity nitrogen atmosphere and at room temperature.

RESULTS AND DISCUSSION

As reported, the main factor affecting the behavior of the fullerene film upon electroreduction is the nature of the cation of the background electrolyte.^{3,7} For charge compensation, either the counteranion must enter the fullerene film during its electroreduction or the film is removed from the electrode surface. We selected TBA⁺, Li⁺, K⁺, and Na⁺ to observe the effect of counteranions because these cations are known to affect the electrochemistry of the fullerene films in different ways. We intend to make a contrast about the influence of the cations with different size to the film stability. TBA⁺ with large size was reported to stabilize the fullerene film. Li⁺ ingress and egress of the fullerene film is important in view of the prospective application as the anode redox system for secondary batteries. Furthermore, K⁺ and Na⁺ with significantly small size were used to compare the effect on the film with TBA⁺ and Li⁺. Only the first CV cycle and the corresponding frequency change are presented. For different films of the same methanofullerene, both voltammogram and microgravimetrogram were quite reproducible.

TBAPF₆ solution

For reference and comparison, we first measured the simultaneous cyclic voltammogram and frequency response for the C_{60} film cast on the Au-quartz electrode in acetonitrile solution containing 0.1 mol L⁻¹ TBAPF₆. The results are shown in Figure 1 and are consistent with previous reports³⁻¹⁰. Within the poten-

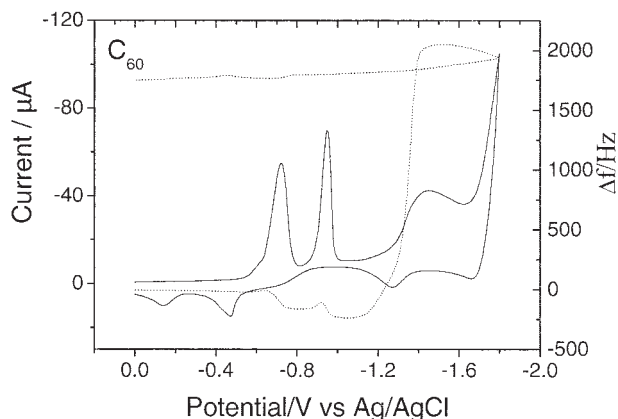


Figure 1 C_{60} film drop-coated onto Au/quartz electrode for cyclic voltammogram (real line) and frequency change (dashed) vs. potential traces in acetonitrile solution with 0.1M TBAPF₆ were simultaneously recorded. Potential scan rate, 50 mV s⁻¹.

tial range scanned (to be accordant, all potential ranges scanned were between 0 and -1.7 V vs. Ag/AgCl), three pairs of redox peaks associated with C_{60} reductions and reoxidations were observed, where the first two peaks are due to film reduction and the large splittings between their reduction and reoxidation peaks are interpreted as the reorganization of the structure, while the third pair of peaks correspond to the third reduction of the film and the reoxidation of dissolved C_{60}^{3-} in solution. On the other hand, the frequency change accompanied during redox process also dominates the results. The first reduction peak was accompanied by a frequency decrease, i.e., mass increase, due to the electrodeposition of a $(TBA^+)(C_{60}^-)$ film. The mass increased again upon the second reduction, indicating that the $(TBA^+)_2(C_{60}^{2-})$ was formed in the film. Then a rapid mass drop is associated with the electrodisolution of the film when C_{60}^{3-} was formed. Almost no frequency changes were seen on the reversal potential scan during reoxidation steps.^{5,6}

From C_{60-1} to C_{60-8} , as shown in Scheme 1, the length of chain introduced into C_{60} cage increases gradually. For C_{60-1} , two short ester groups, formic ether groups, are introduced into C_{60} cage. The features of both CV and frequency change are different from those of C_{60} film in the TBA^+ solution [Fig. 2(a)]. Three cathodic peaks could be observed at its CV curve, but only one reoxidation peak corresponding to the third reduction presented in the anodic scan. A frequency increase was observed soon after the first reduction occurred and continued upon the second reduction peak, after which the frequency kept unchanged associated with the third reduction. This indicates that the film started to dissolve even from its first reduction, which prevailed over the formation of $TBA^+C_{60-1}^{1-}$ or $(TBA^+)_2C_{60-1}^{2-}$ film. Then the third

pair of redox peaks should be ascribed to solution process, corresponding to no mass change on the surface of the electrode. The similar shapes of CV and frequency responses for over C_{60-2} – C_{60-7} were obtained except for small potential shifts in their CVs, indicating that the methanofullerene films with short chains dissolve in acetonitrile by electrochemical reduction even from the first step. That is, the stability of these methanofullerene films in TBA^+ solution may be lower than that of C_{60} film.

When the ester group in C_{60} cage was changed to butyl 12 alkanooate, C_{60-8} , however, presented different behavior. As shown in Figure 2(b), three well-defined cathodic and three anodic peaks were obtained. The first reduction peak was accompanied by the frequency decrease due to the TBA^+ ingress into the film, and then the frequency dropped again corresponding to the second reduction peak, indicating that the cation continued to enter into the film to form $(TBA^+)_2C_{60-8}^{2-}$. Similar to C_{60} film, this film eventually dissolved into the solution when C_{60-8}^{3-} was formed, corresponding to the rapid increase of the frequency after the second reduction. In the reverse scan, three anodic peaks were accompanied by a fre-

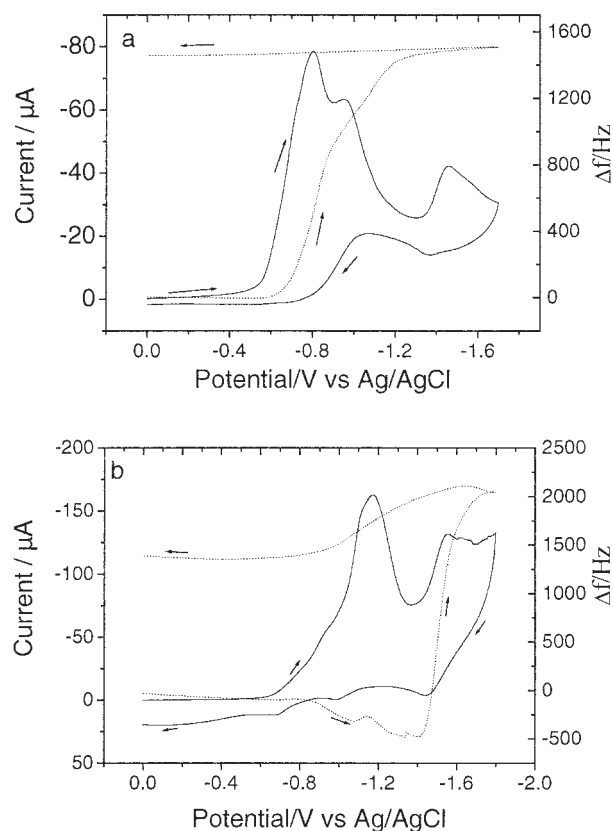
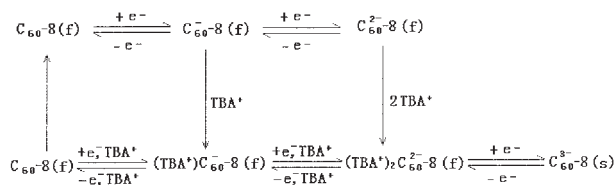


Figure 2 (a) C_{60-1} film and (b) C_{60-8} film drop-coated onto Au/quartz electrode for cyclic voltammogram (real line) and frequency change (dashed) vs. potential traces in acetonitrile solution with 0.1M TBAPF₆ were simultaneously recorded. Potential scan rate, 50 mV s⁻¹.



Scheme 2.

quency decrease, hence these peaks correspond to reoxidation and redeposition of $(\text{TBA}^+)\text{C}_{60-8}^{2-}$ and $\text{TBA}^+\text{C}_{60-8}^-$. After the oxidation was completed, the frequency decreased to some extent. The final frequency was higher than the initial one and all the reoxidation peaks were smaller than corresponding reduction peaks, indicating that this process involved not only injection of cations but also dissolution of ions. The result suggests that introduction of long chain into C_{60} cage will facilitate TBA^+ to electrochemically stabilize the film, most probably because the long chains might inflect and encase the cations into the film.

The mechanism for the electrochemical process of C_{60-8} film in TBA^+ solution is summarized in Scheme 2, where s and f represent "solution" and "film" species, respectively.

KPF₆ and NaClO₄ solutions

Electrochemistry of the C_{60} film is reportedly unstable in a solution containing an alkali metal cation, and it was inferred that alkali metal cations form electrochemically inactive salt with C_{60} anion^{3,4,9}. Figure 3(a,b) show CVs and frequency changes of C_{60-8} in KPF₆ and NaClO₄ solution, respectively. In contrast to TBA^+ solution, the electrochemical behavior of methanofullerenes with short chain, C_{60-1} , or long chain, C_{60-8} , appeared to be quite the same (so here we show only figures for C_{60-8}). The most evident feature of frequency change accompanying the CV is the dramatic increase of frequency from the beginning of the first reduction, showing that the film dissolves at the first reduction process. For K^+ solution, three cathodic peaks as well as three relative small anodic peaks were observed [Fig. 3(a)]. The frequency decreases were substantially small following the second and third reduction steps, and did not change in the reoxidation process. This implies that K^+ may form electrochemically inactive salt with C_{60-8}^- ion, some of which remained on the electrode surface even after the electrochemical activity was lost. Therefore, when small amount of dissolved anions were oxidized at the anodic direction (see CV curve), the frequency remained constant. In NaClO₄ solution [Fig. 3(b)], similar behavior was observed except for that no anodic peak was observed and the frequency did not change at all after

the first reduction. This reflects that most of C_{60-8}^- ion may all form inactive salt with Na^+ .

LiClO₄ solution

As reported, Li^+ effect on the voltammetric behavior of C_{60} film is special and not consistent with that of other alkali metals, which is proved again for the films of methanofullerenes. Three cathodic peaks were observed (Fig. 4), while reoxidation peaks were not seen during the anodic scan. Although the CV behavior for either short chain methanofullerenes, from C_{60-1} to C_{60-7} , or long chain methanofullerene, C_{60-8} , appeared the same, their accompanying frequency changes were quite different. As shown in Figure 4(a), the rapid increase of the frequency associated with the first reduction peak indicates the film dissolution. Then the frequency virtually did not change in the potential range of the second reduction but decreased during the third reduction, suggesting that the second reduction correspond to the reduction of C_{60-1} anion dissolved in the first reduction, and then Li^+ formed inactive salt with C_{60-1}^{2-} anion and remained on the

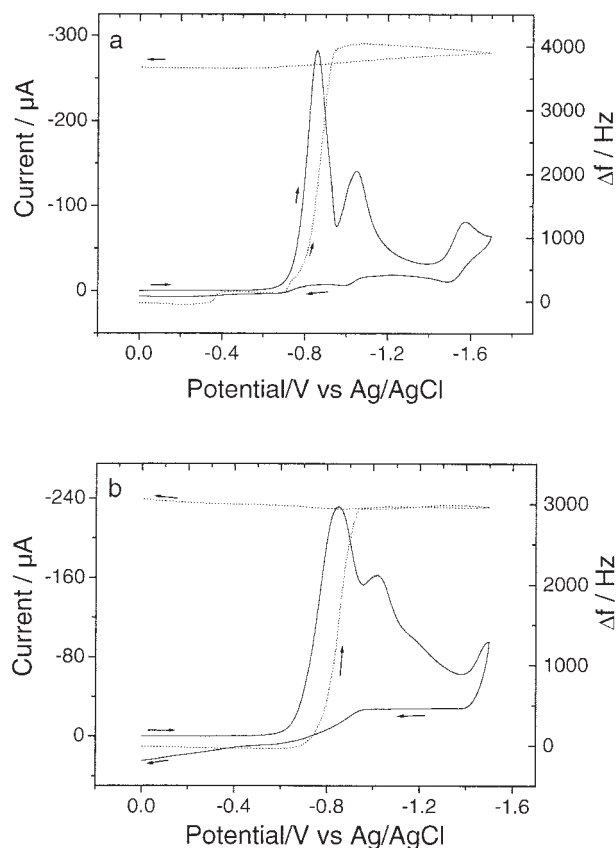


Figure 3 C_{60-8} film drop-coated onto Au/quartz electrode for cyclic voltammogram (real line) and frequency change (dashed) vs. potential traces in acetonitrile solution with (a) 0.1M KPF₆ and (b) 0.1M NaClO₄ were simultaneously recorded. Potential scan rate, 50 mV s⁻¹.

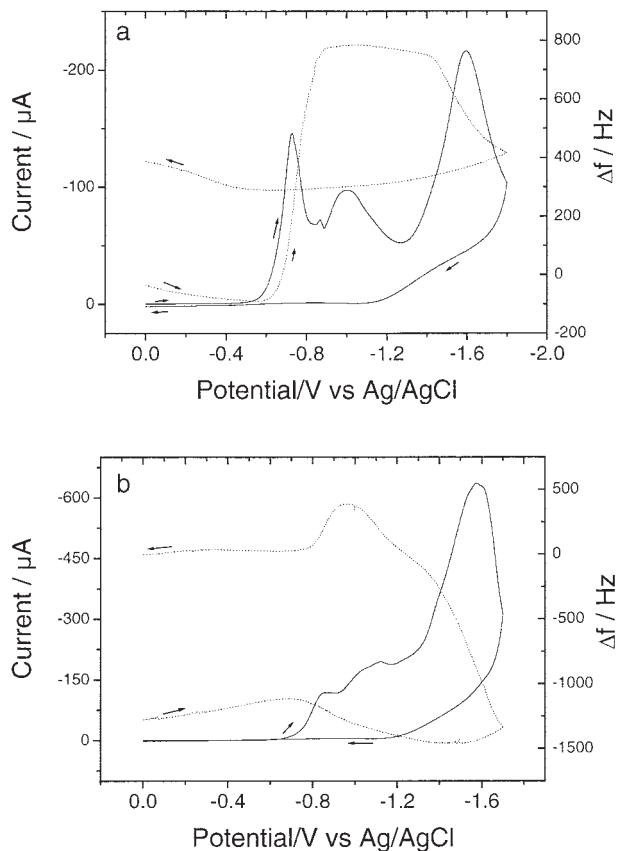


Figure 4 (a) C_{60-1} film and (b) C_{60-8} film drop-coated onto Au/quartz electrode for cyclic voltammogram (real line) and frequency change (dashed) vs. potential traces in acetonitrile solution with 0.1M $LiClO_4$ were simultaneously recorded. Potential scan rate, 50 mV s^{-1} .

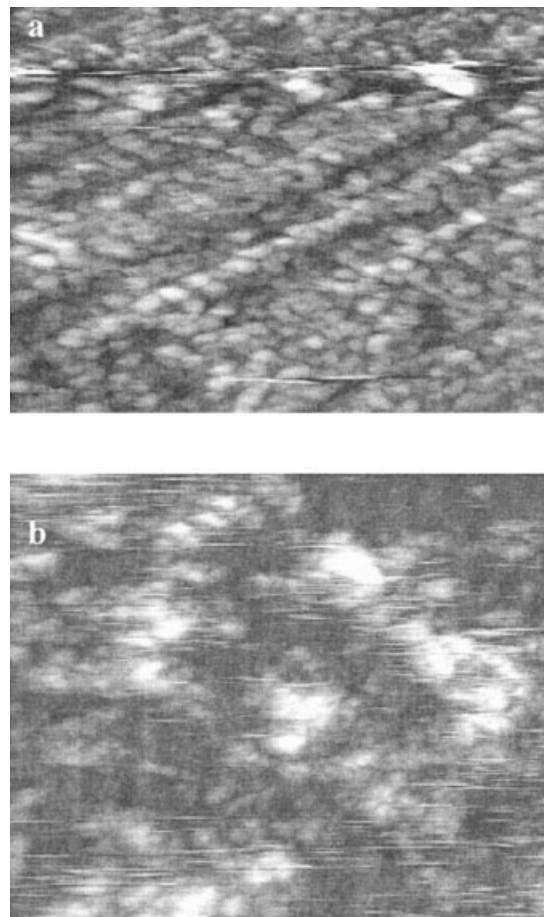
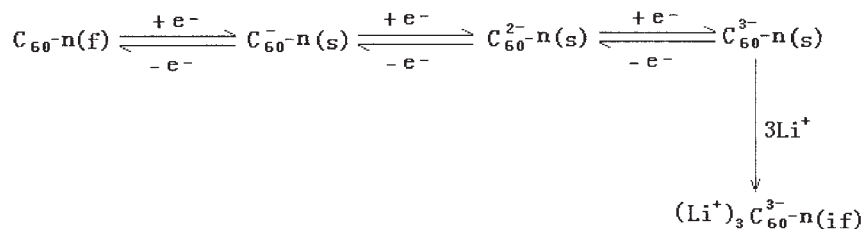


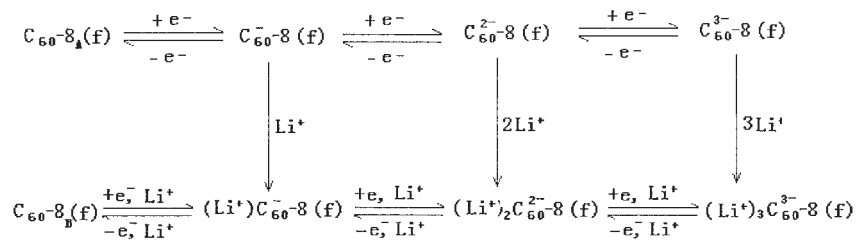
Figure 5 AFM image ($4000 \times 4000 \text{ nm}^2$) of the surface of C_{60-8} films on Au/quartz electrode after potential scanning in acetonitrile solution with 0.1M (a) TBAPF₆ and (b) $LiClO_4$.

electrode surface. Therefore, neither frequency change nor anodic peaks were observed during the anodic scan. On the other hand, for C_{60-8} , the frequency de-

crease accompanied all three reductions, most likely that Li^+ uptake prevails over the film dissolution [Fig. 4(b)]. During the anodic scan, the frequency increased



Scheme 3.



Scheme 4.

TABLE I
The First Reduction Peak Potentials for C₆₀-1 – C₆₀-8 in Different Solutions

	The first reduction potentials of C ₆₀ -1 – C ₆₀ -8/V vs. Ag/AgCl							
	1	2	3	4	5	6	7	8
TBA ⁺	-0.8	-0.78	-0.78	-0.79	-0.76	-0.77	-0.79	-0.92
K ⁺	-0.79	-0.81	-0.79	-0.80	-0.79	-0.80	-0.81	-0.90
Na ⁺	-0.78	-0.79	-0.71	-0.78	-0.72	-0.78	-0.80	-0.89
Li ⁺	-0.80	-0.80	-0.78	-0.77	-0.78	-0.80	-0.80	-0.90

first, then decreased a little, and finally kept unchanged. This complicated behavior of the frequency indicates this process involves dissolution and redeposition of the film. However, considering together with the behavior of CV during anodic scan, there must be some insulating film formed and remaining on the surface of the electrode so that these processes do not appear in the CV.

Schemes 3 and 4 illustrates the mechanisms for the electrochemical processes of methanofullerenes films with short alkyl chains (see Scheme 3) or long alkyl chains (see Scheme 4) in Li⁺ solution.

Here $n = 1-7$ and "if" represents "inactive film." Once C₆₀²⁻- n was reduced to C₆₀³⁻- n , an inactive film (Li⁺)₃C₆₀³⁻- n may deposit on the surface of the electrode.

For C₆₀-8, two forms of the neutral films, labeled as C₆₀-8_A and C₆₀-8_B might be obtained. An insulating film was deposited on the surface of the electrode accompanying the generation of (Li⁺)₂C₆₀²⁻-8 during anodic process.

AFM images

To obtain more information about the structure of electrode surface in different supporting electrolytes, AFM was conducted on the film as a function of the redox in different solutions. Figure 5 displays the AFM images of C₆₀-8 film on a Au electrode after electrochemical potential scans in TBA⁺ and Li⁺ solution, respectively. It is apparent that the structures of these two resulted films are different. The film in TBA⁺ solution consists of grains (~240 nm) with a uniform distribution [Fig. 5(a)], whereas the film in Li⁺ solution shows the shape of floccule. This is consistent with the results of CV and EQCM, suggesting the electrochemical processes of the methanofullerene films in TBA⁺ and Li⁺ solution are different.

The first reduction peak potential comparison for C₆₀-1 – C₆₀-8

Table I lists the first reduction peak potentials of C₆₀-1 – C₆₀-8. It can be seen, in the horizontal line, the reduction potentials differ from one compound to the other in the same solution. Significantly, C₆₀-8 was reduced

at more negative potential, while the reduction potentials for other compounds (from C₆₀-1 to C₆₀-7) varied by less than 40 mV from each other. This reflects the weaker electron-accepting ability of C₆₀-8 with respect to other compounds because of the influence of long chain substituent with electron-donating nature. On the other hand, in the vertical line, there are no large changes in the reduction potentials for the compound in the different solutions, indicating that cations have a small effect on the first reduction potentials for the same compound, although EQCM results show the different electrochemical mechanisms of methanofullerene in the solutions with various cations.

CONCLUSIONS

The stability of the methanofullerene films with respect to the dissolution is associated with the length of the alkyl chains introduced into C₆₀ cage and strongly dependent on the nature of the cation of the supporting electrolyte. When short chains, such as formic ether or butyl butyrate groups, were introduced into C₆₀ cage, the film stability was less than that of C₆₀ and began to dissolve into the solution at its first reduction step whatever be the solution (TBA⁺, Na⁺, K⁺, or Li⁺ solution). When the length of the alkyl chain was extended to butyl 12 alkanooate, the film would not dissolve until the third reduction step in TBA⁺ solution but still dissolved into K⁺ or Na⁺ solution. Li⁺ affected the behavior of the films in different ways. For methanofullerene film with short alkyl chains, the film dissolved from the first reduction but redeposited later to form electrochemically inactive films and remain on the surface of the electrode. While for that with long alkyl chain group, Li⁺ entered the film to form inactive film over the first reduction, and partly breaks away at the oxidation state.

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