



UNIVERSITI PUTRA MALAYSIA

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

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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**

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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

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Electrochemistry behaviours of C₆₀ 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 R₄N⁺. Electroreduction and reoxidation of microcrystalline C₆₀ in 0.1 M TBAPF₆/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 TBAPF₆/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.

C_{60} 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, M_nC_{60} ($M = Li^+, Na^+, K^+, Rb^+, Cs^+, n = 1$ to 6) was formed at the end of potential scan, and the loss of faradaic activity when oxidizing M_nC_{60} 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 C_{60} 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 C_{60}/C_{60}^{n-} redox couples.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGAJIAN ELEKTROKIMIA BAGI C₆₀-FULLERIN MIKROKRISTAL YANG TERLEKAT PADA SESUATU ELEKTROD PEPEJAL

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Tingkah laku elektrokimia bagi C₆₀ 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 R₄N⁺. Elektro-penurunan dan pengoksidaan bagi mikrokristal C₆₀ dalam 0.1 M TBAPF₆/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 TBAPF₆/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.

C₆₀ 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 elektroaktif 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 diubahsuaikan dengan kot C_{60} telah digunakan untuk memediasikan 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 ubahsuaian digunakan. Pemediasan juga berlaku bila julat keupayaan yang dilingkungi termasuk pasangan redoks C_{60}/C_{60}^{n-} .

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