Kinetics of oxygen reduction on gold nanoparticle/multi-walled carbon nanotube hybrid electrodes in acid media

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In this paper, the electrochemical reduction of oxygen has been studied on gold nanoparticle/multi-walled carbon nanotube (AuNP/MWCNT) modified glassy carbon (GC) electrodes in 0.5 M H2SO4 using the rotating disk electrode (RDE) method. The AuNP/MWCNT catalysts were prepared by chemical deposition of AuNPs onto MWCNTs spontaneously grafted with 4-nitrophenyl groups. The composite electrode was characterised by transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS) and cyclic voltammetry (CV). The oxygen reduction behaviour of these electrodes was compared with that of a bulk gold electrode. The AuNP/MWCNT catalyst showed a pronounced electrocatalytic activity towards O2 reduction in acid media. The half-wave potential of O2 reduction on the AuNP/MWCNT catalyst shifted ca 80 mV to more positive potentials as compared to that of a polished Au electrode. The kinetic parameters of oxygen reduction were determined and the specific activity of the hybrid electrode was slightly higher than that of the bulk Au electrode.

1. Introduction

Gold nanoparticles (AuNPs) are frequently employed in the preparation of nanocomposites that are useful in electrochemical and molecular electronics applications. The incorporation of these hybrid materials led to the rapid development of electrode design. The modification of electrodes with nanoparticle-based materials drastically improves the properties of electrodes due to the high surface area-to-volume ratio, chemical stability, and excellent electronic properties. Since their discovery by Iijima [1], the potential application of carbon nanotubes (CNTs) in creating new catalysts has been extensively explored [2]. These materials show a great promise in the fields of electrocatalysis [3], electroanalysis [4] and biosensing [5].

Recently, the study of gold nanoparticles anchored to the surface of CNTs has grown significantly. This hybrid material can be widely used in electrocatalysis and electroanalysis. Here nanoparticle-based materials can be exploited as excellent catalysts for the oxygen reduction reaction. An interesting O2 reduction behaviour on different carbon substrate materials has been reviewed for both alkaline and acid solutions [6–8]. Even though the mechanism of this reaction at different conditions has been extensively studied, there are still many aspects which are unclear. The O2 reduction activity of carbon substrates is low in acid media, which is advantageous for the kinetics investigations of the supported catalysts. Compton and co-workers have recently reviewed the methods used for the preparation of CNT-supported metal nanoparticles and their further applications [9]. Carbon nanotubes can be used as a support material for various catalysts and for this reason their electrocatalytic properties towards O2 reduction are of great fundamental and practical importance. Recently, we studied the oxygen reduction behaviour of acid washed multi-walled carbon nanotubes (MWCNTs) [10]. It was shown on the basis of the RDE results that acid treatment of MWCNTs has a strong effect on the electrocatalytic activity for O2 reduction in acid solution. The half-wave potential shifted more than 250 mV in the negative direction upon MWCNTs treatment in acids. This is related to the removal of metal catalyst impurities (mostly iron), which remain in the carbon nanotube material upon their preparation by chemical vapour deposition (CVD).

The kinetics of oxygen reduction has been thoroughly studied on bulk gold electrodes in acid solution [11–16]. This metal shows a modest O2 reduction activity in acid electrolyte. The structure-sensitivity of O2 reduction on Au(hkl) surfaces is well-established in acid media [12,15,16], the Au(1 0 0) and Au(1 1 1) being the most active and the least active planes, respectively. Obviously,
differences in the structure of Au nanoparticles may influence the electrocatalytic behaviour of the AuNP-modified electrodes. The reduction of O2 on thin-film Au electrodes prepared by vacuum evaporation onto glassy carbon (GC) substrates has been studied in 0.5 M H2SO4 [17]. It was observed that at low overpotentials the 2e\(^{-}\) reduction to H2O2 predominates in acid media and at more negative potentials the peroxide intermediate is further reduced. The specific O2 reduction activity of the Au-film electrodes was almost constant over the entire range of film thickness. Since the first report [17], the reduction of O2 on nanostructured gold electrodes has been extensively investigated [18–47]. Guerin et al. found that the specific activity of vacuum-deposited Au films decreased with decreasing AuNP size below 3 nm [18]. Ohsaka and co-workers studied O2 reduction on gold nanoparticles electrochemically deposited on different substrates [20–27]. Au nanoparticles-electrodeposited electrodes were remarkably active towards oxygen reduction in acid media [20,21]. However, a fundamental understanding of the origin of the electrocatalytic effect observed has not been attained.

The electrocatalytic activity of Au catalysts has also been examined in neutral and alkaline solutions [24–27,34–47]. It is evident, that the nature of gold surface considerably affects the kinetics of electrochemical processes. In the case of O2 reduction, the formation of HO2\(^{\cdot}\) as the final reaction product in the whole potential range studied indicated that the surface of gold nanorods was composed of (1 1 1) and (1 0 0) domains, while (1 0 0) domains were practically absent from the surface [35]. Oxygen reduction has been studied on AuNPs electrodeposited onto NH2-terminated boron-doped diamond [36]. Sarapuu et al. examined the electrocatalytic properties of nanostructured gold electrodes covered with a Nafion film [37]. These electrodes showed a high electrocatalytic activity towards O2 reduction. A two-step reduction of oxygen was observed on Au nanoparticles electrodeposited on an organic template [38].

Attaching gold nanoparticles to nanotube sidewalls is of great interest for obtaining AuNP/CNT hybrids [48]. Zanella et al. [49] performed deposition of gold nanoparticles on the surface of MWCNTs functionalised with aliphatic bifunctional thiols through a direct solvent-free procedure. Carillo et al. [50] used multilayered polymer films to mediate the attachment of AuNPs to a MWCNT surface. Jiang et al. [51] attached selectively AuNPs to chemically functionalised surface sites on N-doped CNTs. Sainsbury et al. reported a detailed experimental and theoretical study of the self-assembly of gold nanoparticles at the surface of carbon nanotubes [52]. They observed that under certain conditions the coverage of gold nanoparticles is very sensitive to the nature of surface modification and the environment, pointing the way to the rational design of CNT-based nanoscale devices with potentially widespread application. Hu et al. introduced an effective and facile reduction approach for the fabrication of CNT-supported Au nanoparticle composite nanomaterials, using linear polyethyleneimine (PEI) as both a functionalising agent for the MWCNTs and a reducing agent for the formation of AuNPs [53]. Their results indicated that the AuNP/CNT composites possess good electrocatalytic activity for O2 reduction in 0.1 M H2SO4.

Recently, we have studied the reduction of oxygen on AuNP/MWCNT catalysts obtained by simple preparation procedures [54–56]. In this work a significantly improved method for fabricating an AuNP/MWCNTs/GC electrode has been developed by attaching gold nanoparticles to the reduced nitrophenyl groups on the sidewalls of multi-walled carbon nanotubes. The electrocatalytic reduction of oxygen on these hybrid materials was studied.

2. Experimental

Multi-walled carbon nanotubes (MWCNTs, purity >95%, diam. 30 ± 10 nm, length 5–20 μm) purchased from NanoLab, Inc. (Brighton, MA, USA) were used in this work. The nanotubes were purified as described elsewhere [10] by refluxing in a concentrated HNO3:H2SO4 (1:1) acid mixture for 2 h at 55 °C and then for 3 h at 80 °C. Functionalisation of MWCNTs with nitrophenyl (NP) groups followed a standard procedure described by Dyke et al. [57]. 1 mg of acid treated MWCNTs were suspended in 10 mM aqueous solution of 4-nitrobenzenediazonium tetrafluoroborate (Aldrich), mixed 30 min and filtered. The investigated AuNP/MWCNT catalyst was prepared by chemical reduction of 100 μl of 1% HAuCl4 aqueous solution and 300 μl of 0.75% NaBH4 in an aqueous suspension of functionalised MWCNTs, mixed 30 min and filtered. Scheme of preparation of AuNP/MWCNT catalyst material is shown below (Scheme 1).

Glassy carbon (GC) was used as the substrate material. GC disks were cut from rods (GC-20SS, Tokai Carbon) and were mounted in a Teflon holder. A bulk gold electrode (99.99%, Alfa Aesar) was used for comparison. The geometric area of the electrodes was 0.2 cm2 and the surface was polished to a mirror finish with 1 and 0.3 μm alumina slurries (Buehler) in Milli-Q water. After alumina polishing the electrodes were sonicated in a water bath twice for 3 min. The modified electrodes were composed of the AuNP/MWCNT catalysts deposited as a thin layer on a GC electrode surface. A 20 μl aliquot of the AuNP/MWCNT suspension (1 mg ml\(^{-1}\)) was pipetted onto the glassy carbon surface, allowing the water to evaporate at room temperature.

Glassy carbon plates of 12 × 15 mm\(^2\) and 2 mm thick were used for the X-ray photoelectron spectroscopy (XPS) studies. The XPS experiments were carried out with a SCIENTA SES-100 spectrometer by using a non-monochromatised Mg Ka X-ray source (incident energy = 1253.6 eV), electron take-off angle θ = 90°. A source power of 300 W was used. The pressure in the analysis chamber was 10\(^{-9}\) Torr. While collecting the survey scan, the following parameters were used: energy range = 600–0 eV, pass energy = 200 eV, step size = 0.5 eV, 1 sweep. For the high resolution scan: energy range = 420–390 eV and 100–70 eV for N1s and Au4f regions, respectively, pass energy = 200 eV, step size = 0.1 eV, 10 sweeps were carried out.

![Scheme 1. Preparation of the AuNP/MWCNT catalyst material.](image-url)
Morphological characterisation of AuNP/MWCNT catalysts was performed by transmission electron microscopy (TEM) using a Tecnai 12 instrument operated at a 120 kV accelerating voltage. The samples were prepared by placing a droplet of the AuNP/MWCNT aqueous suspension on formvar/carbon-coated copper grids and dried in air. The images were acquired by observing many different areas of sample, in order to assess its average characteristics.

The rotating disk electrode (RDE) technique was employed for the O₂ reduction experiments. An EDI101 rotator and CTV101 speed control unit (Radiometer, Copenhagen) were used. The electrode rotation rate (ω) was varied from 360 to 4600 rpm. The potential was applied with an Autolab potentiostat/galvanostat PGSTAT10 (Eco Chemie B.V., The Netherlands) and the experiments were controlled with the General Purpose Electrochemical System (GPES) software. A three-compartment glass cell was used for electrochemical measurements. The counter electrode was separated from the working electrode compartment by a glass frit. A Pt foil served as the counter electrode and a saturated calomel electrode (SCE) was used as a reference. All the potentials are referred to this electrode.

The electrodes were electrochemically characterised by cyclic voltammetry (CV) and oxygen reduction was studied in 0.5 M H₂SO₄ (Suprapur, Merck) solution. The CV response of NP/MWCNT modified GC electrodes was recorded in 0.1 M KOH (p.a. quality, Merck). The solutions were saturated with Ar (99.999%, AGA) or O₂ (99.999%, AGA). A continuous flow of gases was maintained over the solution during the electrochemical measurements. All measurements were carried out at room temperature (23 ± 1 °C).

3. Results and discussion

3.1. TEM images of AuNP/MWCNT samples

Surface morphology of AuNP/MWCNT nanocomposites was investigated by TEM. Fig. 1 shows the representative TEM image of the catalyst. The presence of nm-sized Au particles (average particle size of 5–6 nm) is in evidence. During the preparation of AuNP/MWCNT composites, the attachment of Au nanoparticles to nanotube sites not covered by nitrophenyl groups could also occur. The number density of AuNPs present on the sidewalls of MWCNTs was relatively high, but some individual unmodified nanotubes were also observed.

3.2. XPS analysis

The electrode surfaces were further characterised by XPS. The XPS survey spectrum of the nitrophenyl modified MWCNTs is shown in Fig. 2a. The carbon peaks, stemming from the MWCNTs and organic layer and oxygen peaks related to surface oxides and nitro groups showed the highest intensity. The inset of Fig. 2a reveals two peaks in the N1s region pertaining to XPS spectra of nitrophenyl modified MWCNTs. The first peak with the higher binding energy around 406 eV is attributed to –NO₂ groups. The second peak with a lower binding energy around 400 eV is attributed to amines. It has been demonstrated that the second peak can come from chemical reduction of NO₂ groups under the X-ray beam during the XPS experiment [58,59]. Also, the formation of azo-linkages within the NP film has been proposed to explain the N1s peak at ca 400 eV [60]. In the next stage of experiments, the XPS analysis of AuNP/MWCNT samples was performed. XPS spectrum confirms the presence of gold nanoparticles on the surface of MWCNTs (Fig. 2b). The XPS spectrum of AuNP/MWCNTs shows the presence of oxygen (O1s) and a small peak of nitrogen residues. The peaks centered at 83.8 (Au4f7/2) and 87.5 eV (Au4f5/2) provide a typical signature of metallic Au nanoparticles.

3.3. Cyclic voltammetry (CV)

The strong attachment of nitrophenyl (NP) groups to MWCNTs was confirmed by cyclic voltammetry. Fig. 3 shows the CV response obtained at a scan rate of 100 mV s⁻¹ for a NP/MWCNT modified GC electrode in 0.1 M KOH. These experiments showed
AuNP/MWCNT modified GC and bulk gold electrodes in O2-free
ered as nucleation sites for AuNP formation.

\[ \text{Ph—NO}_2 + 2\text{H}^+ + 2\text{e}^- \rightarrow \text{Ph—N(OH)}_2^- + \text{H}_2\text{O} \rightarrow \text{Ph—NO} \] (1)

Followed by a quasi-reversible two-electron, two-proton reduc-
tion to phenylhydroxylamine:

\[ \text{Ph—NO} + 2\text{H}^+ + 2\text{e}^- \rightarrow \text{Ph—NH(OH)} \] (2)

A pair of CV peaks centered at ca. 0.4 V is attributed to the Ph–
NO/Ph—NH(OH) redox couple. The reduced NP groups are consid-
ered as nucleation sites for AuNP formation.

Cyclic voltammetric experiments were also performed with
AuNP/MWCNT modified GC and bulk gold electrodes in O2-free
0.5 M H$_2$SO$_4$. The electrochemical responses of these electrodes
are shown in Fig. 4. The CV response was recorded in the range
of potentials between 1.4 and 0.3 V. For both electrodes com-
pared typical features of gold surface oxidation on the forward
scan (at ca. 1.15 V) and its reduction on the reverse scan (at ca.
0.88 V) were observed. A clear pair of peaks appears at approxi-
mately 0.4 V for the AuNP/MWCNT catalyst (Fig. 4, curve 2), which
is assigned to carbon–oxygen functionalities on the surface of pre-
treated MWCNTs [10]. A large increase in the background current
observed for curve 2 is typical for MWCNTs because of the high
specific surface area. The charge under the gold oxide reduction
peak for the AuNP/MWCNT modified GC electrode was
4.64 \times 10^{-2} \text{ C cm}^{-2}. These results indicate that the total area
of gold nanoparticles of the hybrid material was higher than that ob-
erved in our previous investigation for the AuNP/MWCNT catalyst
in the absence of nitrophenyl groups [54]. The charge required for
the formation of an oxide monolayer [61] was taken as
400 \mu\text{C cm}^{-2} and hence, the total area of gold nanoparticles
exceeds the geometric area (A = 0.2 cm$^2$) by a factor of 12. The rough-
ness factor of the bulk gold electrode (r = A/A$_g$) was found to be 3.2.

3.4. Oxygen reduction

Electrocatalytic activity towards oxygen reduction was explored using the rotating disk electrode method. The RDE polarisa-
tion curves of oxygen reduction on a bulk gold electrode in O2
saturated 0.5 M H$_2$SO$_4$ are presented in Fig. 5. These are typical
I–E curves for O$_2$ reduction on gold in acid electrolyte. The reduc-
tion current gradually increases with decreasing potential and no
diffusion-controlled current plateau is formed prior to the poten-
tials of hydrogen evolution.

The RDE results clearly demonstrate the excellent electrocata-
lytic activity of AuNP/MWCNT modified GC electrodes for O$_2$
reduction in comparison to the polished Au electrode. Results pre-
sented in Fig. 6 show a significant positive shift of the oxygen
reduction wave and an increase in the current density. The oxygen
reduction wave starts at approximately 0.25 V.

The RDE data were analysed using the Koutecky–Levich (K–L)
equation [62]:

\[
I = \frac{1}{I_k} + \frac{1}{I_d} = \frac{1}{nFAkC_{O_2}} - \frac{0.62nFAkC_{O_2}^{1/2}v^{-1/2}D_{O_2}^{1/2}}{C_{O_2}^{1/2}}
\] (3)

where $I$ is the measured current, $I_k$ and $I_d$ are the kinetic and diffusion-
limited currents, respectively, $n$ is the number of electrons
transferred per O$_2$ molecule, $k$ is the rate constant for O$_2$ reduction,
$F$ is the Faraday constant (96,485 C mol$^{-1}$), $A$ is the geometric
electrode area, $\omega$ is the rotation rate, $C_{O_2}$ is the concentration of oxygen
in the bulk (1.13 \times 10^{-6} \text{ mol cm}^{-3} [63]), $D_{O_2}$ is the diffusion coeffi-
cient of oxygen (1.8 \times 10^{-5} \text{ cm}^2 \text{s}^{-1} [63]) and $v$ is the kinematic
viscosity of the solution (0.01 \text{ cm}^2 \text{s}^{-1} [64]).

The Koutecky–Levich plots for O$_2$ reduction on a bulk gold elec-
trode are shown in Fig. 7. The K–L lines are almost linear. The inset
to Fig. 7 shows that the value of $n$ is slightly higher than two at the
potentials of O$_2$ reduction. This indicates that the 2e$^-$ reduction is
the predominant pathway, which leads to peroxide formation in

![Figure 3](image1.png)

Fig. 3. Cyclic voltammograms for a NP/MWCNT modified GC electrode in Ar
saturated 0.1 M KOH at $v = 100 \text{ mV s}^{-1}$. The number of potential cycles is indicated
for each CV curve.

![Figure 4](image2.png)

Fig. 4. Cyclic voltammograms for polished gold (1) and AuNP/MWCNT modified GC
(2) electrodes in Ar saturated 0.5 M H$_2$SO$_4$ at $v = 100 \text{ mV s}^{-1}$.

![Figure 5](image3.png)

Fig. 5. RDE voltammetry curves for O$_2$ reduction on a polished gold electrode in O$_2$
saturated 0.5 M H$_2$SO$_4$ at $v = 10 \text{ mV s}^{-1}$.
the range of potentials studied. This is in agreement with previous observations [12,16].

Fig. 8 shows the K–L plots obtained from the RDE data presented in Fig. 6. Linear K–L plots are observed and from the slope of these lines the number of electrons transferred per O2 molecule was calculated. The values of \( n \) at different potentials (shown in the inset to Fig. 8) indicate that the reduction of O2 produces H2O2, which reduces further at more negative potentials. It was previously observed, that the number of electrons involved in the AuNP catalysed O2 reduction depends on the surface structure of the gold catalysts [34]. The higher value of \( n \) of the AuNP/MWCNT catalysts as compared to that of bulk Au at high negative potentials could be, at least in part, related to their higher surface area. Gopalan et al. observed a 4e\(^-\)/C0 reduction of oxygen on poly(aminothiophenol)-Au nano-modified GC electrode [19]. They proposed that 4-electron pathway for this reaction was presumably due to the presence of PATP as a modifier for Au nanoparticles.

Indeed, the AuNP/MWCNT modified GC is a more active catalyst for O2 reduction than the bulk Au electrode. The value of half-wave potential \( (E_{1/2}) \) for O2 reduction on AuNP/MWCNT modified GC electrodes is shifted by 80 mV to more positive potentials as compared to that of the bulk gold electrode.

Fig. 9 shows the Tafel behaviour of oxygen reduction on both electrodes studied. In the region of low overpotentials the Tafel slope values were –127 and –109 mV dec\(^{-1}\) for the AuNP/MWCNT modified GC electrode and bulk Au electrode, respectively. This slope corresponds to the transfer of the first electron to the O2 molecule as the rate-limiting step:

\[
\text{O}_2 + \text{H}^+ + \text{e}^- \rightarrow \text{HO}_2
\]

These results are in good agreement with previous work, in which the kinetic parameters of oxygen reduction on thin-film Au electrodes of various thickness were determined [17]. The Tafel slope close to 120 mV dec\(^{-1}\) was also observed for a bulk Au electrode in acid media in earlier reports [12,16].

The specific activity of the AuNP/MWCNT modified electrodes was calculated using the following equation:

\[
SA = \frac{I_k}{A_i}
\]

where \( I_k \) is the kinetic current at a given potential and \( A_i \) is the real surface area of gold. The real area of gold was determined from the stable cyclic voltammograms by charge integration under the oxide reduction peak and using a value of 400 \( \mu \text{C cm}^2 \) for the reduction of an oxide monolayer [61]. The values of specific activity were determined at 0.1 V from the Tafel plots (Fig. 9) taking into account the real surface area for each electrode. In agreement with Sarapuu et al. [17] results the difference of specific activity for O2 reduction on AuNP/MWCNT modified GC and bulk Au electrodes is not remarkable (0.057 mA cm\(^{-2}\) for AuNP/MWCNT modified GC and 0.042 mA cm\(^{-2}\) for bulk gold electrodes). The Tafel behaviour was
essentially the same for composite materials and bulk gold electrodes. Nevertheless, the catalytic properties of Au nanoparticles were still different from those of the bulk metal, the higher reduction current and shifting the half-wave potential 80 mV to more positive value compared to bulk gold. The AuNPs/MWCNT hybrid is a promising material for various applications, including electrocatalysis and electroanalysis.

Finally, it is necessary to stress that the effect of Au particle size on the kinetics of O2 reduction on AuNP-modified electrodes in acid solution is a matter of controversy. The most important kinetic parameter, which has been used for the evaluation of the electrocatalytic activity of nanostructured catalysts for oxygen reduction is the value of SA. Our previous results have shown that the SA of vacuum-evaporated thin Au films is very close to that of bulk Au [17,37]. Guerin et al. observed that the specific activity of O2 reduction was independent of particle size for AuNPs larger than 3 nm [18]. Inasaki and Kobayashi found a decrease in the SA value with decreasing AuNP size [32]. They used Au catalysts supported on high-area carbon with the mean diameter of 1.7, 4.8 and 13.2 nm. In a similar study Bron reported that the surface specific activity was independent of particle size in the range 2.7–42.3 nm [33].

By contrast, Ohsaka and co-workers reported an extraordinary electrocatalytic activity of relatively large AuNPs (20–200 nm) electrodeposited on a gold substrate [20,21]. The increased O2 reduction activity has also been observed on AuNPs deposited onto boron-doped diamond substrates [29,30]. Yagi et al. [31] have studied smaller Au particles (>2 nm), which have displayed a positive shift of the O2 reduction potential and increased current efficiency for the four-electron reduction. However, the specific activity of O2 reduction for AuNP catalysts has not been determined in these works [20,21,29–31] and therefore one cannot evaluate the intrinsic electrocatalytic activity of the nanostructured gold catalysts studied. Despite the numerous research efforts during the last decade, the physico-chemical origin of the Au particle size effect on the rate of oxygen reduction in acid media is still under debate.

A thorough kinetics investigation of oxygen reduction on well-defined AuNPs over a wide size range (1–20 nm) is needed in order to elucidate this important aspect of electrocatalysis of nanoparticulate gold. Recently, an attempt has been made to calculate the theoretically size effect of gold catalysts on the kinetics of O2 reduction [65]. An increase in activity with decreasing Au particle size is predicted, even though the results obtained are rather preliminary.

4. Conclusions

The reduction of oxygen has been studied on gold nanoparticle/multi-walled carbon nanotube modified GC electrodes in 0.5 M H2SO4 using the rotating disk electrode method. The AuNPs/MWCNT catalysts were prepared by chemical deposition of AuNPs onto MWCNTs spontaneously grafted with 4-nitrophenyl groups. The AuNPs/MWCNT catalyst showed a pronounced electrocatalytic activity towards O2 reduction in acid media. The half-wave potential of O2 reduction on the AuNPs/MWCNT catalyst shifted ca 80 mV to more positive potentials as compared to that of bulk gold. These results clearly demonstrated that MWCNTs can be used as support for a three dimensional electrocatalytic layer containing a dispersed metal that specifically catalyses the reduction of oxygen to hydrogen peroxide.

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