A Green Method for the Electroorganic Synthesis of New 1,3-Indandione Derivatives

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This is an environmentally friendly method in the field of electroorganic reactions under controlled potential electrolysis, without toxic reagents at a carbon electrode in an undivided cell which involves the (EC) mechanism reaction and comprises two steps alternatively; (i) electrochemical oxidation and (ii) chemical reaction. In particular, the electrochemical oxidation of 4-*tert*-butylcatechol, 4-methylcatechol and 2,3-dihydroxybenzoic acid in the presence of 2-phenyl-1,3-indandione has been studied in a water–acetonitrile (90 : 10) mixture. The research includes the use of a variety of experimental techniques, such as cyclic voltammetry, controlled-potential electrolysis, and spectroscopic identification of products (FT-IR, ^{1}H-NMR, and MS spectrometry).

Key words electro-organic reaction; *ortho*-dihydroxybenzene; quasi-reversible process; (EC) mechanism; 2-phenyl-1,3-indandione

1,3-Indandione derivatives present anticoagulant properties and the synthesis and pharmacological properties of 1,3-indandione derivatives have been investigated.[1—6]

On the other hand, it has been reported that *ortho*- and *para*-hydroquinones and their quinone derivatives are abundant in nature and play important roles in many biological systems.[7—11] In addition, many chemicals of this category demonstrate an antioxidant activity and are able to prevent auto-oxidation *via* the radical formation inhibition.[12] These compounds exhibit a wide variety of physiological and pharmacological properties.[13] They participate in normal cell functions such as neurotransmitters. Furthermore, natural pyrocatechols extracted from plants (catechins and other polyphenols from green tea) have been found to illustrate anticancer properties.[14]

For these reasons, knowledge of the redox properties of these compounds is important for a better understanding of their behavior in biological environments. Redox properties of some derivatives of these compounds in aqueous solutions is documented.[15—22]

We thought that synthesis of a new 1,3-indandione derivatives with both structures of 1,3-indandione and hydroquinone would be useful from the point of view of pharmaceutical properties. This idea prompted us to development of a facile and environmentally friendly reagentless electrochemical method for the synthesis of some new 1,3-indandione derivatives in aqueous solutions with high atomic economy under ambient conditions and in an undivided cell using a graphite electrode and delineates the application of electrochemical studies in the design of an electrochemical method for the synthesis of new 1,3-indandione derivatives (**5a**—**c**).

Electroorganic Reactions of 4-*tert*-Butylcatechol (1a) and 4-Methylcatechol (1b) Figure 1, CV a displays the typical successive cyclic voltammogram (CV) of 0.25 mM 4-*tert*-butylcatechol (**1a**) in aqueous media (H_2O : AN, 90 : 10), containing phosphate buffer (pH=7.0, c=0.15 M). This figure demonstrates one anodic peak (A_1) (at 0.291 V) and one corresponding cathodic peak (C_1) (at 0.105 V), which corresponds to the transformation of 4-*tert*-butylcatechol (**1a**) to *o*-quinone-4-*tert*-butyl (**2a**) and *vice versa* within a quasi-reversible two-electron and two-proton process (ΔEp= 0.186 V).[23,24] A peak current ratio (I_P^{A1}/I_P^{C1}) of nearly unity, particularly during the repetitive potential recycle, can be considered as a criterion for the stability of *o*-quinone produced on the electrode surface under the experimental conditions. In other words, any hydroxylation[25,26] or dimerization[27,28] reactions are too slow to be observed on the time scale of the cyclic voltammetry.

The oxidation of 4-*tert*-butylcatechol (**1a**) in the presence of 2-phenyl-1,3-indandione (**3**) (pK_a=4.1)[29] as a nucleophile was studied in detail. Figure 1, CV b presents the cyclic voltammogram obtained for a 0.25 mM solution of **1a** in the presence of 0.25 mM **3**. The corresponding voltammogram exhibits one anodic peak at 0.364 V (A_1) *vs.* the reference electrode, where the cathodic counterpart of the anodic peak A_1 decreases. In this condition, the positive shift of the A_1 peak in the presence of 2-phenyl-1,3-indandione (**3**) (from 0.291 V in CV a to 0.364 V in CV b) is attributed to the thin film formation of the product (**5a**) on the electrode surface, insulating it.[15]

It is seen that proportionally to the augmentation of the potential sweep rate, the height of C_1 peak increases (Fig. 2). A similar situation is observed when the concentration ratio of 2-phenyl-1,3-indandione (**3**) to **1a** decreased. A plot of the peak current ratio (I_P^{C1}/I_P^{A1}) *vs.* the scan rate confirms the reactivity of *o*-quinones (**2a**) towards **3**, appearing as an increase in the height of the cathodic peak C_1 at higher scan rates (Fig. 2, curve I).

Controlled-potential coulometry was performed for determination of the number of transferred electrons in aqueous solution, containing 0.25 mmol of **1a** and 0.25 mmol of **3** at 0.25 V *vs.* the RE. It is illustrated that proportionally to the advancement of coulometry, all anodic and cathodic peaks decrease and disappear when the charge consumption becomes about 2e$^-$ per molecule of **1a**.

A cyclic voltammogram (CV) of 0.25 mM 4-methylcatechol (**1b**) in aqueous solution (H_2O : AN, 90 : 10) shows one anodic (A_1 at 0.335 V) and the corresponding cathodic peak (C_1 at 0.023 V) which correspond to the 4-methylcatechol

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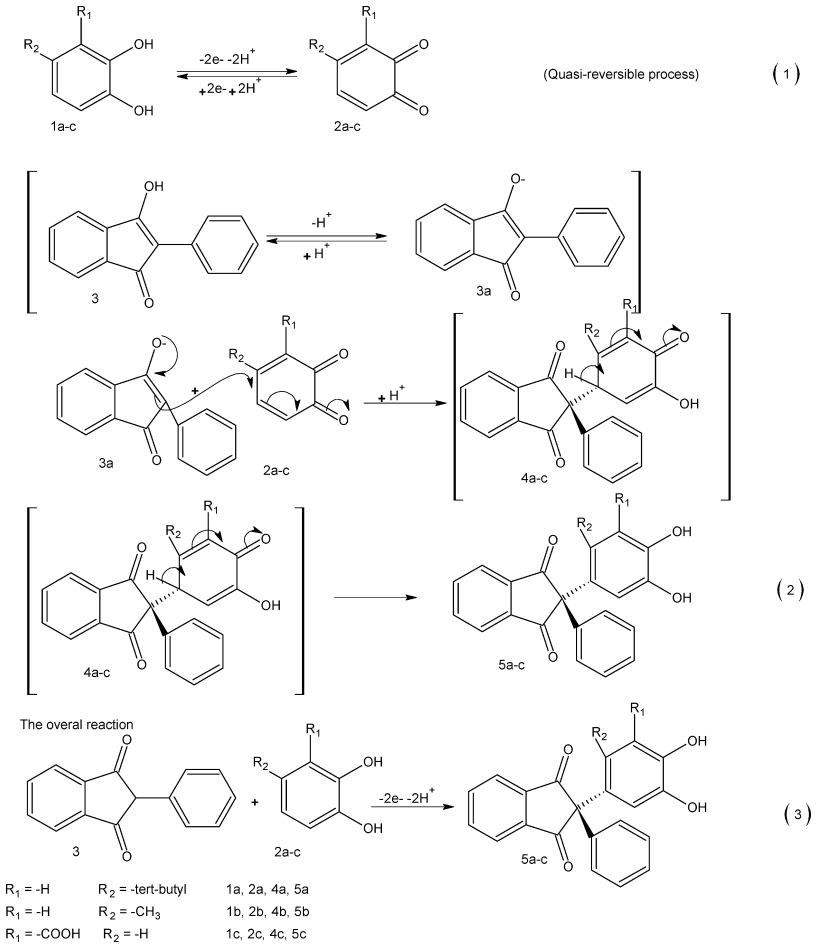

Chart 1

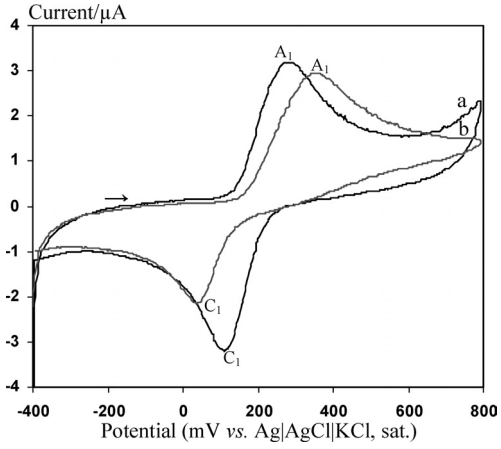

Fig. 1. Comparative Cyclic Voltammograms of 0.25 mM 4-*tert*-Butylcatechol without (a) and with (b) 2-Phenyl-1,3-indandione, at a Glassy-Carbon Electrode ($S=\pi$ mm^2) in Aqueous Solution (H_2O : AN, 90 : 10) Containing of Phosphate Buffer (pH=7.0, c=0.15 M)

Scan rate: 150 mV·s^{-1}.

(**1b**)/*o*-quinone-4-methyl (**2b**) couple within a quasi-reversible two-electron and two-proton process (Fig. 3, CV b). Figure 3, c and d shows the cyclic voltammograms (CVs) of 0.25 mM **1b** in the presence of 0.25 mM **3**, the voltammogram exhibits cathodic counterpart of the anodic peak A_1 decreases and a new cathodic peak C_0 is related to electroreduction of intermediate **4b**. In this figure, voltammogram a is related to the electrochemical oxidation of 2-phenyl-1,3-indandione with an irreversible oxidation peak. Controlled-potential coulometry for determination the number of transferred electrons was performed in aqueous solution containing 0.125 mmol of **1b** and 0.125 mmol of **3** at 0.30 V *vs.* RE. It shown that consumption about 2e$^-$ per molecule of **1b**.

Electroorganic Reactions of 2,3-Dihydroxybenzoic Acid (1c) Figure 4, CV a displays the typical successive cyclic voltammogram (CV) of 0.25 mM 2,3-dihydroxybenzoic acid (**1c**) in aqueous solution (H_2O : AN, 90 : 10), containing phosphate buffer (pH=7.0, c=0.15 M). This CV demonstrates one anodic peak (A_2) (at 0.441 V) and one corresponding cathodic peak (C_2) (at 0.012 V), which corresponds to the transformation of 2,3-dihydroxybenzoic acid (**1c**) to *o*-quinone-3-carboxilic acid (**2c**) and *vice versa* within a quasi-reversible two-electron and two-proton process (ΔEp= 0.429 V).[23,24]

CV b presents the cyclic voltammogram obtained for a 0.25 mM solution of **1a** in the presence of 0.25 mM **3**. The corresponding voltammogram exhibits two anodic peaks at 0.214 V (A_1) and 0.501 V (A_2) *vs.* the reference electrode, where the cathodic counterpart of the anodic peak A_2 decreases. In this condition, a first irreversible peak A_1 (Fig. 4, CVs b and c) is related to the electrochemical oxidation of 2-phenyl-1,3-indandione (**3**). The positive shift of the A_2 peak in the presence of 2-phenyl-1,3-indandione (**3**) (from 0.441 V

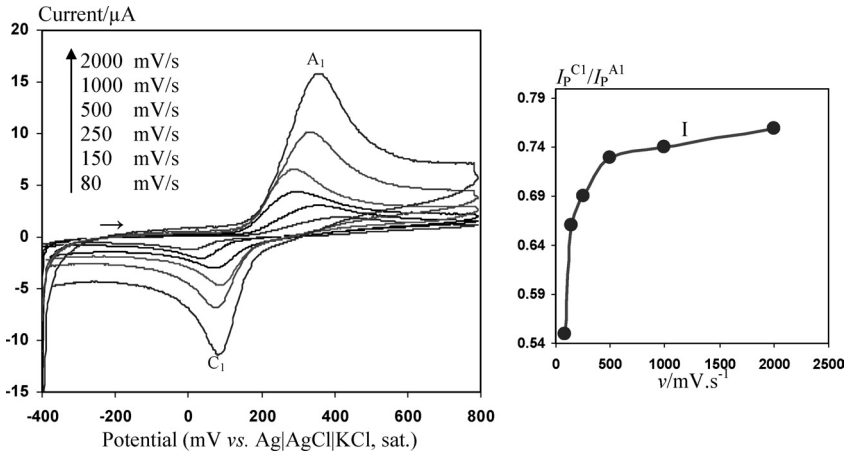

Fig. 2. Typical Cyclic Voltammograms of 0.25 mM 4-*tert*-Butylcatechol in the Presence of 0.25 mM 2-Phenyl-1,3-indandione, at a Glassy-Carbon Electrode ($S=\pi$ mm^2) in Aqueous Solution (H_2O : AN, 90 : 10) Containing of Phosphate Buffer (pH = 7.0, c = 0.15 M) in Different Scan Rates

(I) Variation of the peak current ratio (I_P^{C1}/I_P^{A1}) *versus* scan rate.

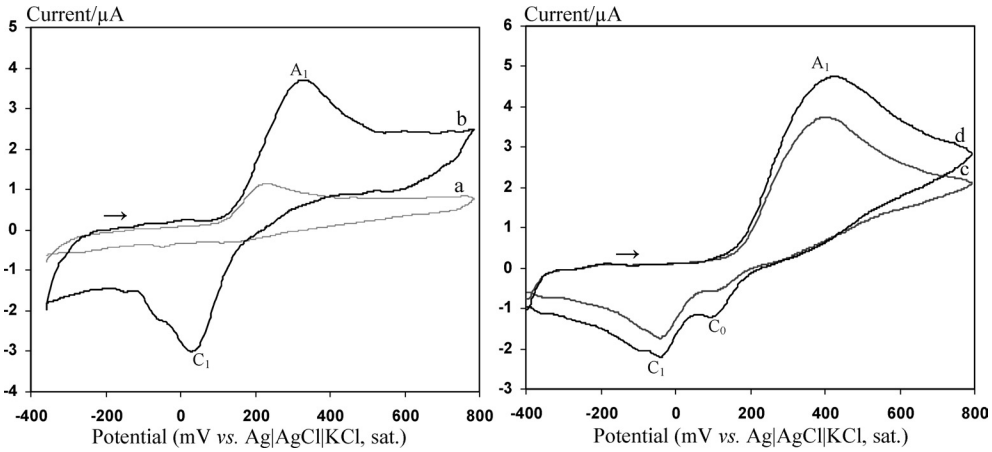

Fig. 3. Comparative Cyclic Voltammograms of 0.25 mM 4-Methylcatechol without (b) and with (c and d) 2-Phenyl-1,3-indandione, (a) Cyclic Voltammogram of 0.25 mM 2-Phenyl-1,3-indandione, at a Glassy-Carbon Electrode ($S=\pi$ mm^2) in Aqueous Solution (H_2O : AN, 90 : 10) Containing of Phosphate Buffer (pH = 7.0, c = 0.15 M)

Scan rates: a, b, c at 250 mV·s^{-1} and d at 500 mV·s^{-1}.

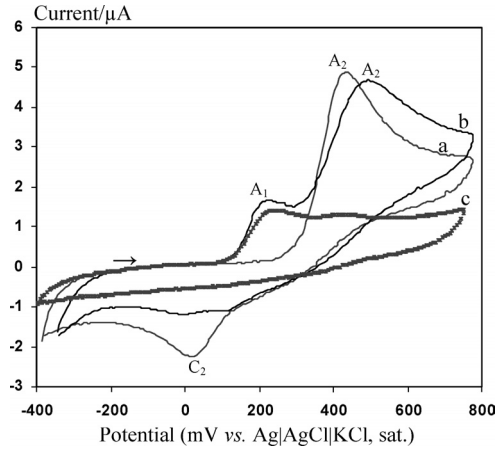

Fig. 4. Comparative Cyclic Voltammograms of 0.25 mM 2,3-Dihydroxybenzoic Acid without (a) and with (b) 2-Phenyl-1,3-indandione, (c) Cyclic Voltammogram of 0.25 mM 2-Phenyl-1,3-indandione, at a Glassy-Carbon Electrode ($S=\pi$ mm^2) in Aqueous Solution (H_2O : AN, 90 : 10) Containing of Phosphate Buffer (pH = 7.0, c = 0.15 M)

Scan rate: 500 mV·s^{-1}.

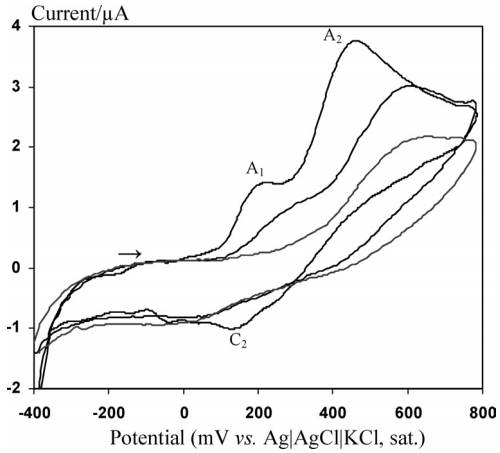

Fig. 5. Continuous Cyclic Voltammograms of 0.25 mM 2,3-Dihydroxybenzoic Acid in the Presence of 0.25 mM 2-Phenyl-1,3-indandione, at a Glassy-Carbon Electrode ($S=\pi$ mm^2) in Aqueous Solution (H_2O : AN, 90 : 10) Containing of Phosphate Buffer (pH = 7.0, c = 0.15 M)

Scan rate: 250 mV·s^{-1}.

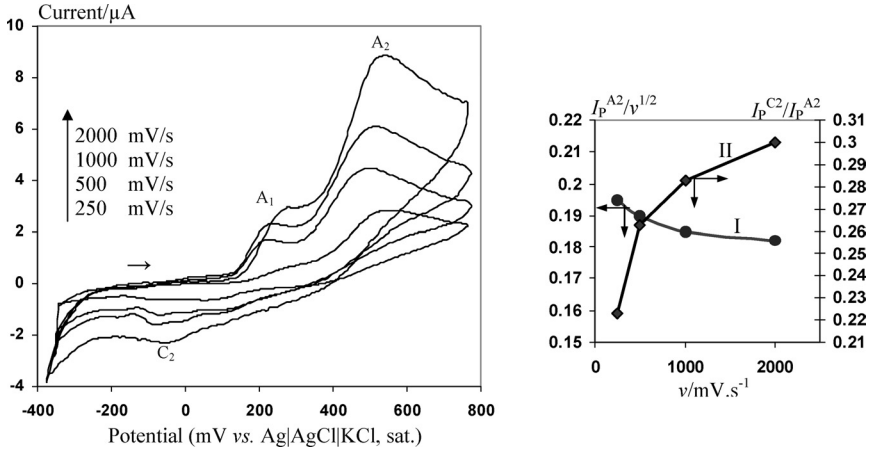

Fig. 6. Typical Cyclic Voltammograms of 0.25 mM 2,3-Dihydroxybenzoic Acid in the Presence of 0.25 mM 2-Phenyl-1,3-indandione, at a Glassy-Carbon Electrode ($S=\pi$ mm^2) in Aqueous Solution (H_2O : AN, 90 : 10) Containing of Phosphate Buffer (pH=7.0, c=0.15 M) in Different Scan Rates

(I) Variation of the peak current function ($I_P^{A2}/v^{1/2}$) (μA s$^{1/2}$ mV$^{-1/2}$) *versus* scan rate. (II) Variation of the peak current ratio (I_P^{C2}/I_P^{A2}) *versus* scan rate.

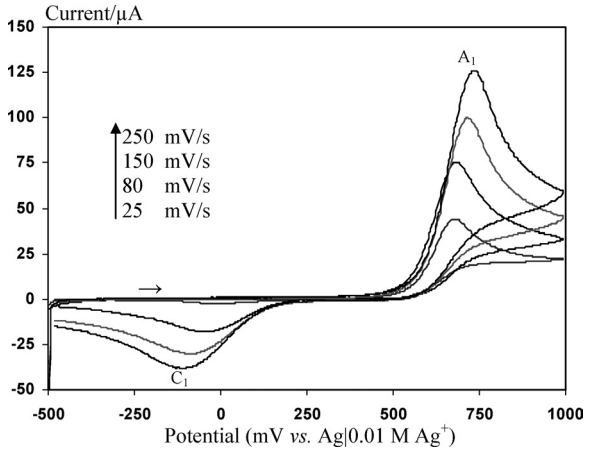

Fig. 7. Cyclic Voltammograms of **5b** at a Glassy Carbon Electrode ($S=\pi$ mm^2) in 0.05 M $LiClO_4$–AN

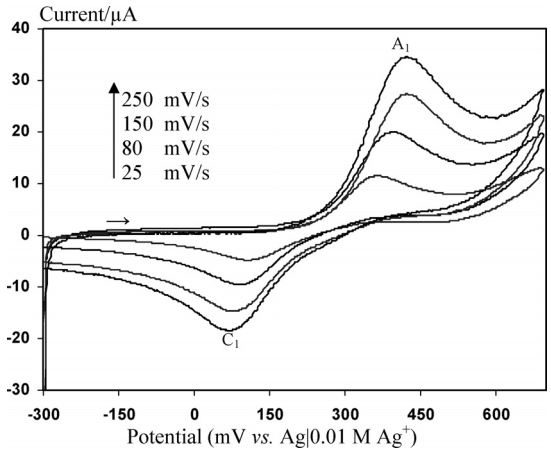

Fig. 8. Cyclic Voltammograms of **5c** at a Glassy Carbon Electrode ($S=\pi$ mm^2) in 0.05 M $LiClO_4$–AN

in CV a to 0.501 V in CV b) is attributed to the thin film formation of the product (**5c**) on the electrode surface, insulating it.[15] This can be inferred from Fig. 5 during the course of continuous cyclic voltammograms.

It is seen that proportionally to the augmentation of the potential sweep rate, the height of C_2 peak increases (Fig. 6). A similar situation is observed when the concentration ratio of 2-phenyl-1,3-indandione (**3**) to **1a** decreased. In this figure, anodic peak A_1 corresponds to the oxidation of 2-phenyl-1,3-indandione (**3**). The current function for the A_2 peak ($I_P^{A2}/v^{1/2}$) changes only slightly with the scan rate increase (Fig. 3, curve I). In fact, such behaviour is indicative of an EC mechanism.[30,31] On the other hand, a plot of the peak current ratio (I_P^{C2}/I_P^{A2}) *vs.* the scan rate confirms the reactivity of *o*-quinones (**2a**) towards **3**, appearing as an increase in the height of the cathodic peak C_2 at higher scan rates (Fig. 3, curve II).

Controlled-potential coulometry for determination the number of transferred electrons was performed in aqueous solution containing 0.125 mmol of **1c** and 0.125 mmol of **3** at 0.40 V *vs.* RE. It shown that consumption about 2e$^-$ per molecule of **1c**.

The existence of –COOH group with electron-withdrawing character at molecular ring (**1c**) causes an augmentation in the activity of respective *o*-quinone toward the side reactions such as polymerization reaction. In spite of the fact that the rate of polymerization reaction increases with augmentation of pH, because the needs to the formation of enolate anion from 2-phenyl-1,3-indandione through acid dissociation reaction, electrochemical synthesis of **5a**—**c** was carried out in pH=7.0.

At the end of this section, a brief discussion about the anodic oxidation of 2-phenyl-1,3-indandione (**3**) during the electrolysis progress seems to be interesting. It should be taken into consideration that although 2-phenyl-1,3-indandione (**3**) has an oxidation peak in 0.214 V (Fig. 4, CV c), the oxidation of **1a**—**c** were more intensive than the oxidation of **3** and circumvent during the preparative reaction, which the resultants led to the new 1,3-indandione derivatives formation as final products (**5a**—**c**). The cause of this phenomenon is the greater anodic current of **1c** (Fig. 4, CV a) in comparison with that of **3** (Fig. 4, CV c) for the same concentration and scan rate in the comparative cyclic voltammograms. The oxidation form of **3** does not present any reaction with the electrochemically generated *o*-quinones. The oxidation of **3** decreases the yield of products in the preparative reaction.

Finally, we obtained the cyclic voltammograms of **5b** and **5c** at glassy carbon electrode in acetonitrile solution that in-

Table 1. Electroanalytical and Preparative Data

Conversion	Peak potentials (V)					Applied potential	Purification	Product yield (%)
	A_1	A_2	C_1	C_2	C_0			
1a→5a	0.364	—	0.026	—	—	0.250 V	H_2O : AN	52
1b→5b	0.411	—	0.046	—	0.095	0.300 V	H_2O : AN	77
1c→5c	0.214	0.501	—	−0.010	—	0.400 V	H_2O : AN	61

volves a transfer of two electrons and two protons to provide the associated hydroquinones (Figs. 7, 8).

Experimental

Apparatus All of the electrochemical experiments were performed with the aid of a setup, comprising a PC PIII Pentium 300 MHz microcomputer equipped with a data acquisition board (PCL-818PG, PC-Labcard Co.) and a custom made potentiostat.[32] The working electrode (WE) used in the voltammetry experiment was a glassy carbon electrode (disc, $S=\pi$ mm^2). A platinum wire was used as the counter electrode (CE). Moreover, the used WE in controlled-potential coulometry was a carbon rod (4 mm in diameter and 3 cm in length). In addition, the macroscale electrolysis was an assembly of three carbon rods (8 mm in diameter and 4 cm in length). The WE potentials were measured *versus* the Ag|AgCl|KCl, sat. as a reference electrode. The cyclic voltammograms of **5b** and **5c** obtained in acetonitrile (AN) containing 0.05 M $LiClO_4$ as a supporting electrolyte and the working electrode (WE) potentials were measured *versus* the Ag|0.01 M Ag^+ couple in the electrolyte solution as a reference electrode.

Also, the NMR spectra were recorded on a Bruker FT-NMR-80 AC, the IR spectra were recorded on a Shimadzu FT-IR-4300 Spectrophotometer. MS spectra were obtained using a HP (Agilent Technology) GC-6890, MS-5973 (EI at 20 eV and 70 eV). The melting point of the products were obtained with the use of an electrothermal melting point model 9200.

Reagents Furthermore, 4-*tert*-butylcatechol, were reagent-grade material and phosphate salts was of pro-analysis grade from Merck. In addition, 2-phenyl-1,3-indandione, 4-methylcatechol and 2,3-dihydroxybenzoic acid were reagent-grade materials from Aldrich and $LiClO_4$, $AgNO_3$ and HPLC-grade acetonitrile (Fluka) were used as received. These chemicals were used without further purification. All experiments were carried out at room temperature.

Electroorganic Synthesis of Products (5a—c) In a typical procedure, 100 ml mixture of water–acetonitrile (90 : 10) containing of phosphate buffer (pH=7.0, c=0.15 M) was pre-electrolyzed at the chosen potential (Table 1), in an undivided cell; subsequently, 2 mmol of 4-*tert*-butylcatechol (**1a**), 4-methylcatechol (**1b**) or 2,3-dihydroxybenzoic acid (**1c**), and 2-phenyl-1,3-indandione (**3**) (2 mmol) were added to the cell. The electrolysis was stopped when the current reached a value that was less than 5% of the initial value. The process was interrupted several times during the electrolysis and the carbon anode was washed in acetone in order to reactivate it. At the end of electrolysis, the precipitated solid was collected by filtration and purification from a mixture of water–acetonitrile (H_2O : AN). Then products were characterized by using FT-IR, ^{1}H-NMR, and MS.

Products Characteristics 2-(2-*tert*-Butyl-4,5-dihydroxyphenyl)-2-phenyl-2*H*-indene-1,3-dione ($C_{25}H_{22}O_4$, **5a**): mp >205 °C. MS (70 eV): m/z=387 $[M+1]^+$ (20), 386 $[M]^+$ (90), 371 $[M-CH_3]^+$ (100), 235 (5), 221 (5), 165 (15), 105 (20), 77 (10). FT-IR ν_{max}^{KBr} cm^{-1}: 3463 (OH, br), 3058 (C–H, sp^3), 1737 (C=O), 1695 (C=O), 1596, 1521 (Ar), 1441, 1429 (*tert*), 1307, 1263, 1213 (C–O), 1039, 698. ^{1}H-NMR (DMSO-d_6, 500 MHz) δ: 1.18 (9H, s, *tert*-butyl), 5.88 (1H, s, catechol ring proton), 6.15 (1H, s, catechol ring proton), 7.35—7.41 (5H, m, Ar protons), 7.97—7.99 (4H, m, Ar protons), 8.02 (1H, s, hydroxy proton), 8.07 (1H, s, hydroxy proton).

2-(4,5-Dihydroxyphenyl-2-methylphenyl)-2-phenyl-2*H*-indene-1,3-dione ($C_{22}H_{16}O_4$, **5b**): mp >205 °C. MS (70 eV): m/z=345 $[M+1]^+$ (20), 344 $[M]^+$ (100), 308 (15), 239 (15), 221 (10), 165 (20), 105 (10), 76 (20). FT-IR ν_{max}^{KBr} cm^{-1}: 3490 (OH, br), 3367 (OH, br), 1741 (C=O), 1699 (C=O), 1606, 1516 (Ar), 1361, 1290, 1255, 1051 (C–O), 771, 611. ^{1}H-NMR (DMSO-d_6, 500 MHz) δ: 1.67 (3H, s, Me), 6.04 (1H, s, catechol ring proton); 6.51 (1H, s, catechol ring proton); 7.24—7.41 (5H, m, Ar protons); 8.01—8.07 (4H, m, Ar protons), 8.64 (1H, s, hydroxy proton), 8.87 (1H, s, hydroxy proton).

5-(2,3-Dihydro-1,3-dioxo-2-phenyl-1*H*-inden-2-yl)-2,3-dihydroxybenzoic Acid ($C_{22}H_{14}O_6$, **5c**): mp >205 °C. MS (20, 70 eV): m/z=330 $[M-CO_2]^+$ (100), 273 (10), 255 (10), 165 (25), 110 (20), 76 (15), 44 (25). FT-IR ν_{max}^{KBr} cm^{-1}: 3475 (OH, br), 3300—2003 (OH, br, carboxylic acid), 1741 (C=O), 1699 (C=O), 1618, 1595, 1444 (Ar), 1380, 1340, 1236 (C–O), 925, 771, 696. ^{1}H-NMR (DMSO-d_6, 500 MHz) δ: 5.44 (1H, s, catechol ring proton), 6.46 (1H, s, catechol ring proton), 7.26—7.30 (5H, m, Ar protons), 7.69—7.74 (4H, m, Ar protons), 7.94 (1H, s, hydroxy proton), 8.21 (1H, s, hydroxy proton), 17.61 (1H, br, carboxilic acid proton).

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