ELECTROCHEMICAL STUDIES OF C₆₀-FULLERENE MICROCRYSTALS ATTACHED TO A SOLID STATE ELECTRODE

LIM EI BEE

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ELECTROCHEMICAL STUDIES OF C_{60} FULLERENE MICROCRYSTALS ATTACHED TO A SOLID STATE ELECTRODE

By

LIM EI BEE

Thesis Submitted to the School of Graduate Studies, University Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

March 2002
Dedicated to my parents, my brothers Leng, Hall, Teong and Hao, my friends Chew, Sim and Lee, for their support, patience and friendship.
Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in the fulfilment of the requirement for the degree of Master of Science

ELECTROCHEMICAL STUDIES OF C_{60}-FULLERENE MICROCRYSTALS ATTACHED TO A SOLID STATE ELECTRODE

By

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December 2001

Chairman: Associate Professor Tan Wee Tee, Ph.D.

Faculty: Science and Environmental Studies

Electrochemistry behaviours of C_{60} attached with physical method: solvent casting and mechanically attachment, to various solid-state electrode surfaces (gold, glassy carbon and platinum) were studied in aqueous and non-aqueous solution containing a variety of doping cations such as Group I alkali-metal cations and quaternary-ammonium R_{4}N^{+}. Electroreduction and reoxidation of microcrystalline C_{60} in 0.1 M TBAPF_{6}/ACN showed one to four pairs, characteristic solid state current-potential curve, with only first and second pairs appeared to be stable and reversible.

In contact with 0.1 M TBAPF_{6}/ACN, the large peak separation and small maximum at reverse scan observed with cyclic voltammetric experiments, as well as the current-time transients obtained in chronoamperometric experiments produce evidence of nucleation and growth (N&G) processes at the electrolyte-solid-electrode interface.
C₆₀ cast onto glassy carbon or gold electrodes showed very different results of cyclic voltammetry (CV), chronocoulometry (CC), and chronoamperometry (CA) in aqueous and non-aqueous solutions. With the presence of alkali-metal cations in aqueous electrolyte, MₙC₆₀ (M = Li⁺, Na⁺, K⁺, Rb⁺, Cs⁺, n = 1 to 6) was formed at the end of potential scan, and the loss of faradaic activity when oxidizing MₙC₆₀ at reverse of potential scan indicated the formation of non-electroactive and irreversible species. In aqueous solution, CV results showed a parallel shift in reduction peak position as the sizes of cation increased.

A glassy carbon electrode modified by C₆₀ coat was used to mediate the oxidation of cysteine in contact with an aqueous electrolyte containing potassium cation. Under conditions of cyclic voltammetry, the potential of cysteine is lowered by approximate 100 mV and current is enhanced significantly relative to the situation prevailing when a bare glassy carbon electrode is used. Mediation also occurs when the potential range covered include that of C₆₀/C₆₀⁻ redox couples.
Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGAJIAN ELEKTROKIMIA BAGI C_{60} FULLERIN MIKROKRISTAL YANG TERLEKAT PADA SESUATU ELEKTROD PEPEJAL

Oleh

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Tingkahlaku elektrokimia bagi C_{60} yang dilekatkan secara fizikal melalui “kot larutan” dan “lekatan mekanikal”, kepada permukaan pelbagai elektrod pepejal (emas, karbon kaca dan platinum) telah dikaji dalam larutan akues dan tak akues yang mengandungi pelbagai kation berdop seperti kation logam-alkali Kumpulan I dan kuaternari-ammonium R_4N^+. Elektro-penurunan dan pengoksidaan bagi mikrokristal C_{60} dalam 0.1 M TBAPF_6/ACN menunjukkan satu hingga empat pasang kelok arus-keupayaan yang berperanan pepejal, dengan hanya pasangan pertama dan kedua berkelakuan stabil serta boleh berulang.

Berhubungan dengan 0.1 M TBAPF_6/ACN, pemisahan besar antara puncak dan maksima kecil pada imbasan terbalik yang diperhatikan dengan eksperimen voltammetri berkitar, serta transian arus-masa yang diperolehi dalam eksperimen kronoamperometri menghasilkan bukti bagi proses penukleusan dan tumbesaran (N&G) pada antara-fasa elektrolit-pepejal-elektrod.

C_{60} yang disapu ke atas elektrod karbon kaca atau emas menunjukkan keputusan voltammetri berkitar (CV), kronokulometri (CC), dan kronoamperometri (CA)
dalam larutan akues dan tak akues yang amat berbeza. Dengan kehadiran kation logam-alkali dalam elektrolit akues, $M_nC_{60}$ ($M = Li^+, Na^+, K^+, Rb^+, Cs^+, n = 1 \text{ to } 6$) telah dihasilkan pada akhir imbasan keupayaan, dan juga, kehilangan aktiviti faradaik semasa pengoksidaan $M_nC_{60}$ pada imbasan keupayaan terbalik menandakan pembentukan sesuatu spesies yang tidak eletroaktif dan tidak berulang. Dalam larutan akues, keputusan CV menunjukkan satu anjakan selari pada kedudukan puncak penurunan dengan saiz kation yang meningkat.

Satu elektrod karbon kaca yang diubahsuiakan dengan kot $C_{60}$ telah digunakan untuk memediankan pengoksidaan sistena dalam suatu elektrolit akues yang mengandungi kation potassium. Di bawah kawalan voltammetri berkitar, keupayaan sistena telah diturunkan lebih kurang 100 mV dan arusnya jelas ditingkatkan berbanding dengan situasi biasa iaitu bila sesuatu elektrod karbon kaca tanpa ubahsuiian digunakan. Pemedianan juga berlaku bila julat keupayaan yang dilingkungi termasuk pasangan redoks $C_{60}/C_{60}^-$. 
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I certify that an Examination Committee met on 11 February 2002 to conduct the final examination of Lim Ei Bee on her Master of Science thesis entitled “Electrochemical Studies of C$_{60}$-Fullerene Microcrystals Attached to a Solid State Electrode” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

Date: 23-03-2002
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4.9 Cyclic voltammograms of C₆₀ attached onto 3 mm diameter glassy carbon electrode by (a) and (b) mechanical attached (MA) of microcrystalline C₆₀, (c) Solvent casting (SC) of 20 μL C₆₀/CH₂Cl₂, in contact with 0.1 M NaCl/H₂O, scan rate 100 mV/s ................................................................................................. 4.23

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4.11 Plot of peak current versus peak potential at varying scan rates for varying amount of C₆₀ coated onto 1 mm diameter gold electrode surface via SC method, experiment was performed in 0.1 M TBAPF₆ in ACN, with scan rate ranged from 10 to 400 mV/s ......................................................................................................................... 4.32

4.12 The shifts of peak potential versus scan rate for 20 (+), 40 (·) and 60 (+) μL of C₆₀ coated onto 1 mm diameter gold electrode surface via SC method, performed in 0.1 M TBAPF₆/ACN, with scan rate varied from 10 to 400 mV/s ............... 4.33
4.13 Plot of peak current for first redox couple versus scan rate of 20, 40, and 60 µL C_{60}/CH₂Cl₂ coated onto 1 mm diameter gold electrode via SC method, performed in 0.1 M TBAPF₆/ACN, scan rate ranged from 10 to 400 mV/s................................. 4.33

4.14 Plot of log₁₀ i_{pc} (a) and i_{pa} (b) versus log₁₀ v, for 40 µL C_{60}/CH₂Cl₂ coated onto 1 mm diameter gold electrode via SC method, in contact with 0.1 M TBAPF₆/ACN........................... 4.34

4.15 CV of 20 µL C_{60}/CH₂Cl₂ coated onto 1 mm diameter gold, reduced in 0.1 M TBAPF₆/ACN, swept with scan rates 10, 15, 20, 30, 40, 50, 60, 80, 100, 125, 150, 175, 200, 250, 300, 350, 400 mV/s, third cycle is shown........................... 4.34

4.16 CV of 60 µL C_{60}/CH₂Cl₂ coated onto 1 mm diameter gold, reduced in 0.1 M TBAPF₆/ACN, swept with scan rates 10, 15, 20, 30, 40, 50, 60, 80, 100, 125, 150, 175, 200, 250, 300, 350, 400 mV/s, third cycle is shown........................... 4.35

4.17 CV of microcrystalline C₆₀ attached mechanically onto 1 mm diameter gold electrode, reduced in 0.1 M TBAPF₆/ACN, swept with scan rates 10, 15, 20, 30, 40, 50, 60, 80, 100, 150, 200, 250, 300, 350, 400 mV/s, second cycle is shown......................... 4.35

4.18 Cyclic voltammograms for 20 µL C_{60}/CH₂Cl₂ coated onto 3 mm diameter glassy carbon by SC method, reduced in 0.1 M KCl, with scan rate (a) 20, (b) 50, (c) 100, (d) 200 mV/s.................. 4.38

4.19 Cyclic voltammograms for 20 µL C_{60} coated onto 3 mm diameter glassy carbon by SC method, reduced in 0.1 M KCl with various scan rates. From bottom to top: 5, 10, 20, 30, 50, 70, 100, 150, 200, 300, 400, 500 mV/s................................. 4.38

4.20 Plot of total charge transferred versus scan rate for 20 µL C_{60} coated onto 3 mm diameter glassy carbon electrode by SC method, supporting electrolyte 0.1 M KCl................................. 4.39

4.21 Plot of current of peak (i) versus scan rate for 20 µL C_{60} coated onto 3 mm diameter glassy carbon electrode by SC method, supporting electrolyte 0.1 M KCl................................. 4.39

4.22 Plot of peak potential of peak (i) versus scan rate for 20 µL C_{60} coated onto 3 mm diameter glassy carbon electrode by SC method, supporting electrolyte 0.1 M KCl................................. 4.40

4.23 Plot of the first reduction-reoxidation peak current versus varying amount of C_{60}/CH₂Cl₂ coated onto 1 mm diameter gold electrode via SC method, performed in 0.1 M
4.24 CV of varying amount of C_{60}/CH_{2}Cl_{2} coated onto 1 mm diameter gold electrode via SC method, (a) 20 µL, (b) 40 µL, (c) 60 µL, reduced in 0.1 M TBAPF_6/ACN, using 100 mV/s, swept past the first redox couple, third cycle is shown.

4.25 CV of microcrystalline C_{60} attached onto 1 mm gold electrode via MA method, reduced in 0.1 M TBAAPF_6/ACN, using 100 mV/s, swept past the first redox couple, third cycle is shown.

4.26 Cyclic voltammograms for varying amount of C_{60}/CH_{2}Cl_{2} coated onto 1 mm diameter gold electrode via SC method, (a) 10, (b) 20, (c) 40, (d) 80 µL; performed in 0.1 M TBAPF_6/ACN, using scan rate 100 mV/s, swept past the second redox couple, third cycle is shown.

4.27 CV of microcrystalline C_{60} attached (MA) onto 1 mm diameter gold electrode, performed in 0.1 M TBAPF_6/ACN at scan rate 100 mV/s, with different amount of microcrystalline used: (a), (c) larger quantity, (b), (d) less, swept past the first (a, b) and second (c, d) redox couple, three cycles are shown.

4.28 Cyclic voltammograms for varying amount (in volume, µL) of C_{60}/CH_{2}Cl_{2} coated onto 3 mm diameter glassy carbon GC electrode using SC method, performed in 0.1 M KCl, swept with 100 mV/s from -200 to -1800 mV.

4.29 Cyclic voltammograms of (a) 2, (b) 5, (c) 20, (d) 60 µL C_{60}/CH_{2}Cl_{2} coated onto 3mm GC electrode via SC method, reduced in 0.1 M KCl, swept with 100 mV/s from -200 to -1800 mV.

4.30 Total charge transferred (i) and number of electron transferred, n per molecule (ii) versus amount of C_{60}/CH_{2}Cl_{2} coated onto 3 mm diameter glassy carbon electrode via SC method, performed in 0.1 M KCl with 100 mV/s, swept from -200 to -1800 mV.

4.31 Plot of peak potential versus temperature for first reduction-reoxidation couple of 20 µL C_{60}/CH_{2}Cl_{2} coated onto 1 mm diameter gold electrode, performed in 0.1 M TBAPF_6, with 100 mV/s.

4.32 Plot of peak current versus peak potential at varying scan rate for first reduction-reoxidation couple of 20 µL C_{60}/CH_{2}Cl_{2} coated onto 1 mm diameter gold electrode, performed at 5, 25 and 45°C, in 0.1 M TBAPF_6/ACN, 100 mV/s.
4.33 CV of first reduction-reoxidation couple of C₁₀₀/CH₂Cl₂ coated onto 1 mm diameter gold electrode studied in 0.1 M TBAPF₆/ACN at different temperatures, swept with 100 mV/s, third cycle is shown ................................................................. 4.57

4.34 CV of first and second reduction-reoxidation couple of C₁₀₀/CH₂Cl₂ coated onto 1 mm diameter gold electrode reduced in 0.1 M TBAPF₆/ACN at 5 and 60°C, swept with 100 mV/s, three cycles are shown ................................................................. 4.57

4.35 Plot of first reduction-reoxidation peak current versus peak potential for 20 μL C₁₀₀/CH₂Cl₂ coated onto 1 mm diameter gold electrode via SC method, reduced in (a) 0.5 M and (b) 1.0 M TBAPF₆/ACN, scan rates from 10 to 400 mV/s ................................................................. 4.60

4.36 Cyclic voltammograms of 20 μL C₁₀₀/CH₂Cl₂ coated onto 3 mm diameter glassy carbon electrode via SC method, reduced in (a) 2.0, (b) 1.0, (c) 0.5, (d) 0.2, (e) 0.1, (f) 0.05 M KCl/H₂O as the supporting electrolyte, scanned with 100 mV/s from −200 to −1800 mV ................................................................. 4.61

4.37 Cyclic voltammograms of 20 μL C₁₀₀/CH₂Cl₂ coated onto 3 mm diameter glassy carbon electrode via SC method, reduced in (a) 0.05, (b) 0.5 M KCl/H₂O as the supporting electrolyte, scanned with 100 mV/s from −200 to −1800 mV ................................................................. 4.61

4.38 CV of 20 μL C₆₀ coated onto 1 mm diameter gold electrode by solvent casting method, in ACN containing 0.1 M (a) TBACIO₄, (b) TOACIO₄. Scanned from −200 to −2000 mV using 100 mV/s, first cycle is shown ................................................................. 4.64

4.39 CV of 1st reduction couple of 20 μL C₆₀ coated onto 1 mm diameter gold electrode by solvent casting method, in ACN containing 0.1 M TBAX (X = (a) PF₆⁻, (b) BF₄⁻, (c) ClO₄⁻, (d) Cl⁻), scanned with 100 mV/s, three cycles each and third cycle is shown ................................................................. 4.65

4.40 CV of 2nd reduction couple of 20 μL C₆₀ coated onto 1 mm diameter gold electrode by solvent casting method, in ACN containing 0.1 M TBAX (X = (a) PF₆⁻, (b) BF₄⁻, (c) ClO₄⁻, (d) Cl⁻), scanned with 100 mV/s, three cycles each and third cycle is shown ................................................................. 4.65

4.41 Cyclic voltammograms of microcrystalline C₆₀ attached mechanically onto 1 mm diameter gold electrode, reduced in (a) 0.1 M LiClO₄/ACN, (b) 0.1 M NaClO₄/ACN, (c) 0.1 M LiClO₄/DMSO, (d) 0.1 M NaClO₄/DMSO, scan with scan rate of 100 mV/s, two cycles are shown. 20 μL C₁₀₀/CH₂Cl₂ deposited by solvent casting method onto (e) 1 mm diameter
gold electrode, (f) 1 mm diameter platinum electrode, reduced in 0.1 M NaClO\textsubscript{4}/DMSO, scan with scan rate of 100 mV/s, 1\textsuperscript{st} cycle is shown...........................

4.42 Cyclic voltammograms of 20 \( \mu \)L \( C_{60}/CH_2Cl_2 \) coated onto 3 mm diameter glassy carbon electrode reduced in 1.0 M KX (X = Cl, Br, I, NO\textsubscript{3}, OH) and 0.5 M K\textsubscript{2}Ox with scan rate 100 mV/s........................................ 4.69

4.43 Cyclic voltammograms of 20 \( \mu \)L \( C_{60}/CH_2Cl_2 \) coated with SC method onto 3 mm diameter glassy carbon electrode, reduced in 0.1 M (a) CsCl, (b) RbCl, (c) KCl, (d) NaCl and (e) LiClO\textsubscript{4}. For (a) to (d), the potential was swept from -200 to -1800 mV versus Ag/AgCl, and for (e) was swept from -200 to -2000 mV versus Ag/AgCl, all with scan rate of 100 mV/s.............. 4.75

4.44 Cyclic voltammograms of \( C_{60}/CH_2Cl_2 \) coated onto 3 mm diameter glassy carbon using SC method in (a) 1.0 M CsCl, (b) to (d) 1.0 M mixed supporting electrolyte of CsCl:NaCl with ratio 8:2, 5:5, 2:8, (e) 1.0 M NaCl, with scan rate of 100 mV/s.. 4.81

4.45 Cyclic voltammograms of \( C_{60}/CH_2Cl_2 \) coated onto 3 mm diameter glassy carbon using SC method in (a) 1.0 M RbCl, (b) to (d) 1.0 M mixed supporting electrolyte of RbCl:NaCl with ratio 8:2, 5:5, 2:8, (e) 1.0 M NaCl, with scan rate of 100 mV/s......................................................... 4.81

4.46 Cyclic voltammograms of \( C_{60}/CH_2Cl_2 \) coated onto 3 mm diameter glassy carbon using SC method in (a) 1.0 M CsCl, (b) to (d) 1.0 M mixed supporting electrolyte of CsCl:KCl with ratio 8:2, 5:5, 2:8, (e) 1.0 M KCl, with scan rate of 100 mV/s... 4.82

4.47 Current maximum for first reoxidation peak when the direction of the potential on the reverse scan is switched at the foot of process 1\textsuperscript{st} reoxidation. Performed in 0.1 M TBAPF\textsubscript{6}/ACN using 1 mm diameter gold electrode, scan rate 20 mV/s, swept from -200 mV to -1200 mV......................... 4.88

4.48 Current maximum for second reoxidation peak when the direction of the potential on the reverse scan is switched at the foot of process 2\textsuperscript{nd} reoxidation. Performed in 0.1 M TBAPF\textsubscript{6}/ACN using 1 mm diameter gold electrode, scan rate 20 mV/s, swept from -200 mV to -1600 mV................................. 4.88

4.49 Current maximum for first reduction peak when the direction of the potential on the reverse scan is switched at the foot of process 1\textsuperscript{st} reduction. Performed in 0.1 M TBAPF\textsubscript{6}/ACN using 1 mm diameter gold electrode, scan rate 20 mV/s, swept from -200 mV to -1200 mV........................................... 4.89
4.50 Current maximum for second reduction peak when the direction of the potential on the reverse scan is switched at the foot of process 2nd reduction. Performed in 0.1 M TBAPF$_6$/ACN using 1 mm diameter gold electrode, scan rate 20 mV/s, swept from -200 mV to -1600 mV.

4.51 Cyclic voltammograms of microcrystalline C$_{60}$ attached mechanically onto 1 mm diameter gold electrode, scanned with 100 mV/s, reduced in (a) 0.1 M KCl/H$_2$O, followed by reduction in (b) 0.1 M TBAPF$_6$/ACN, run 1, three cycles, (c) run 2, three cycles, (d) run three, 10 cycles, (e) in 0.1 M KCl/H$_2$O.

4.52 Double step chronoamperometry for 20 µL C$_{60}$/CH$_2$Cl$_2$ coated onto 3 mm diameter glassy carbon electrode performed in 0.1 M NaCl/H$_2$O, potential stepped from -200 mV to -1800 mV versus Ag/AgCl with different pulse width. (a) Plot of $i$ versus $t$, from left to right: 150, 200, 250, 300, 500, 1000, 2000 msec, forward and reverse current shown; (b) plot of $i$ versus $t^{1/2}$, from longest line to shortest line: 150 msec to 2000 msec, forward current shown.

4.53 Forward and reverse plot of $i$ versus $r^{1/2}$ of CA, same as those shown in Fig. 4.52 (b), from (a) to (g): 150, 200, 250, 300, 500, 1000, 2000 msec. Noted that the scale of x-axis is the same but y is not.

4.54 Double step chronocoulomograms for 20 µL C$_{60}$/CH$_2$Cl$_2$ coated onto 3 mm diameter glassy carbon electrode, reduced in 0.1 M (a) KCl, (b) NaCl, (c) RbCl, (d) CsCl in aqueous (with background current shown). Potential stepped from -200 mV to -1800 mV versus Ag/AgCl, with 2000 msec pulse width. Highlighted area of (d) is enlarged and shown in (e); (f) plot of $i$ versus $t^{1/2}$ of (d).

4.55 Double step chronocoulomograms for 20 µL C$_{60}$/CH$_2$Cl$_2$ coated onto 3 mm diameter glassy carbon electrode, reduced in 0.1 M KCl/H$_2$O, with background current shown. Potential stepped from -200 mV to -1800 mV versus Ag/AgCl, with 2000 msec pulse width. (a) Plot of $Q$ vs. $t$, (b) plot of $Q$ vs. $r^{1/2}$.

4.56 Plot of $i$ versus $t$ of CA for 10 µL C$_{60}$ coated onto 1 mm diameter gold electrode by solvent casting method, performed in 0.1 M TBAPF$_6$/ACN, initial potential stepped from -200 mV to (i) first, (ii) second and (iii) third redox couples versus Ag/Ag$^+$, with default pulse width of 250 msec.

4.57 Plot of (a) $Q$ versus $t$ and (b) $Q$ versus $r^{1/2}$ of CC for 10 µL C$_{60}$ coated onto 1 mm diameter gold electrode by solvent casting.
method, performed in 0.1 M TBAPF₆/ACN, initial potential stepped from -200 mV to (i) first, (ii) second, and (iii) third redox couples versus Ag/Ag⁺, with default pulse width of 250 msec.

4.58 Cyclic voltammogram of C₆₀ microcrystals attached mechanically onto 125 μm diameter gold disk microelectrode, reduced in 0.1 M TBAPF₆/ACN, scanned from -200 to -1600 mV versus Ag/Ag⁺ with 100 mV/s. 1ˢᵗ and 2ⁿᵈ cycles are shown.

4.59 Scan rate study of C₆₀ microcrystals attached mechanically onto 125 μm diameter gold disk microelectrode, performed in 0.1 M TBAPF₆/ACN, scan from -200 mV to first redox couple versus Ag/Ag⁺, with scan rate of 20, 50, 100, 150, 200, 300, 400, 500 and 600 mV/s (from bottom to top).

4.60 Plot of peak current versus scan rate for C₆₀ microcrystals attached mechanically onto 125 μm diameter gold disk microelectrode, performed in 0.1 M TBAPF₆ in ACN, scanned from -200 mV to first redox couple versus Ag/Ag⁺.

4.61 Scan reversal studies of C₆₀ microcrystals attached mechanically onto 125 μm diameter gold disk microelectrode, performed in 0.1 M TBAPF₆/ACN with 20 mV/s. Current maximum occurred at the reverse scan is switched at the foot of process: (a) 1ˢᵗ reoxidation, (b) 2ⁿᵈ reduction, (c) 2ⁿᵈ reoxidation. (d) is the enlarge part for the highlighted area of (c).

4.62 Double step chronoamperometric (a) - (b) and chronocoulometric (c) experiments of C₆₀ microcrystals attached mechanically onto 125 μm diameter gold disk microelectrode, reduced in 0.1 M TBAPF₆/ACN, potential stepped from -200 to -1600 mV versus Ag/Ag⁺ with default pulse width of 250 msec. (b) is enlarged part of highlighted area on (a).

4.63 In-situ optical microscope pictures for 10 μL C₆₀/CH₂Cl₂ coated onto 1 mm diameter gold electrode; (a) before electrochemical reaction, (b) electroreduction up to first reduction peak at potential -1200 mV versus Ag/Ag⁺ for 360 sec. in 0.1 M TBAPF₆/ACN.

4.64 SEM photos for C₆₀ microcrystals attached mechanically onto 5 mm diameter basal plane graphite electrode (a) before electrochemical reaction; after electrochemical reaction at (b) - (c) -1200 mV, (d) - (f) -1600 mV versus Ag/Ag⁺ in 0.1 M TBAPF₆/ACN.
SEM photos for C₆₀ microcrystals coated via solvent casting method onto 5 mm diameter basal plane graphite electrode, (i) before electrochemical reaction; after electrochemical reaction at (j) -1200 mV, (k) -1600 mV versus Ag/Ag⁺ in 0.1 M TBAPF₆/ACN...

SEM photos for C₆₀ microcrystals attached via solvent casting method onto 5 mm diameter basal plane graphite electrode, reduced at -1800 mV versus Ag/AgCl in aqueous electrolyte containing 0.1 M (a) KCl, (b) RbCl, (c) CsCl...

Comparison of voltammogram for the oxidation of cysteine in 0.1 M KH₂PO₄, pH 7.0, with scan rate of 100 mV/s, scanning in a positive direction from 0 to 1500 mV: (a) without L-cysteine using bare GC electrode; (b) without L-cysteine using modified GC electrode coated with 10 μL C₆₀/CH₂Cl₂ (solution); (c) without L-cysteine using modified GC electrode coated with C₆₀ saturated in toluene (dropwise); (d) oxidative current of 0.2 mM L-cysteine on bare GC electrode; (e) oxidative current of 0.2 mM L-cysteine on modified GC electrode coated with 10 μL C₆₀/CH₂Cl₂ (solution); (f) oxidative current of 0.2 mM L-cysteine on modified GC electrode coated with C₆₀/toluene (dropwise)...

CV of 6.0 mM L-cysteine solution (a) 3 mm diameter GC bare electrode, (b) modified GC electrode, coated with 10 μL C₆₀ from stock solution of 150 μM C₆₀/CH₂Cl₂. Performed in 0.1 M KH₂PO₄ (pH 7.0) electrolyte with scan rate 100 mV/s. Potential swept oxidatively (positively) from E_initial = 0 mV to 1500 mV, and reversed to -1800 mV. Segments 2 and 3 are shown...

Potential cycling of 0.2 mM L-cysteine solution at C₆₀/GC electrodes. Similar conditions as in Fig. 4.66 (b); (i) 1ˢᵗ to 3ʳᵈ cycles, (ii) next 4ᵗʰ to 1⁰ᵗʰ cycles...

Effect of varying C₆₀ dosage (coated on 3 mm diameter GC) on the oxidative currents of 0.2 mM L-cysteine in 0.1 M KH₂PO₄ (pH 7.0), sweep positively from E_initial = 0 mV with a scan rate of 100 mV/s. Segment 2 and 3 are shown. (a) 5 μL; (b) 10 μL; (c) 15 μL; (d) 20 μL. (i) Enlargement of highlighted part in (ii)...

Variation of oxidation current with cysteine concentration using C₆₀ modified GC electrode in 0.1 M KH₂PO₄, pH 7.0, scan rate 100 mV/s, scanning through potential region where C₆₀ is electroactive...