Novel amperometric carbon monoxide sensor based on multi-wall carbon nanotubes grafted with polydiphenylamine—Fabrication and performance

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Abstract

A novel amperometric sensor based on multi-wall carbon nanotubes (MWNT) grafted polydiphenylamine (PDPA), MWNT-g-PDPA, has been developed for the determination of carbon monoxide (CO). Cyclic voltammetry is used to fabricate the modified electrode and to demonstrate the electroactivity of MWNT-g-PDPA-ME toward gaseous CO. The MWNT-g-PDPA-ME exhibits high sensitivity for oxidation of CO in a 0.5 M HClO4 solution. Chronoamperometry is carried to determine the working range of CO at MWNT-g-PDPA-ME. The dependence of the response current on CO concentration is explored under optimal conditions and an excellent linear concentration range between 10 and 200 ppm (correlation coefficient \( r = 0.9941 \)) with a substantially low detection limit of 0.01 ppm is obtained. Further, the response and recovery transients of CO gas at MWNT-g-PDPA-ME are determined.

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1. Introduction

Carbon monoxide (CO), a major atmospheric pollutant is produced by the incomplete combustion of fossil fuels-gas, oil, coal and wood used in boilers, engines, oil burners, gas fires, water heaters, solid fuel appliances and open fires [1]. CO is a toxic chemical, which is harmful to human health. CO poisons are caused by entering the lungs via the normal breathing mechanism and displacing oxygen from the bloodstream. Interruption of the normal supply of oxygen puts at risk the functions of the heart, brain and other vital functions of the body.

Many analytical sensors based on semiconductors [2], catalyst-combustion [3] and electrochemical reactions [4–6] have been developed to measure the CO concentration in air. Low selectivity is one of the major disadvantages with the first two types of CO sensors. On the other hand, amperometric CO sensors offer high sensitivity and better resolution [7]. Also, various room-temperature CO sensors have been developed for commercial application based on semiconductors such as SnO2, ZnO, TiO2, MoO3 and Fe2O3 [8–10]. The main concern is that metal oxide sensors tend to suffer from baseline drifts upon interaction with poisoning species.

Carbon nanotubes (CNTs) have been attracting considerable attentions over the last decade due to their remarkable mechanical and electronic properties [11,12]. CNTs have been proved to be suitable material as electrodes or electrocatalyst supports due to their many advantages such as high electronic conductivity for the promotion of electron transfer reactions and better electrochemical and chemical stabilities in aqueous and non-aqueous solutions. At the same time, conducting polymers are extensively studied for their electrocatalytic activity [13]. Composite materials based on the combination of CNT and conducting polymers have shown properties of the individual components with a synergistic effect [14–16].

The aim of the present study is to develop an amperometric sensor for CO gas. Multiwall carbon nanotubes (MWNT) are suitably functionalized and used to form grafted chains of polydiphenylamine (PDPA), MWNT-g-PDPA, on a glassy carbon
(GC) electrode through electrochemical polymerization. The modified electrode has augmented superior performance for CO detection from the combined influence of MWNT and PDPA. The results are presented here.

2. Experimental

2.1. Reagents

Diphenylamine (DPA, Aldrich) was distilled and used. Poly(ethylene glycol) bis(3-aminopropyl) terminated (PEG-AT) and cetyltrimethyl ammonium bromide (CTAB) were of analytical grades from Aldrich and used as received. MWNTs (10–50 nm in diameter) obtained from CNT Co. Ltd. Incheon, Korea were purified by adopting the following procedure and used. The MWNTs were subjected to air oxidation at 150 °C for a period of 12 h, followed by annealing in argon atmosphere at 800 °C for 1 h. Finally, MWNTs were sonicated in concentrated HCl (10 mg in 20 mL), filtered, washed thoroughly with water and 2-propanol, and dried under vacuum. The residual iron content in the MWNTs was found to be less than 0.3% after the purification process.

2.2. Fabrication of MWNT-g-PDPA modified electrode and sensor studies

2.2.1. Fabrication of MWNT-g-PDPA modified electrode

MWNT-g-PDPA-ME was fabricated by modifying a GC electrode through electrochemical polymerization of a solution consisting of amine functionalized MWNT (MWNT–NH₂) and DPA. Prior to the fabrication of modified electrode, a GC electrode was mechanically polished to a mirror polish using polishing microcloth containing 0.05 μM Al₂O₃ slurry and carefully cleaned with 1:1 HNO₃–H₂O (1:1), ethanol and water by ultrasonication.

Fabrication of MWNT-g-PDPA-ME involved two steps. In the first step, MWNT was amine functionalized (MWNT–NH₂) with PEG-AT. In the second step, cyclic voltammetry was used to deposit MWNT-g-PDPA as a film on the surface of working electrode (GC) to obtain MWNT-g-PDPA-ME (Scheme 1).

MWNT–NH₂ was prepared by adopting the procedure detailed in our previous report [17]. One hundred and fifty milligram of MWNT–NH₂ was homogeneously dispersed in 40 mL of 0.5 M CTAB using ultrasonication (BRANSON Digital Sonifier) for 3 h. Ten millilitre of 50 mM of DPA (in 4 M H₂SO₄) was added to the above solution. Ten millilitre of the solution containing MWNT–NH₂ (0.1%) and DPA (10 mM) was subjected to electrochemical polymerization by continuous potential cycling in the potential range of 0–800 mV using a three electrode cell assembly consisting of GC as a working electrode, saturated calomel electrode (SCE) and platinum wire as reference and counter electrodes, respectively. Green coloured MWNT-g-PDPA was found to be deposited on the surface of GC electrode. The MWNT-g-PDPA-ME was washed with water and stored in a nitrogen atmosphere.
2.2.2. Sensor studies
Measurement of CO gas sensing properties was performed in an electrochemical cell assembly as described in literature [18]. The GC electrode modified with MWNT-g-PDPA was used as a working electrode, and SCE and a platinum wire were used as reference and counter electrodes, respectively.

The standard CO gas (99.999% purity) used in this study contained impurities of less than 10 ppm (mol/mol), which includes nitrogen (<3 ppm), oxygen (<2 ppm), CO2 (<2 ppm) and sulfur (<1 ppm). The CO gas was used without further purification. Various concentrations of sample gases were prepared by diluting CO gas with air. The total flow rate of the CO gas was adjusted at 500 mL/min. Cyclic voltammetry, chronoamperometry and impedance spectroscopy were used to characterize the sensor electrode. Cyclic voltammetry was performed in the potential range between 0 and 1000 mV versus SCE at a scan rate of 50 mV s$^{-1}$. The impedance measurement was performed with the induced ac amplitude of 10 mV and a frequency of 5 MHz-100 mHz. Chronoamperometry was carried out at an applied potential of +550 mV versus SCE under magnetic stirring (150 rpm). After the background current decayed to a steady value, an aliquot amount of CO gas was introduced into the electrolyte solution and the steady state current was measured. The dependence of the amperometric current upon the CO concentration was measured as a sensing signal. All the electrochemical measurements were carried out using EG & G PAR 283 Electrochemical Analyzer with Frequency Response Analyzer 1025.

3. Results and discussion

3.1. Fabrication of MWNT-g-PDPA-ME
Cyclic voltammetry was used for the fabrication of MWNT-g-PDPA-ME through the electro-polymerization of DPA in the presence of MWNT–NH2. Fig. 1 displays the cyclic voltammograms (CVs) recorded during the electrochemical polymerization of the solution consisting of MWNT–NH2 and PDPA. CV patterns of the polymer film deposited from the mixture of MWNT–NH2 and DPA showed a single oxidation wave around 600 mV with a cathodic wave at 300 mV in the reverse scan. In the case of electrochemical polymerization of diphenylamine (in the absence of MWNT–NH2), two anodic processes were noticed at 650 mV and 740 mV with cathodic counter parts at 570 mV and 690 mV, respectively [15,19]. These two anodic peaks were assigned for the generation of N,N′-diphenyl benzidine type radical (DBP$^{*+}$, polaronic form of PDPA structure) and N,N′-diphenyl benzidine dication (DBP$^{2+}$, bipolaronic form of PDPA structure), respectively. In the case of the polymer film deposited with the mixture of MWNT–NH2 and DPA, polaron formation in PDPA structure was augmented by the presence of MWNT–NH2.

The formation of MWNT-g-PDPA was witnessed from the differences in the electrochemical characteristics between polymerization of DPA in the presence and absence of MWNT–NH2. In the first instance, a continuous anodic current was noticed for the initial anodic scan of potential for the solution containing MWNT–NH2. This is in contrast to an oxidation peak noticed around 700 mV for the solution containing only DPA. The continuous increase in peak in the case of the mixture of DPA and MWNT–NH2 is attributed to the simultaneous oxidation of amine sites present in MWNT–NH2 and DPA. As a result, a cross-reaction occurs between the amine cation radicals formed from MWNT–NH2 and DPA. The cation radical sites thus generated in MWNT–NH2 and DPA interact to result in grafting of PDPA onto MWNT. This is the nucleation step for grafting of PDPA onto MWNT. The gradual increase in current value with the number of cycles (Fig. 1) confirms the continuous building up of the MWNT-g-PDPA film over the surface of GC electrode. The grafting of the conducting polymer with amine functionalized MWNT is well documented in our previous reports [14–17].

The field emission transmission electron microscopy (FETEM) image of MWNT-g-PDPA (Fig. 2) reveals the morphology of MWNT-g-PDPA. The surface of MWNT was covered by a layer of PDPA to a thickness of 10–15 nm. This type of morphology is entirely different from the previous reports on the preparation of a polyaniline (PANI) modified MWNT electrode. In the previous report, a partial masking of CNT with PANI has been reported [20].

Hence, electropolymerization of a mixture of DPA and MWNT–NH2 generates MWNT-g-PDPA-ME. This MWNT-g-PDPA-ME was used as a sensor electrode for carbon monoxide gas. The MWNT-g-PDPA-ME showed good sensor characteristics for CO.

3.2. Sensor characteristics of MWNT-g-PDPA-ME
Cyclic voltammetry was performed to understand the electrocatalytic behavior of MWNT-g-PDPA-ME towards the oxidation of CO in a 0.5 M HClO4 solution and compared with MWNT–NH2-ME and PDPA-ME. Fig. 3A shows the CVs recorded at MWNT-g-PDPA-ME in the presence (400 ppm) and absence of CO in a 0.5 M HClO4 solution, respectively. MWNT-g-PDPA-ME shows pronounced electrocatalytic activ-
ity for oxidation of CO in comparison to MWNT–NH2–ME and PDPA-ME (Fig. 3B and C, respectively). This can be clearly witnessed from the enhanced peak current for CO oxidation (515 μA/cm²) with a far less oxidation potential \(E_{\text{ox}}: 570\text{ mV}\) at MWNT-g-PDPA-ME in comparison with MWNT–NH2–ME \(E_{\text{ox}}: 725\text{ mV}\) and PDPA-ME \(E_{\text{ox}}: 732\text{ mV}\). Interestingly, the MWNT-g-PDPA-ME exhibits enhanced sensitivity for oxidation of CO over the nano-Au modified Pt electrode [18] and Sn modified Pt/Nafion assembly [21]. The enhanced peak current and the negative shift in the oxidation overpotential for CO oxidation indicate the enhanced electrocatalytic activity of MWNT-g-PDPA-ME. The shift in the overpotential of CO may be due to the kinetic and transport effect of CO at a MWNT-g-PDPA-ME interface that may probably cause a substantial increase in the electron transfer rate for the oxidation of CO. Further, experiments were performed and the diffusion coefficient \(D\) for the charge transfer reaction was evaluated. The mean value of \(D\) determined as \(2.18 \times 10^{-5}\text{ cm}^2\text{ s}^{-1}\) at MWNT-g-PDPA-ME which was far higher than that observed at MWNT–NH2–ME \(7.46 \times 10^{-5}\text{ cm}^2\text{ s}^{-1}\) and PDPA-ME \(1.93 \times 10^{-12}\text{ cm}^2\text{ s}^{-1}\). The higher \(D\) value at MWNT-g-PDPA-ME substantiates the enhanced electrocatalytic of the modified electrode.

The combined presence of MWNT and PDPA in MWNT-g-PDPA-ME is the reason for the pronounced sensitivity for oxidation of CO. A three-dimensional electron-conductive network extending throughout the MWNT matrix is expected to be formed and result in the improvement of its electronic and ionic transport capacity. The excellent sensitivity for the oxidation of CO at MWNT-g-PDPA-ME arises from the additional surface area provided by PDPA for the oxidation process. Besides, at the MWNT-g-PDPA-ME, CO was oxidized with a lesser overpotential (>150 mV). This is an indication of more efficient electron transfer at MWNT-g-PDPA-ME than at MWNT–NH2–ME.

The effect of scan rate on the voltammetric response of CO at MWNT-g-PDPA-ME was investigated by cyclic voltammetry. The peak currents for CO oxidation increased linearly with the square root of scan rate in the range of 10–300 mV s⁻¹ with a correlation coefficient of 0.9956. This suggests that the process of electrode reaction is controlled by the diffusion process. Fig. 4 shows the CV curves obtained with MWNT-g-PDPA-ME in the presence of different concentrations of CO in 0.5 M HClO₄. Increasing the concentration of CO resulted in an increased current response toward CO oxidation. There was a good linear correlation between CO oxidation current and CO concentration. The effect of electrolyte solution, amount of MWNT–NH2 loading and film thickness were determined and optimized to have better CO sensitivity.
3.3. Factors influencing the oxidation of CO

3.3.1. Effect of electrolyte solution

The effect of electrolyte solution on the oxidation of CO was determined at MWNT-g-PDPA-ME in different acid solutions such as HClO₄, H₂SO₄, HCl and H₃PO₄ through cyclic voltammetry measurements. Among the electrolyte investigated, MWNT-g-PDPA-ME showed a lower onset potential and higher current response for CO oxidation in 0.5 M HClO₄. The enhanced catalytic activity for the oxidation of CO at MWNT-g-PDPA-ME in 0.5 M HClO₄ is due to the following fact. It is reported [22] that there is a competition between anions and surface intermediates of an electrochemical process for sites on the electrode surface. The anions inhibit the electrochemical processes by occupying the active sites. The amount an anion inhibits a process increases with its adsorption strength. The following shows the order of the anions in their adsorption strength:

\[ \text{ClO}_4^- < \text{HSO}_4^- < \text{H}_2\text{PO}_4^- < \text{Cl}^- \]

The properties of the electrolyte improve as the anion adsorption strength decreases, i.e. the lower the anion strength, the better the electrolyte. Chlorate anions have one of the lowest adsorption strengths and hence showed enhanced electrochemical properties when used as a medium for the oxidation of CO at MWNT-g-PDPA-ME.

3.3.2. Effect of MWNT concentration

MWNT-g-PDPA films were deposited on a GC electrode from solutions of DPA (10 mM) having different amounts of MWNT–NH₂. The loading of MWNT–NH₂ increases with increasing the amount of MWNT in the electrolyte solution. The voltammograms of CO oxidation on MWNT-g-PDPA-ME at different MWNT–NH₂ loadings (with the same film thickness of PDPA) are given in Fig. 5. From this figure, it is clear that the onset oxidation is shifted to a lower potential with increasing the MWNT–NH₂ loading up to 30 mg and reaches the minimum at a MWNT loading of 30 mg. Further increasing the MWNT–NH₂ loading resulted in the shift of peak potential to more positive potential. Hence, the optimum MWNT–NH₂ loading of 30 mg was chosen for CO oxidation at MWNT-g-PDPA-ME.

3.3.3. Effect of PDPA thickness

In order to study the effect of PDPA film thickness on the electrocatalytic activity for CO oxidation, electrodes with different film thickness were fabricated. The thickness of modified layer (MWNT-g-PDPA) on a GC electrode was controlled by changing the number of potential cycles during electropolymerization. The charge consumed during the electropolymerization process was used to calculate the thickness of the electroactive film over the GC electrode. The cyclic voltammograms (CVs) for electro-oxidation of CO at MWNT-g-PDPA-ME with different thickness of PDPA, but with the same MWNT loadings, are shown in Fig. 6. No apparent shift in the potential was noted, which means that the peak potential is independent of the film thickness of PDPA on MWNT-g-PDPA-ME towards oxidation of CO. The results are in agreement with many other authors [23,24]. On the other hand, the peak current at the MWNT-g-PDPA-ME increases with an increase in thickness of PDPA up to 10 μm. After a thickness of 10 μm, only a slight increase in peak current was observed. This may be due to the slow diffusion of CO into the thin film and therefore a thickness of 10 μm was found as optimum and used in further experiments.

3.4. Impedance spectroscopy

In order to have an insight into the interfacial behavior of MWNT-g-PDPA-ME, impedance spectroscopic measurements were performed at the MWNT-g-PDPA-ME in the presence and absence of CO. Fig. 7 shows the Nyquist plots obtained from the MWNT-g-PDPA-ME in the absence and presence of 500 ppm CO. The interfacial impedance is dependent on the material com-
Fig. 7. Nyquist plots for the MWNT-g-PDPA-ME showing the effect of the presence of CO; (A) impedance plot in the full-range; (B) the details in the circled zone in (A).

Combination of electrolyte and electrode [25]. Any change in either electrolyte or sensing electrode could lead to variation of the interface impedance. In both cases (in the presence and absence of CO), two semicircles were observed within the frequency range studied. It can be seen from the figure that the large semicircle in the low frequency region was shifted down toward the real $Z'$-axis in the presence of CO. However, the small semicircle in the high frequency region was not affected by the presence of CO gas [26].

The Nyquist plot obtained at MWNT-g-PDPA-ME can be interpreted by an equivalent circuit:

$$\begin{align*}
C_p & \quad \text{CPE} \\
R_b & \quad R_i
\end{align*}$$

The small semicircle in the high frequency region represented by RC elements, where $R_b$ and $C_b$ correspond to the bulk resistance and bulk capacity, respectively. The frequency $f_s$ (at maximum $Z''$) was about 98 kHz. $R_b$ and $C_b$ were determined as 0.9 kΩ and $2.7 \times 10^{-8} \text{ F}$, respectively, for MWNT-g-PDPA-ME in the absence of CO. It is interesting to note that the semicircle in the high frequency region was insensitive to the presence of CO gas. The large semicircle in the low frequency region was attributed to the interfacial contribution comprising of a constant phase element (CPE) and $R_i$. CPE was used to account for the non-ideal behavior of the double layer at the interface of the sensor electrode. $R_i$ represents the charge transfer resistance at the interface. Based upon the equivalent circuit modeling results, $R_i$ was found to be 16.2 kΩ in the absence of CO. $R_i$ was reduced from 16.2 to 12.4 kΩ by the introduction of 500 ppm CO. It is clear from the Fig. 7 that only the interfacial impedance was significantly affected by the presence of CO. Hence, it can be concluded that the oxidation of CO to CO$_2$ occurred at the triple-phase boundary between the electrolyte, MWNT-g-PDPA-ME and CO gas. The application of small AC amplitude (10 mV) induces the oxidation of CO at the triple-phase boundary. The electrochemical oxidation introduced the charge carriers to the electrolyte/electrode interface, leading to change in the kinetics of charge transfer at the electrolyte/electrode interface. This is responsible for the decrease in the interface impedance on introduction of CO gas.

Further, the effect of the dc bias on the impedance behavior of CO at MWNT-g-PDPA-ME was studied. Nyquist plots were obtained by the application of different dc and are shown in Fig. 8. It can be seen from the figure that the interfacial resistance ($R_i$) decreased with an increase in the amplitude of dc bias, implying the effect of the applied bias on the rate-limiting step of the electrode reaction. The results suggest that an electrochemical reaction rather than a surface-catalyzed chemical reaction was occurred for the oxidation of CO at the triple-phase boundary, which resulted in a decrease in $R_i$ with an increase of dc bias at the MWNT-g-PDPA-ME/electrolyte interface [27].

3.5. Amperometric CO sensor behavior

In order to examine the sensing character of MWNT-g-PDPA-ME to CO, amperometry measurements were performed in a 0.5 M HClO$_4$ solution. Fig. 9A shows amperograms recorded during the successive addition of 10 ppm CO under stirred conditions, where the potential of the MWNT-g-PDPA-ME was kept at +550 mV. The amperogram shows an increase in current with subsequent CO injections. A linear relationship between peak currents and CO concentrations was obtained in a range of 10–200 ppm with a correlation coefficient of $r = 0.9941$ (Fig. 9B). The detection limit has been estimated to be 0.01 ppm.

The response and recovery transients of 100 ppm CO gas at MWNT-g-PDPA-ME were determined. The response to each concentration of CO was rather quick; the 96% response and the 98% recovery times to 100 ppm CO were about 2 and 3 s,
respectively. The response rate to CO was confirmed to be also rather high, as in the case of CO detection.

3.6. Selectivity and stability

The selectivity of MWNT-g-PDPA-ME towards CO gas was assessed. Significant interference from NH$_3$, CH$_4$, C$_3$H$_8$ and N$_2$O (500 ppm) were observed at MWNT-g-PDPA-ME. However, when the MWNT-g-PDPA-ME was coated with 0.5% Nafion, such interference from the gases was not detected at MWNT-g-PDPA-ME. The insignificant or no interference from the gases is attributed to the solubility and dielectric properties of the gases at a fluorinated polymer [28], Nafion coated on the MWNT-g-PDPA-ME. The dielectric constants of the gases are less than that of CO and hence do not show interfering signals at the Nafion film/gas interface. The fluorinated polymer membrane effectively prevented these and makes the MWNT-g-PDPA-ME selective towards CO gas.

Long-term stability of the MWNT-g-PDPA-ME was checked for 10 days and CO gas sensing characteristics of the sensors were evaluated continuously. The MWNT-g-PDPA-ME did not show any significant change in the sensing current in comparison to the freshly prepared sensor. Sample gases containing CH$_4$, C$_3$H$_8$, NO$_x$, SO$_2$ and C$_2$H$_3$OH were mixed with CO and the stability of the sensor was also checked. No change in the sensing current was observed. These results demonstrate the stability of the MWNT-g-PDPA-ME against these gases.

4. Conclusions

A new kind of electrode for electrochemical sensing of CO was developed by grafting PDPA on the surface of MWNT. The modified electrode has augmented superior performance for CO detection from the combined influence of functionalized MWNT and PDPA. MWNT-g-PDPA-ME is anticipated to provide a basis for practical approach to CO monitoring.

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