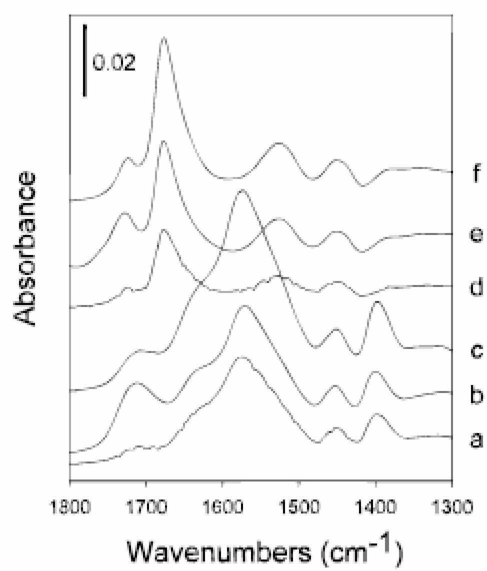
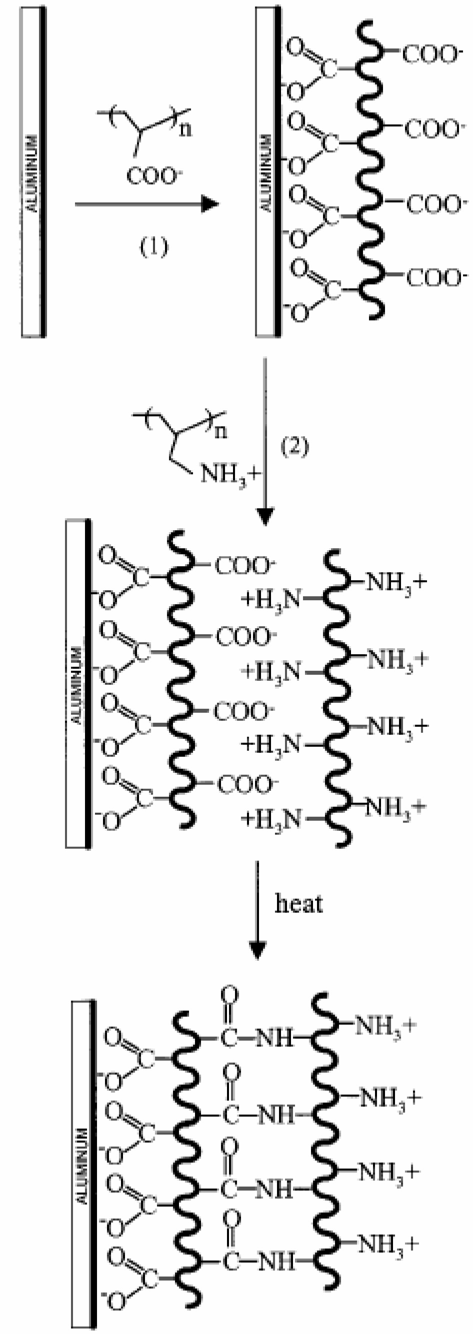
APPLICATIONS

3528 *Ind. Eng. Chem. Res.* 2000, *39*, 3528−3535

Ultrathin, Layered Polyamide and Polyimide Coatings on Aluminum

Jinhua Dai, Daniel M. Sullivan, and Merlin L. Bruening*



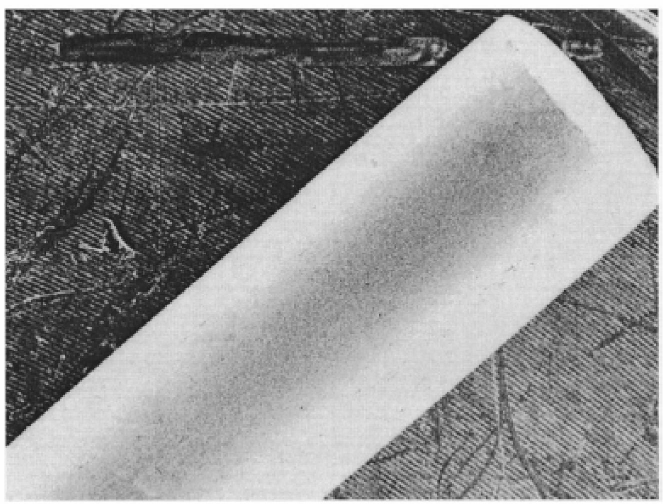


Electrochemical and Solid-State Letters, **5** (4) B13-B15 (2002)
0013-4651/2002/5(4)/B13/3/$7.00

Corrosion Control Using Polyelectrolyte Multilayers

Tarek R. Farhat and Joseph B. Schlenoff[*,z]

Department of Chemistry and Center for Materials Research and Technology, The Florida State University, Tallahassee, Florida 32306-4390, USA



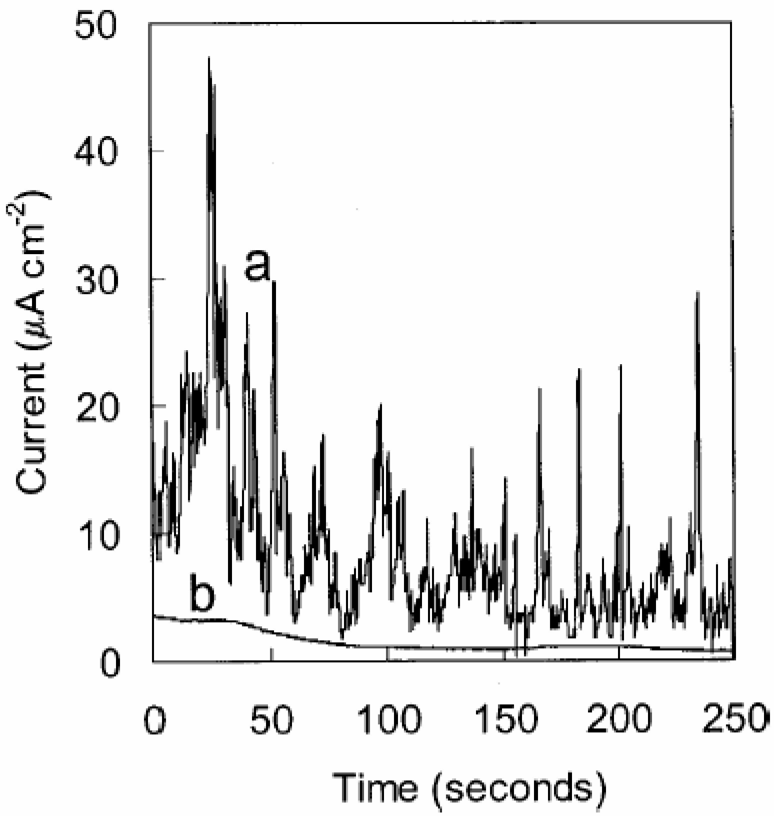


Figure 2. Current *vs.* time in the metastable pitting region (0.6 V *vs.* SCE) in 0.6 M NaCl for (a) a bare steel wire and (b) wire coated with 70 nm PDADMA/PSS.

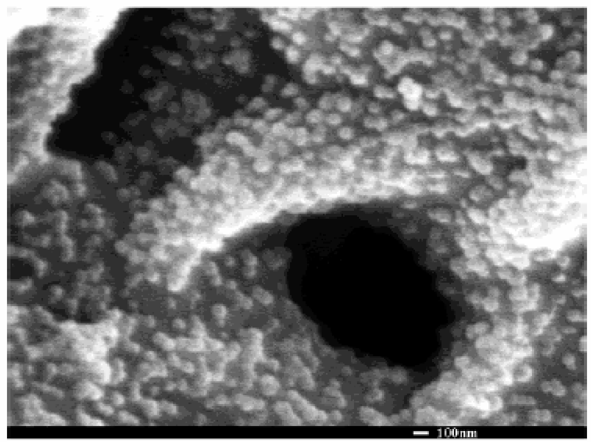
NANO LETTERS

2004
Vol. 4, No. 7
1349−1353

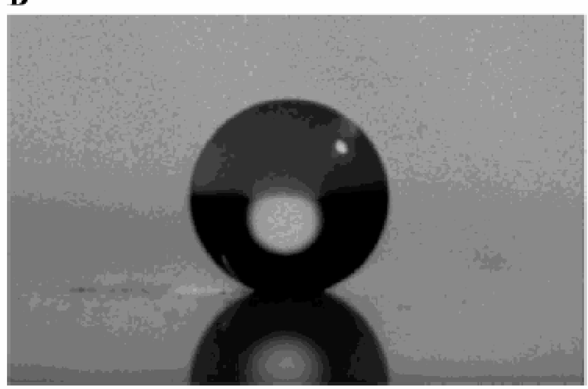
Stable Superhydrophobic Coatings from Polyelectrolyte Multilayers

Lei Zhai,[‡] Fevzi Ç. Cebeci,[‡,§] Robert E. Cohen,[*,†] and Michael F. Rubner[*,‡]

A



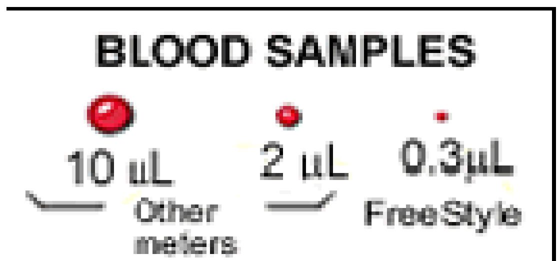
B

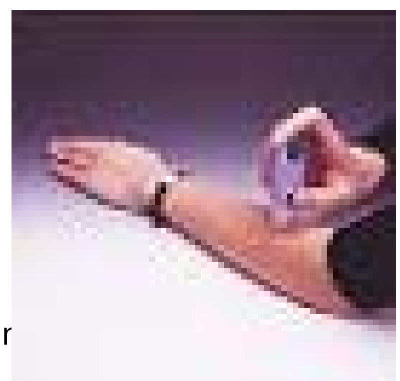


Biosensors

Molecular Recognition and
Generation of Analytical Signal

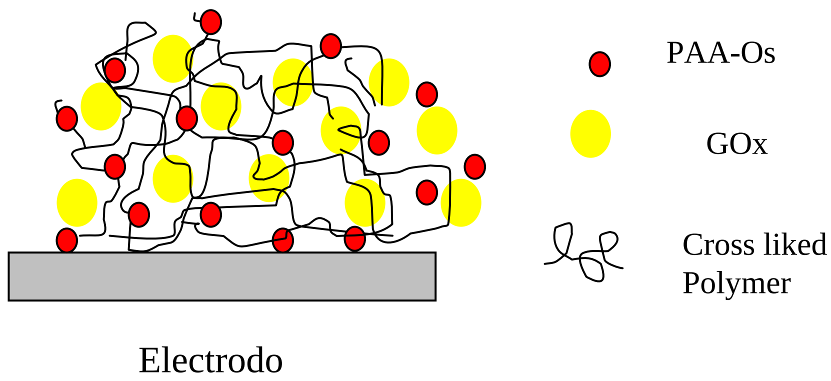
Enzyme Biosensors



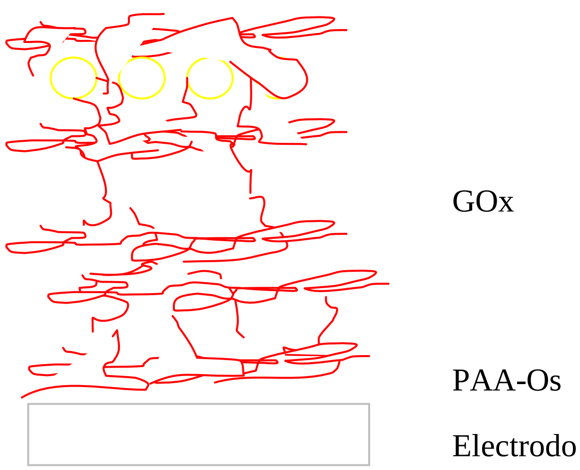


- Automatically shuts off 2 mir

From hydrogels to organized multilayer films

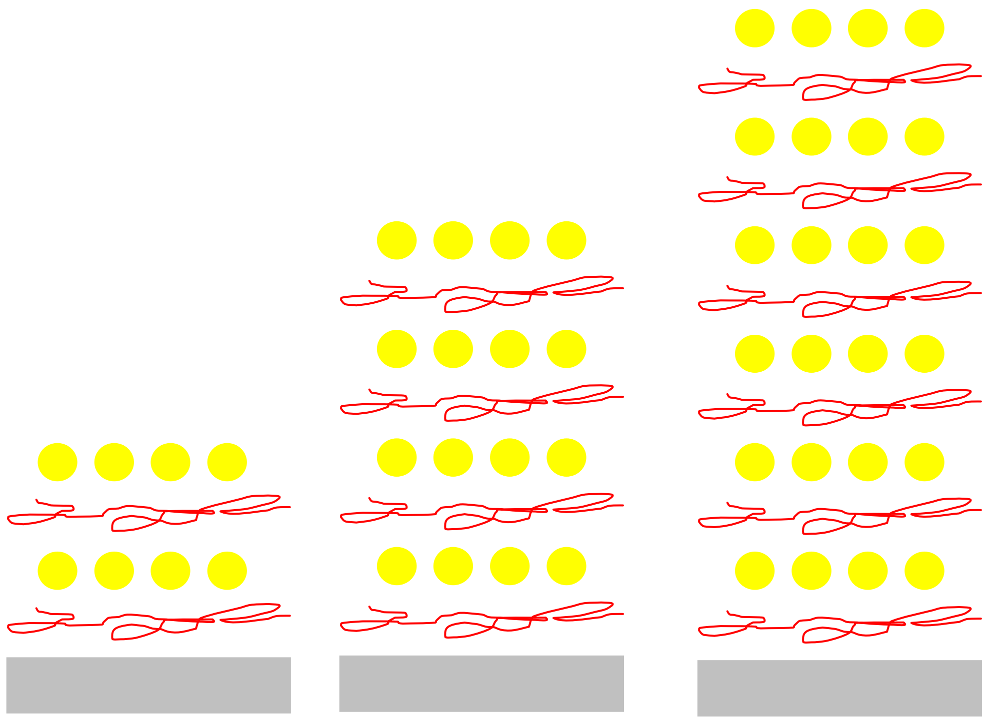


Hidrogel Redox with no control of molecular architecture.



LbL Self-Assembled enzyme multilayer organized in the nanoscale

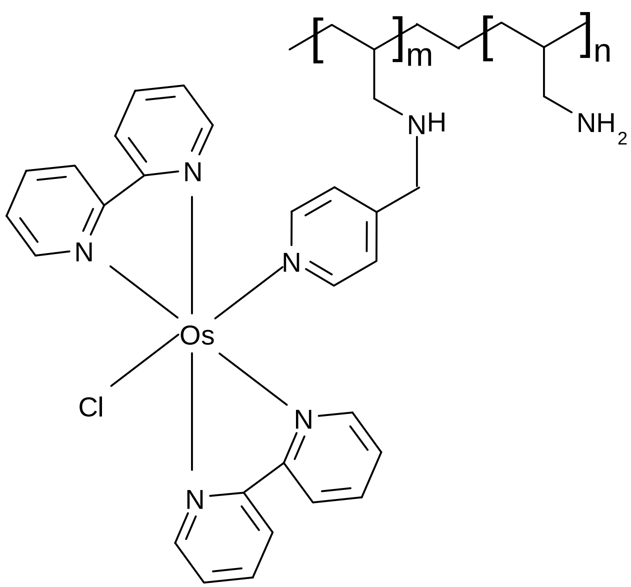
We can build up organized molecular multilayers with control of the enzyme and molecular wire spatial distribution



Two-component self-contained wired enzyme biosensor

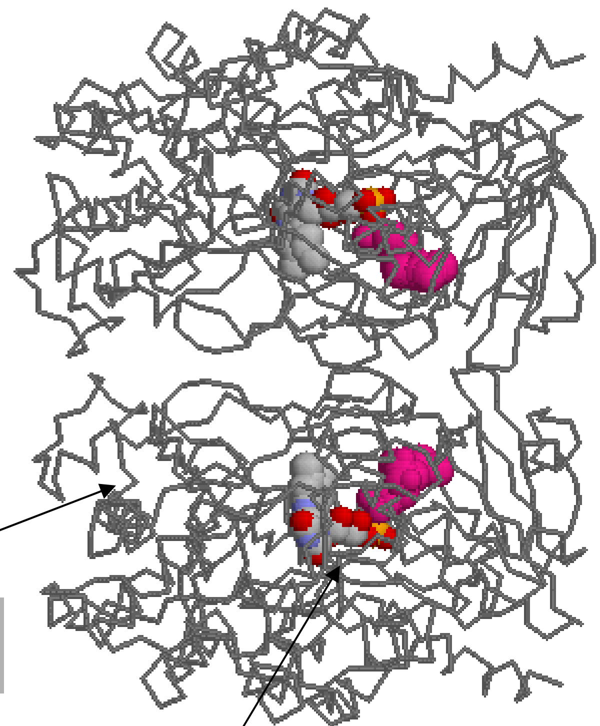
PAH-Os Redox Polymer Wire

PAH-Os (+)



PAH-Os (+)

GLUCOSE OXIDASE

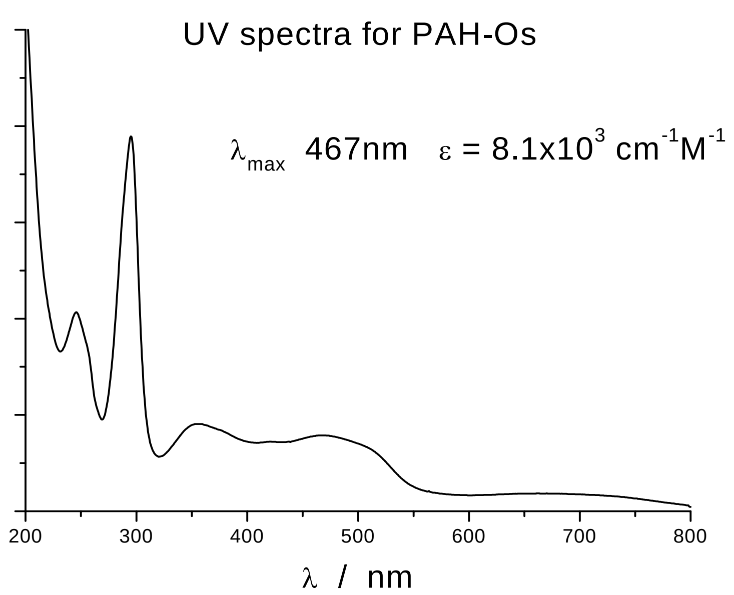


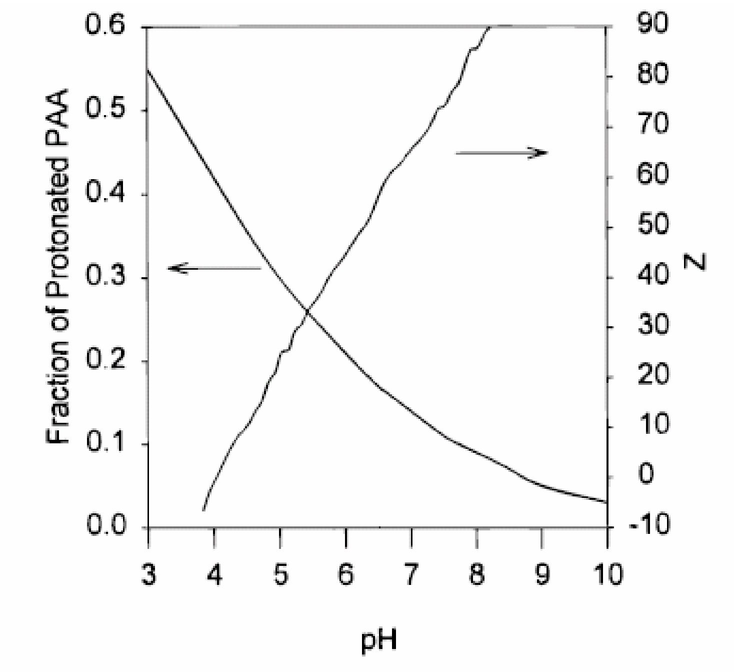
2 subunits

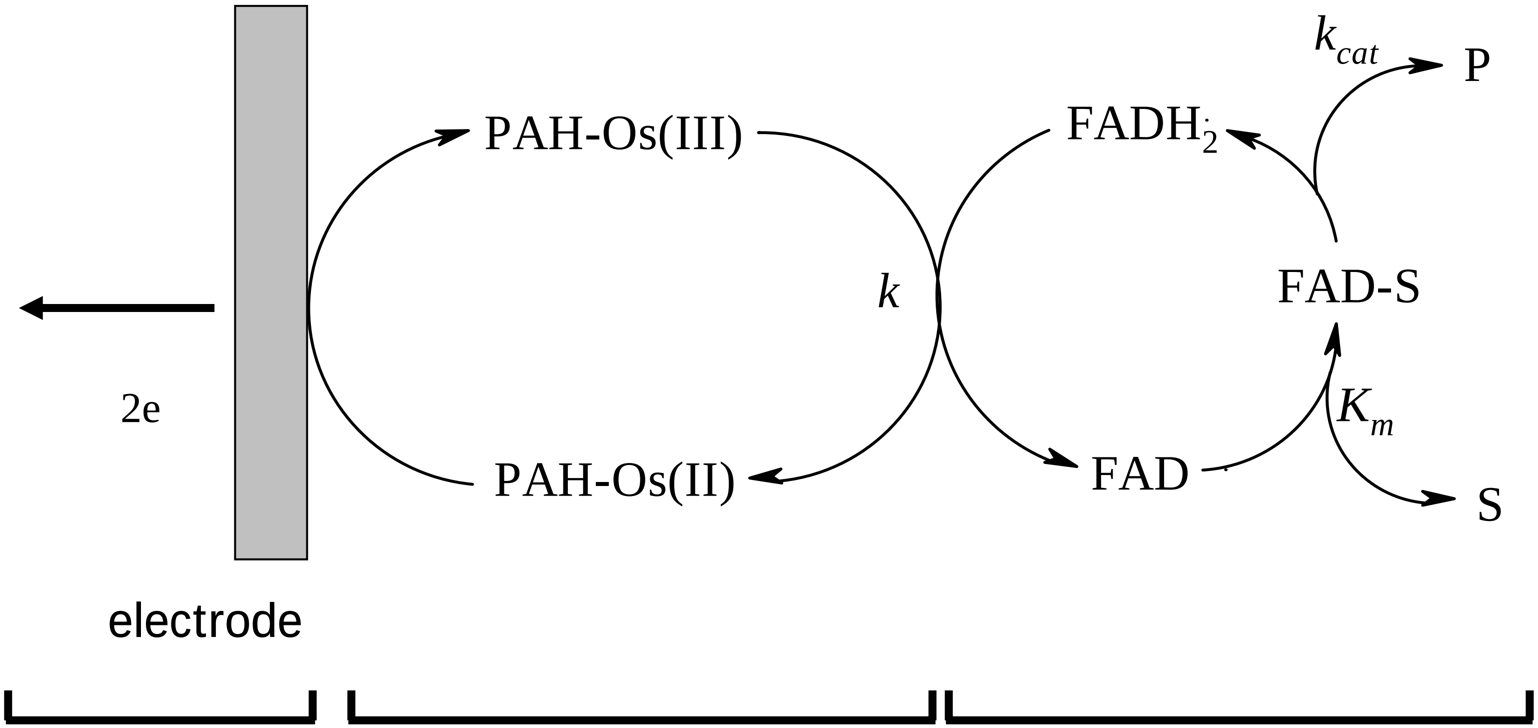
Glucose

FAD

Amperometric Enzyme Biosensor with two components integrated and electrically wired







Signal Generation

Redox Mediation

Molecular Recognition

2708 *Langmuir* **1997**, *13*, 2708−2716

Layer-by-Layer Self-Assembly of Glucose Oxidase with a Poly(allylamine)ferrocene Redox Mediator

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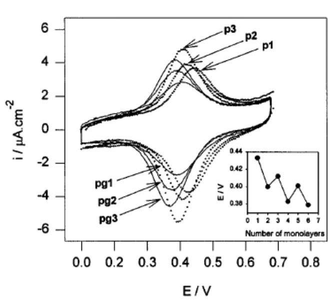
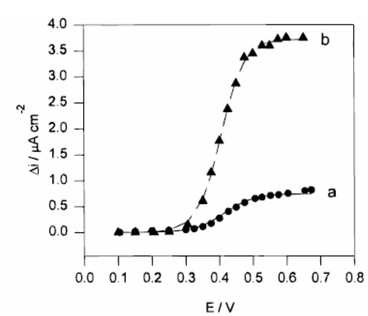
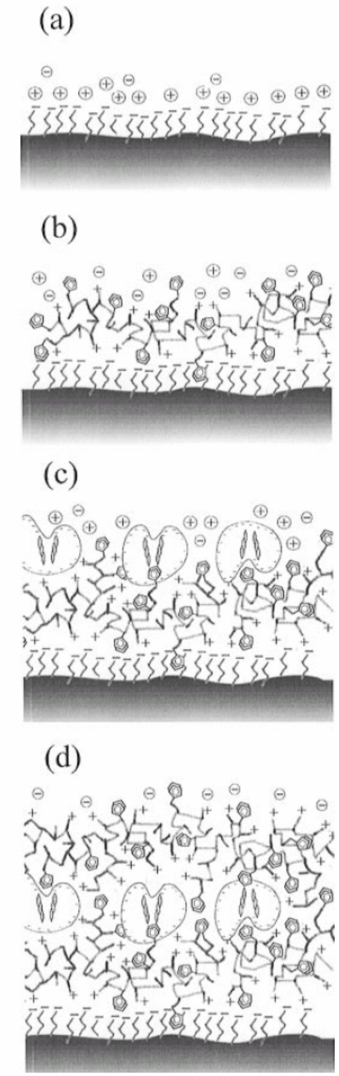
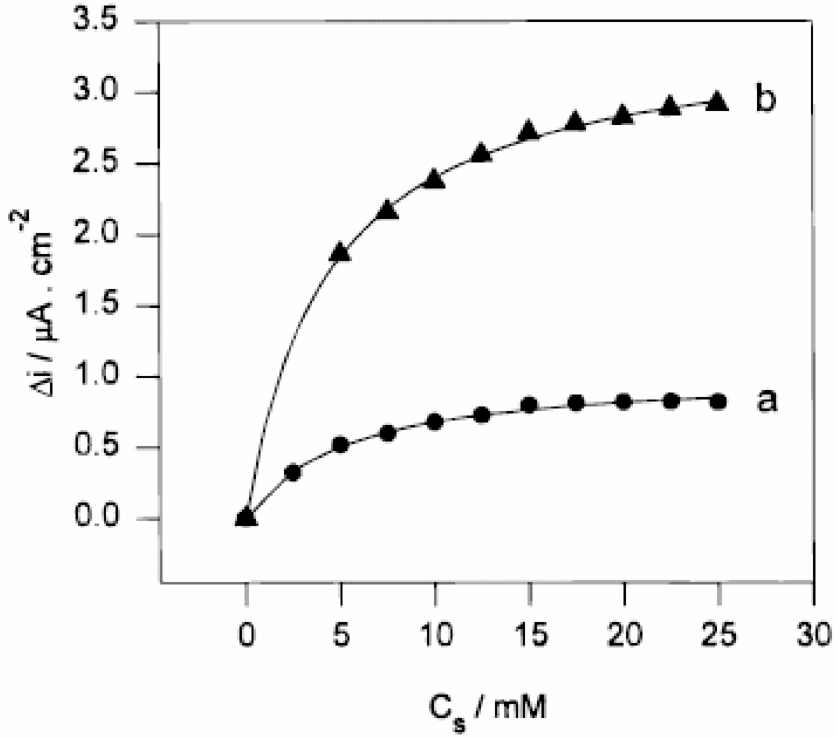
Received November 18, 1996. In Final Form: February 27, 1997®

We report the redox mediation of glucose oxidase (GOx) in a self-assembled structure of cationic poly(allylamine) modified by ferrocene (PAA-Fc) and anionic GOx deposited electrostatically layer-by-layer on negatively charged alkanethiol-modified Au surfaces. Successive PAA-Fc and GOx layers were deposited by alternate immersion of the thiol-modified Au in the respective polyelectrolyte and enzyme solutions. The uptake of thiol, redox polymer, and GOx on the surface was monitored by quartz crystal microbalance. Cyclic voltammetry shows nearly ideal surface waves of ferrocene in the polymer with charge independent of sweep rate; the redox surface concentration was obtained from integration of the ferrocene/ferricinium voltammetric peaks. The redox charge increases in step with the number of PAA-Fc layers deposited. Enzyme catalysis for the oxidation of β-D-glucose was achieved with a multilayer PAA-Fc/GOx assembly, with each GOx layer contributing equally to the catalytic response. Only a small fraction of the active assembled GOx molecules are "electrically wired" by the ferrocene polymer although all of the enzyme could be oxidized by soluble ferrocenesulfonate when added to solution.







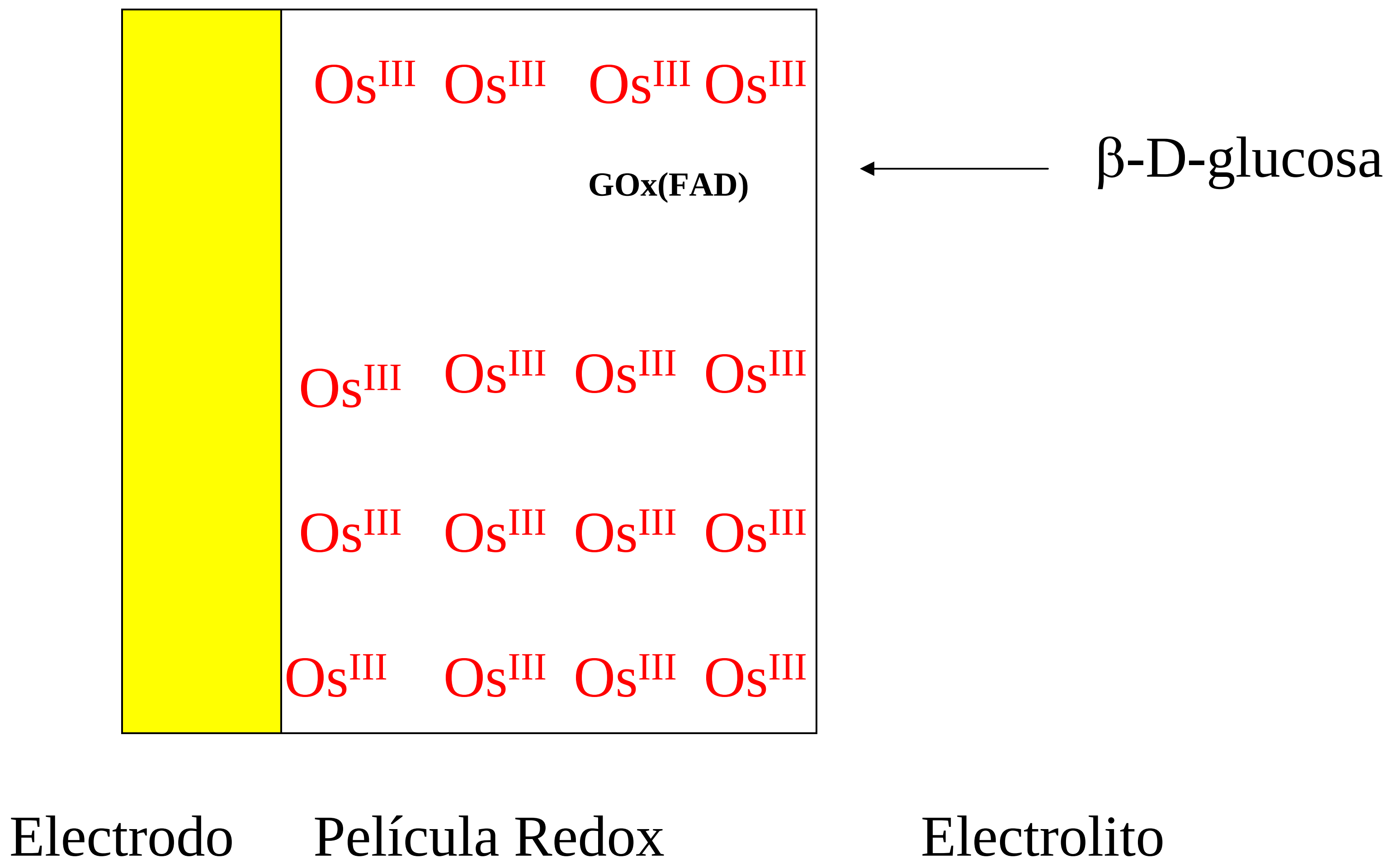


$$\text{PAA-Fc} \rightarrow \text{PAA-Fc}^{+} + e \qquad (3)$$

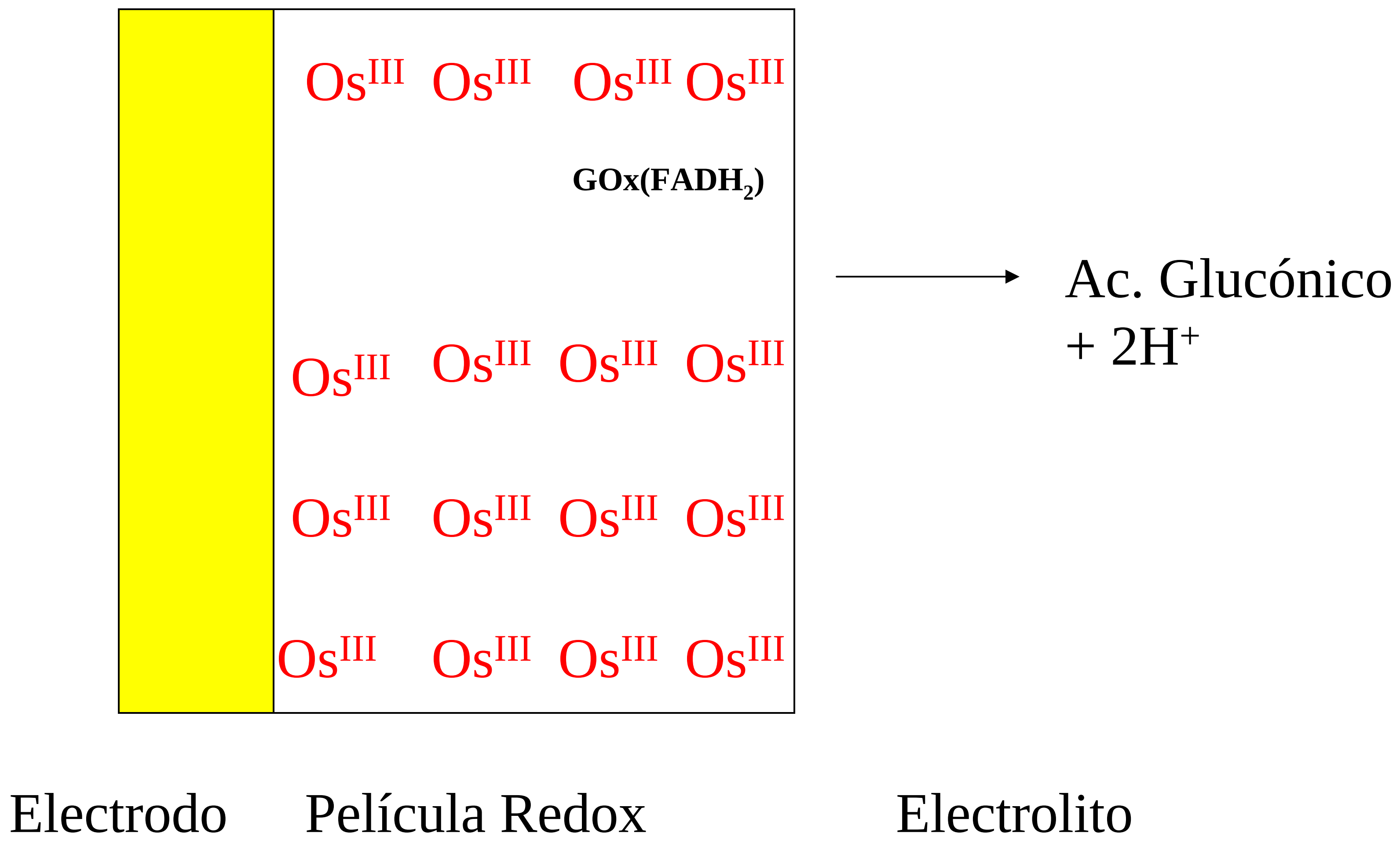
$$2\text{PAA-Fc}^{+} + \text{GOx(FADH}_2) \xrightarrow{k} \text{GOx(FAD)} + 2\text{PAA-Fc} \qquad (4)$$

$$\text{GOx(FAD)} + \text{S} \overset{K_m}{\longleftrightarrow} \text{GOx(FAD-S)} \xrightarrow{k_{cat}} \text{GOx(FADH}_2) + \text{P} \qquad (5)$$

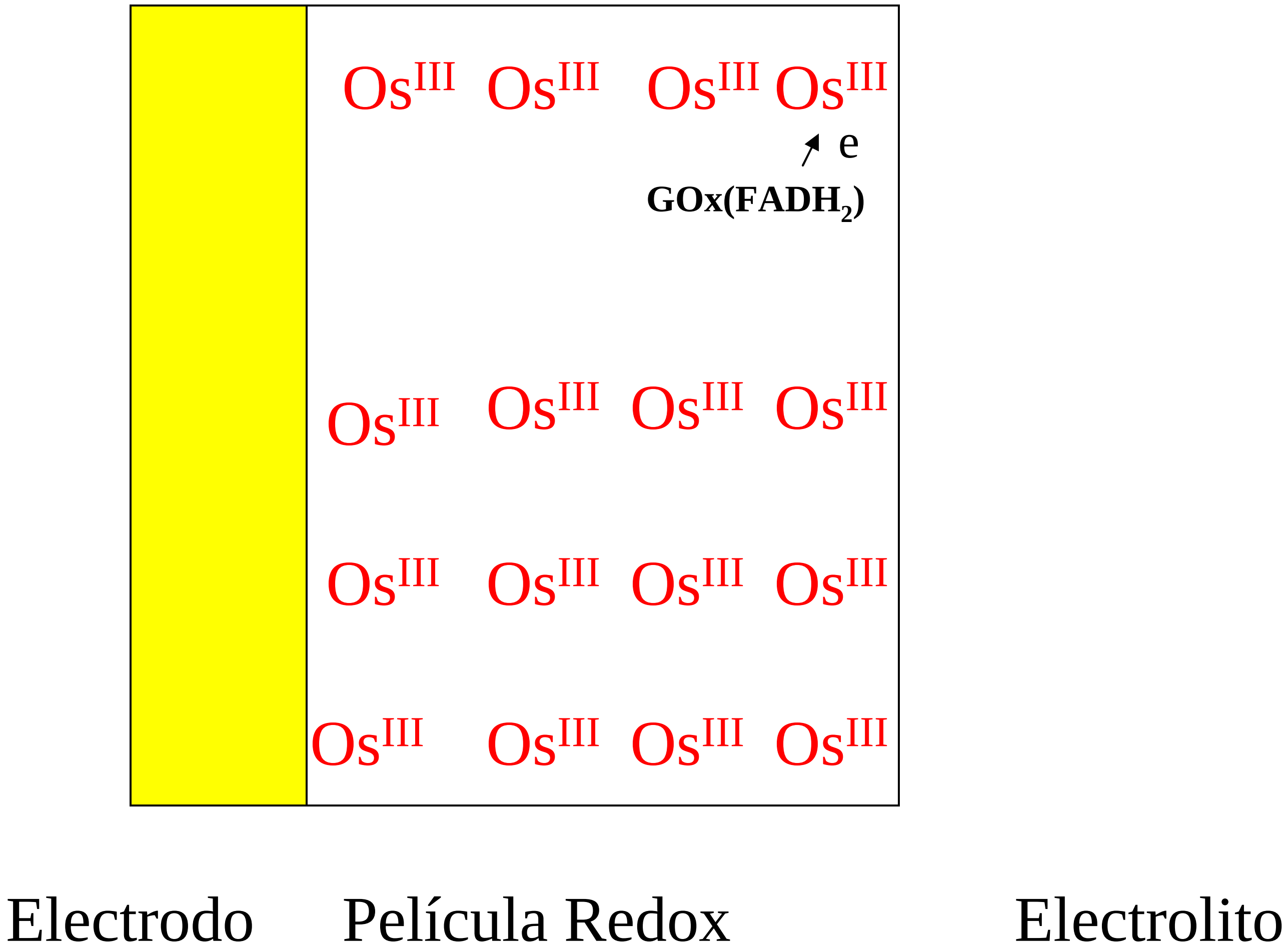
$$I_{cat} = \frac{2F\, k_{cat}\Gamma_{ET}}{1 + \dfrac{k_{cat}}{k[\text{Fc}^{+}]} + \dfrac{K_m}{[\text{S}]}} \qquad (6)$$



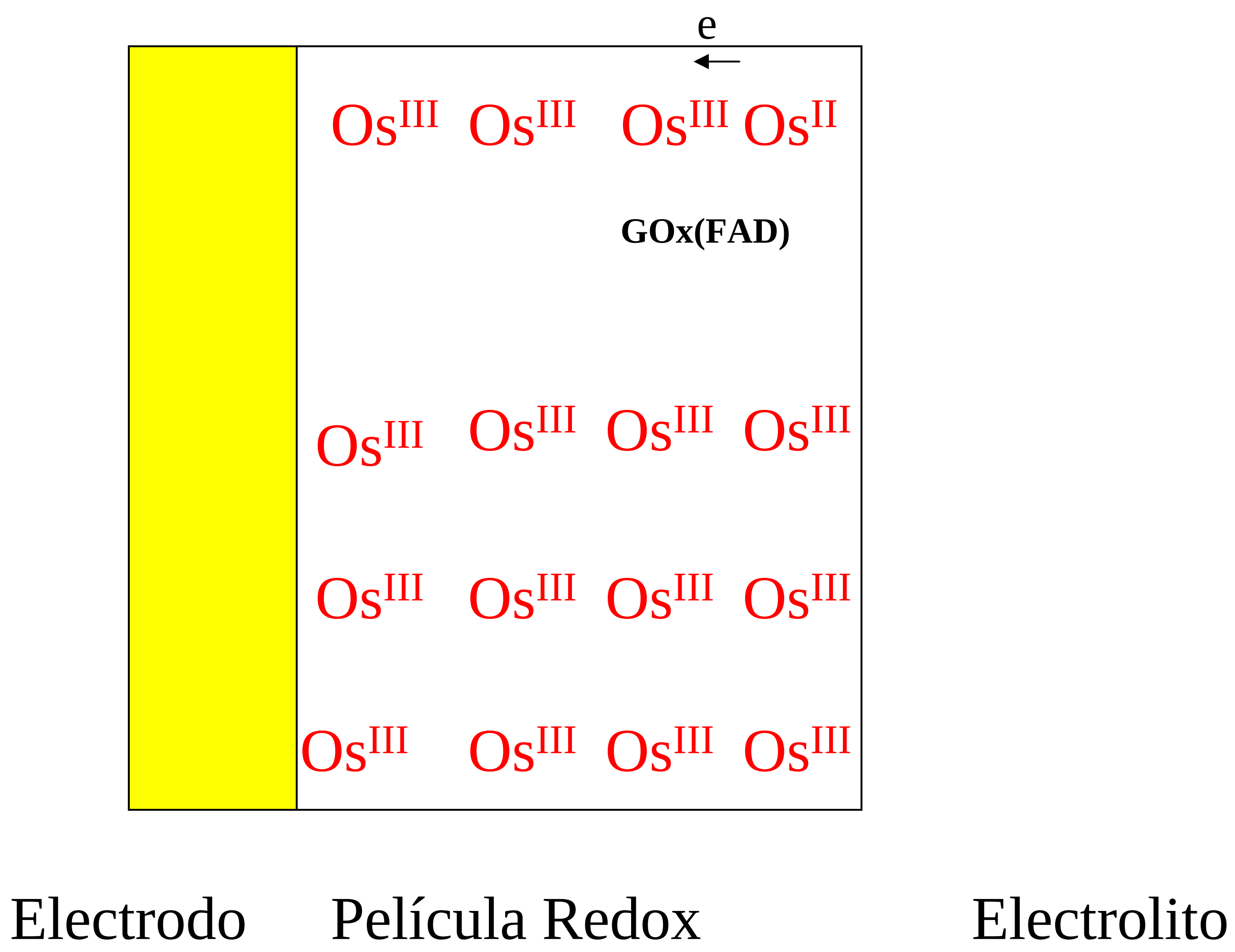
Os^{III} Os^{III} Os^{III} Os^{III}
GOx(FAD)
β-D-glucosa
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Electrodo
Película Redox
Electrolito

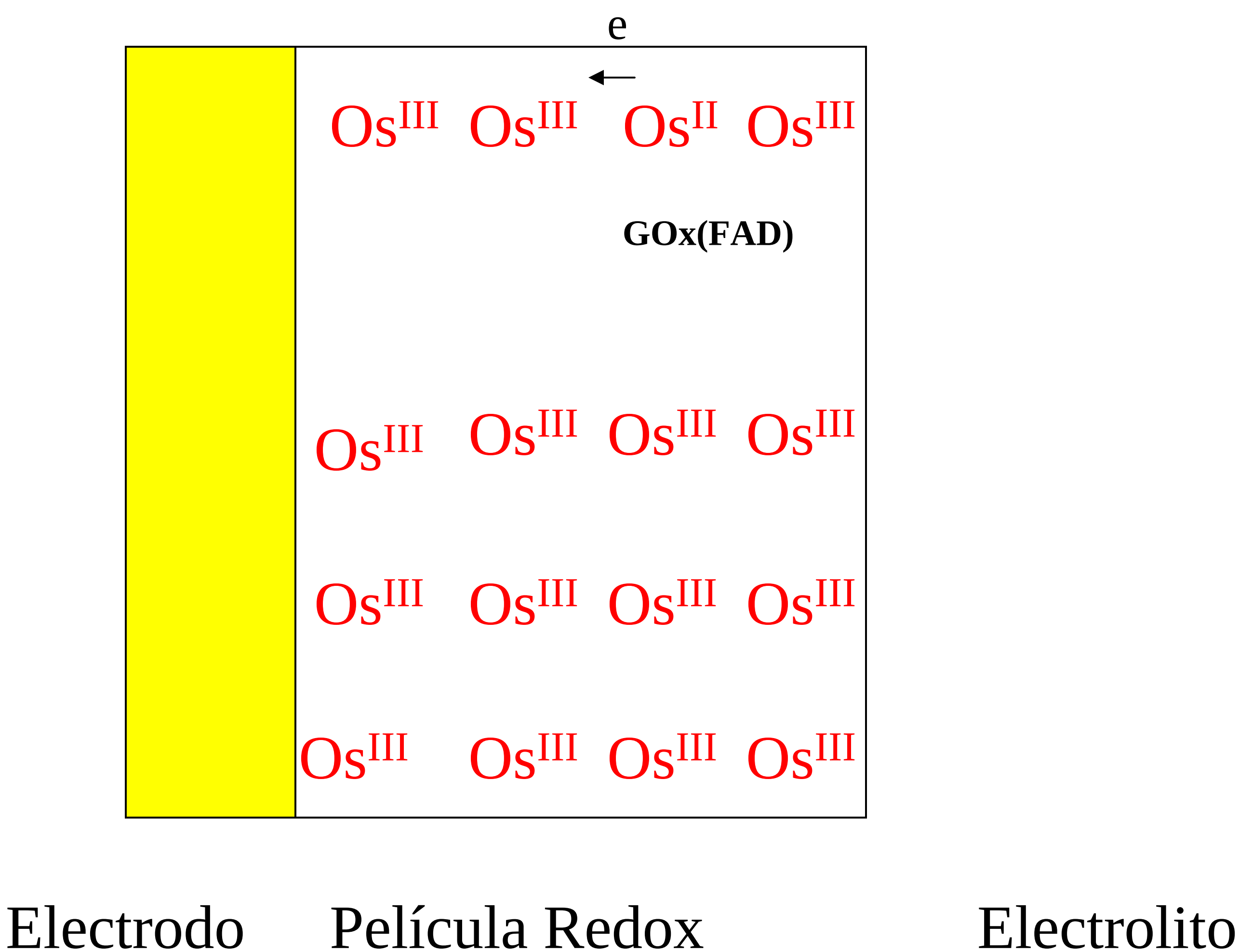


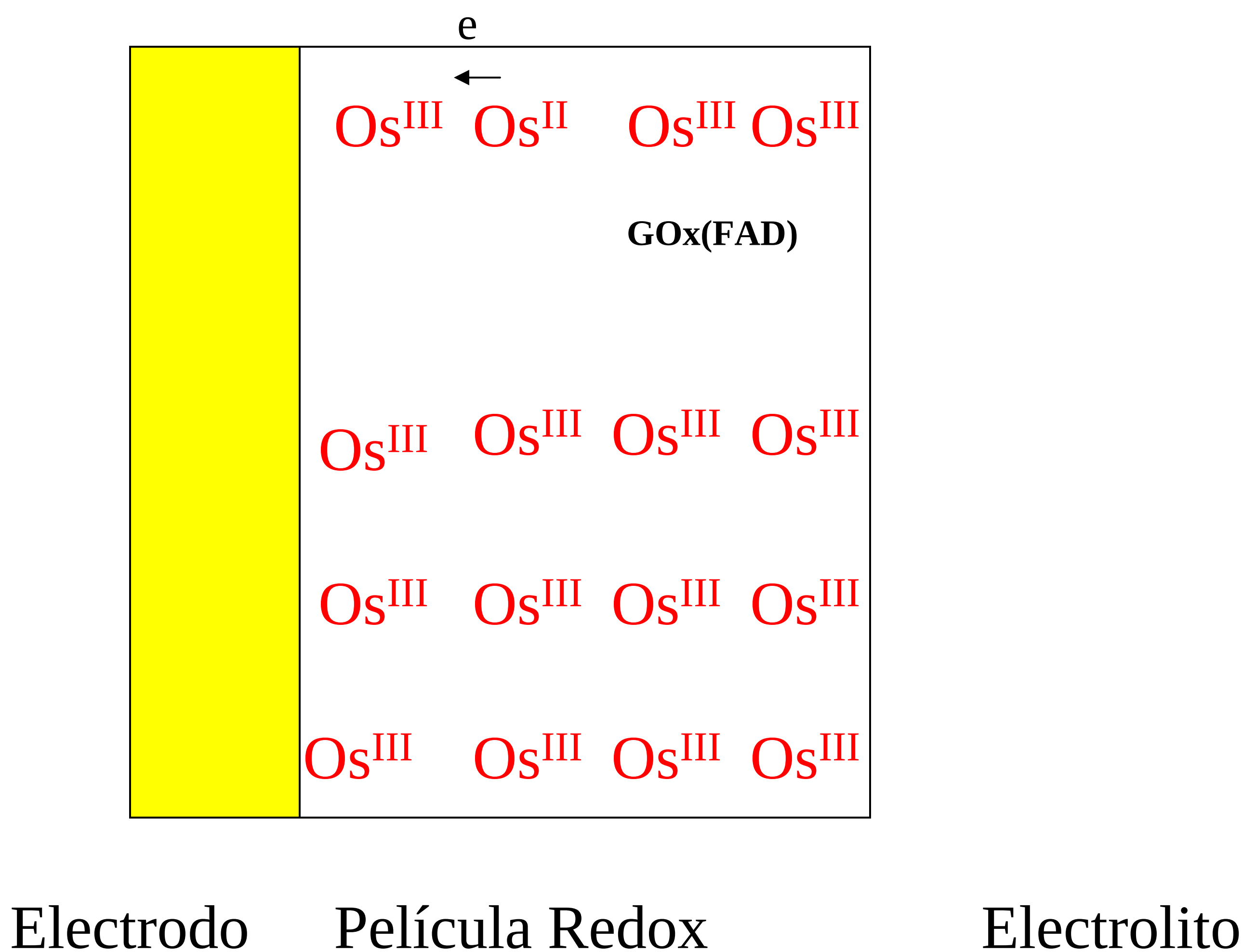
Os^{III} Os^{III} Os^{III} Os^{III}
GOx($FADH_2$)
Ac. Glucónico
+ $2H^+$
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Electrodo
Película Redox
Electrolito

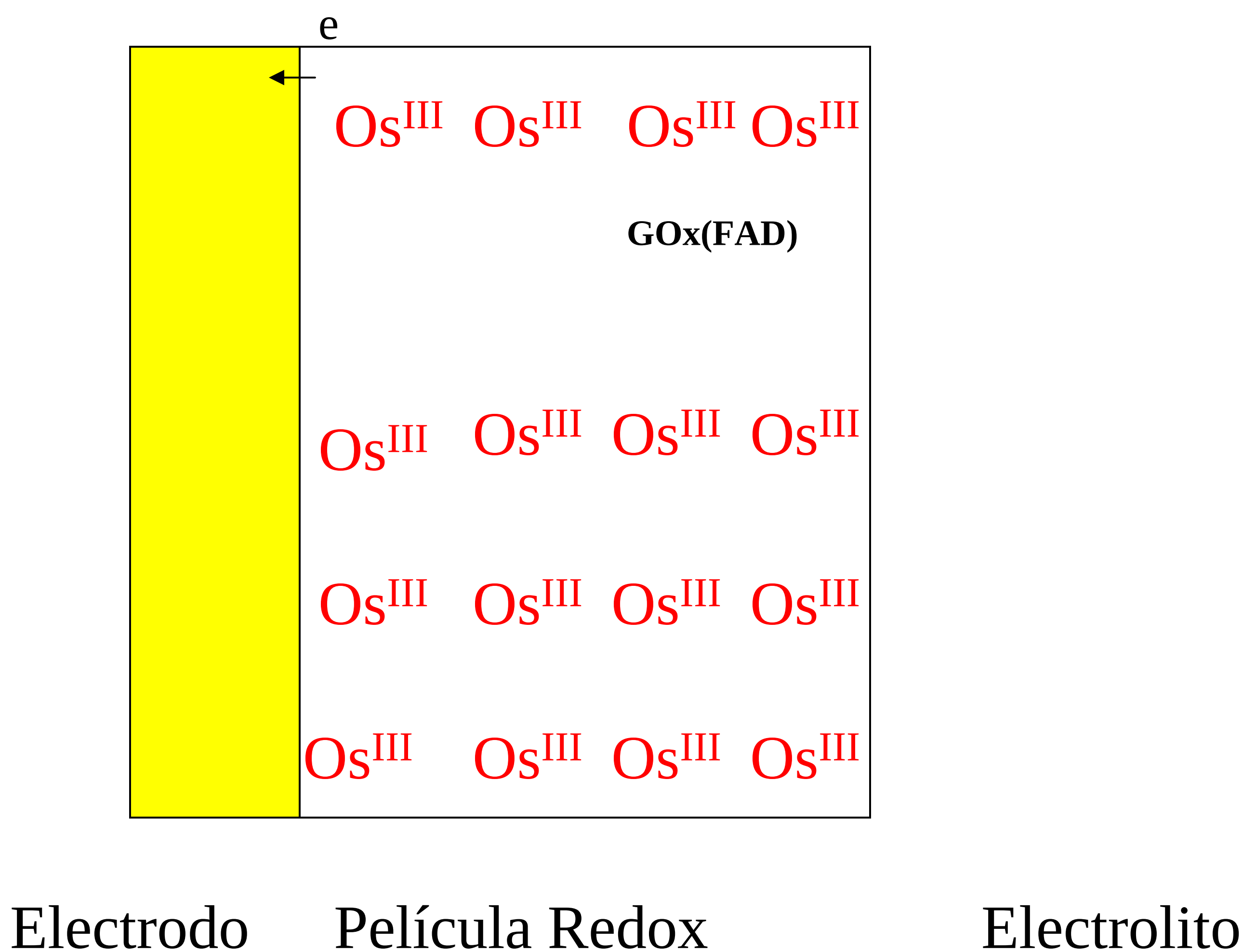


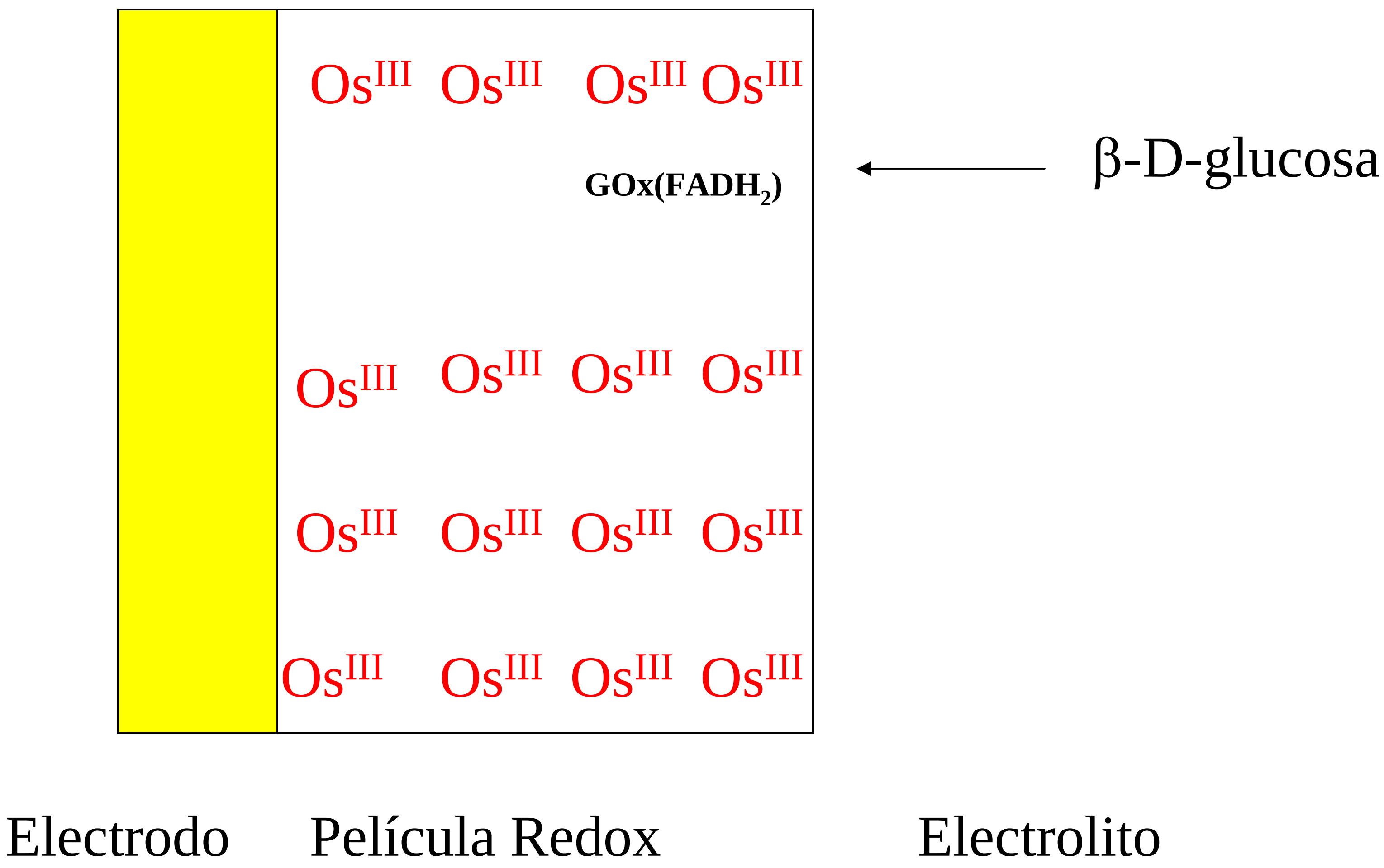
Os^III Os^III Os^III Os^III
e
GOx(FADH2)
Os^III Os^III Os^III Os^III
Os^III Os^III Os^III Os^III
Os^III Os^III Os^III Os^III
Electrodo
Película Redox
Electrolito


e
Os^{III} Os^{III} Os^{III} Os^{II}
GOx(FAD)
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Electrodo
Película Redox
Electrolito


e
Os^III Os^III Os^II Os^III
GOx(FAD)
Os^III Os^III Os^III Os^III
Os^III Os^III Os^III Os^III
Os^III Os^III Os^III Os^III
Electrodo
Película Redox
Electrolito


e
Os^{III} Os^{II} Os^{III} Os^{III}
GOx(FAD)
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Electrodo
Película Redox
Electrolito

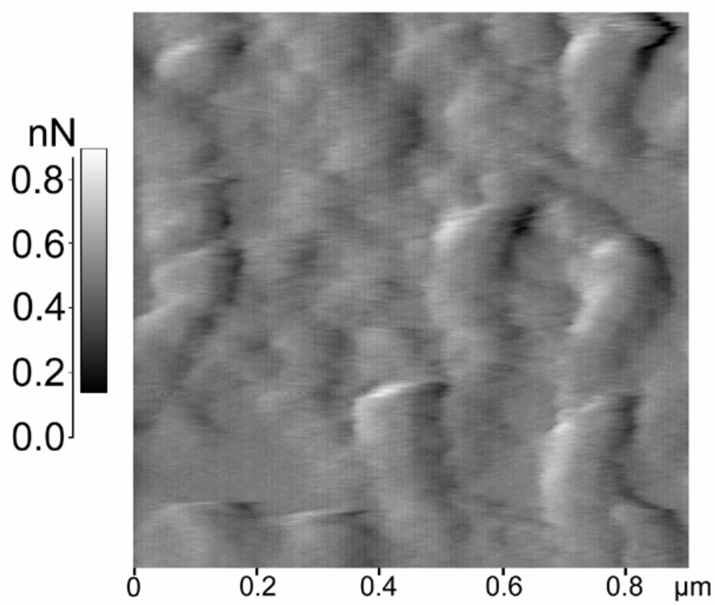

e
Os^{III} Os^{III} Os^{III} Os^{III}
GOx(FAD)
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Electrodo
Película Redox
Electrolito



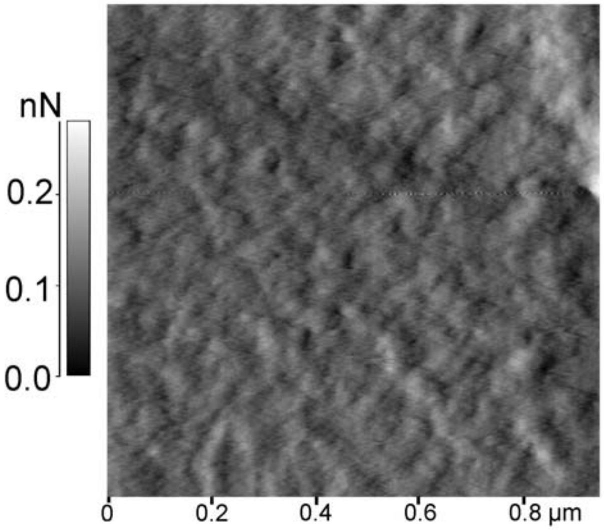
Os^{III} Os^{III} Os^{III} Os^{III}
β-D-glucosa
GOx($FADH_2$)
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Os^{III} Os^{III} Os^{III} Os^{III}
Electrodo
Película Redox
Electrolito

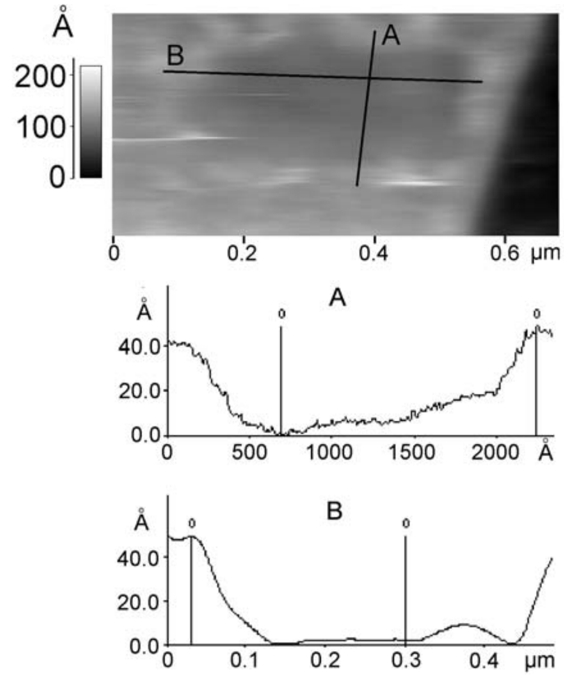
The redox charge increases in step with the number of PAA-Fc layers deposited. Enzyme catalysis for the oxidation of β-D-glucose was achieved with a multilayer PAA-Fc/GOx assembly, with each GOx layer contributing equally to the catalytic response. Only a small fraction of the active assembled GOx molecules are "electrically wired" by the ferrocene polymer although all of the enzyme could be oxidized by soluble ferrocenesulfonate when added to solution.

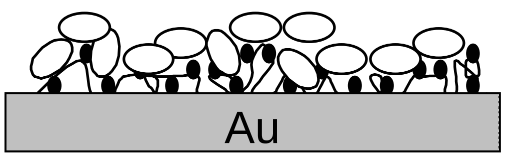
ATOMIC FORCE MICROSCOPY



Nanodosing by AFM

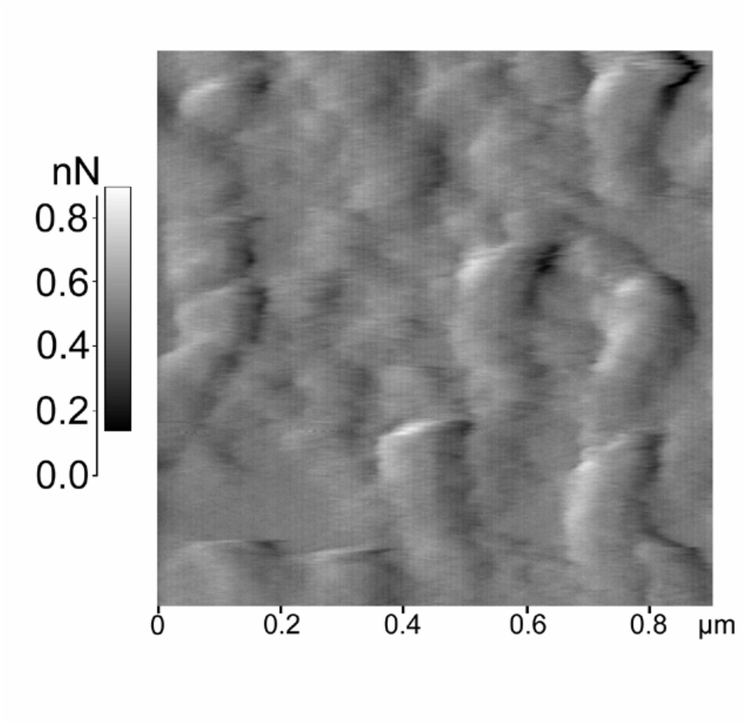


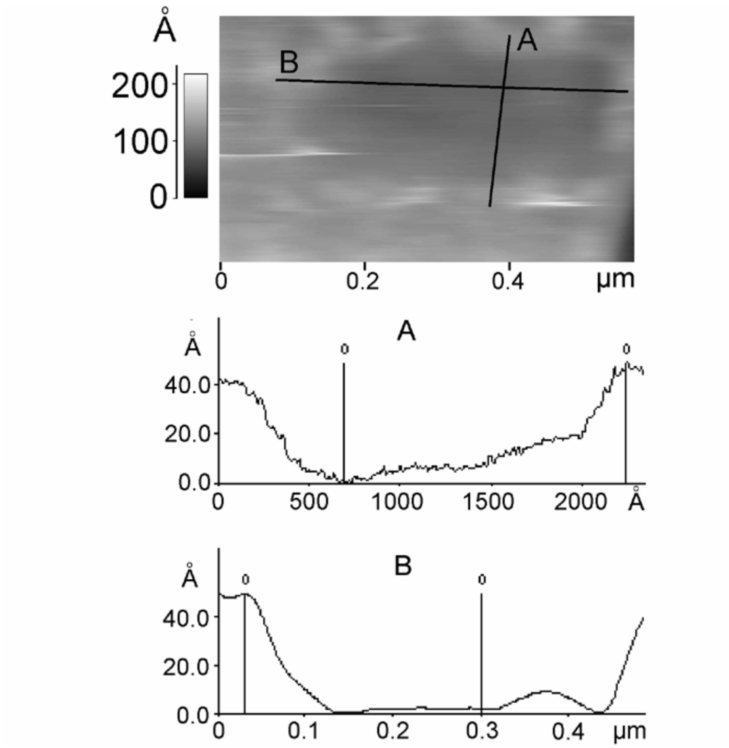


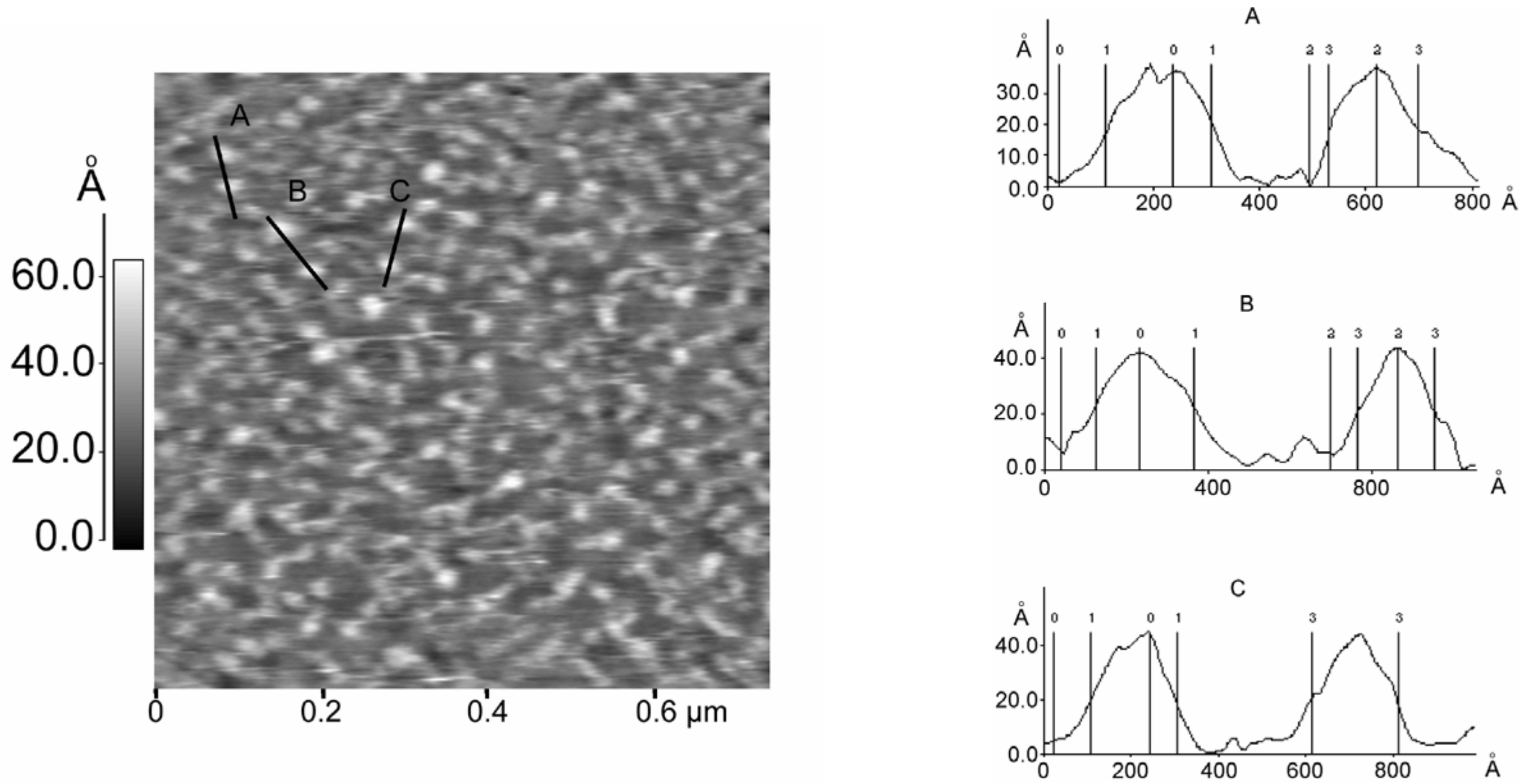


2D aggregation of the enzyme molecules is observed on the surface

ATOMIC FORCE MICROSCOPY







Å
60.0
40.0
20.0
0.0
A
B
C
0
0.2
0.4
0.6 µm
A
Å
30.0
20.0
10.0
0.0
0
200
400
600
800
Å
B
Å
40.0
20.0
0.0
0
400
800
Å
C
Å
40.0
20.0
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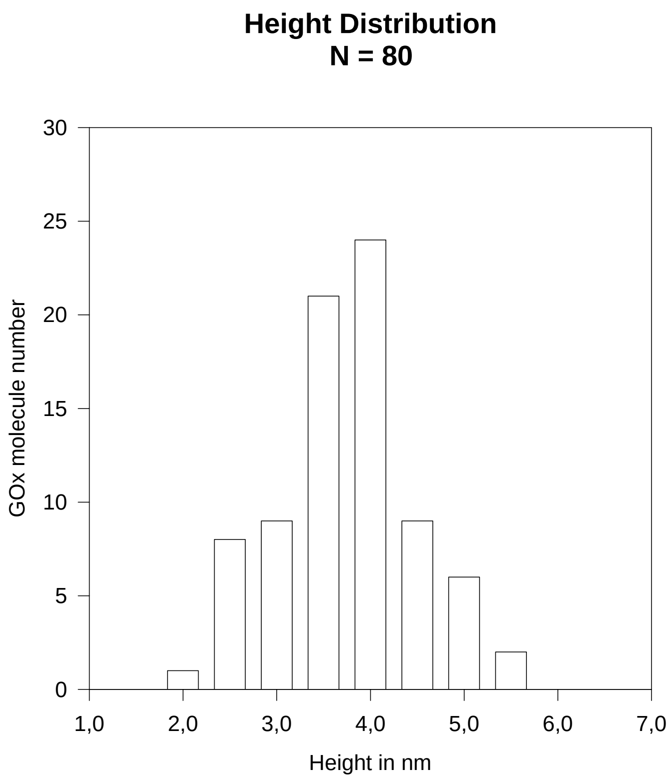
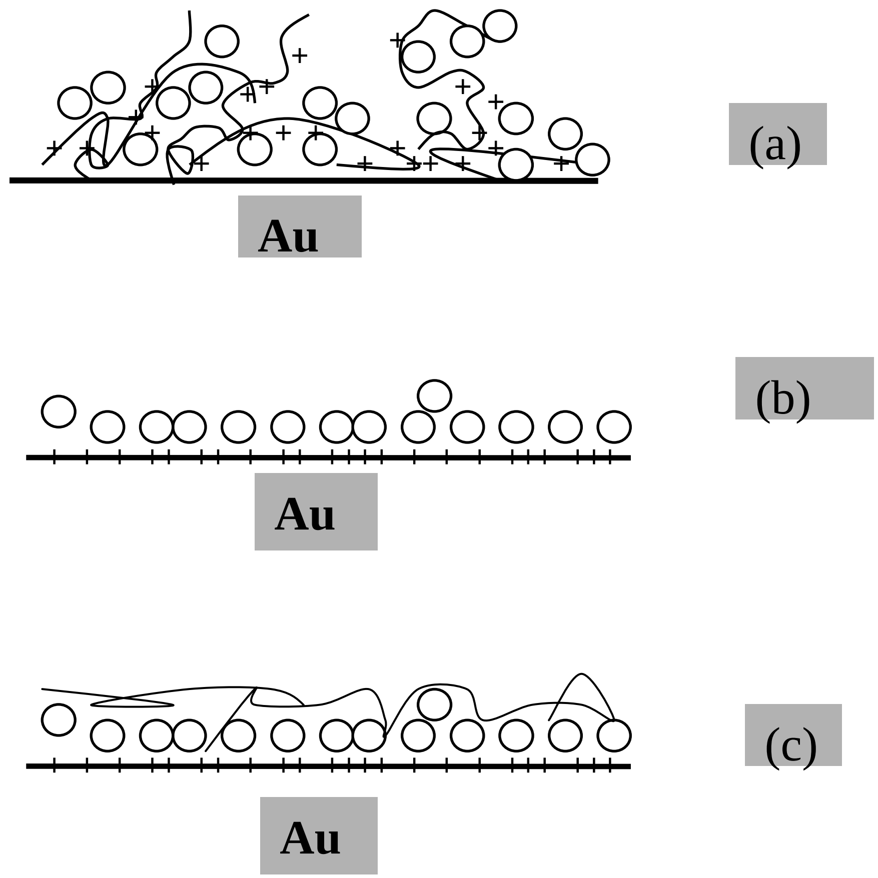
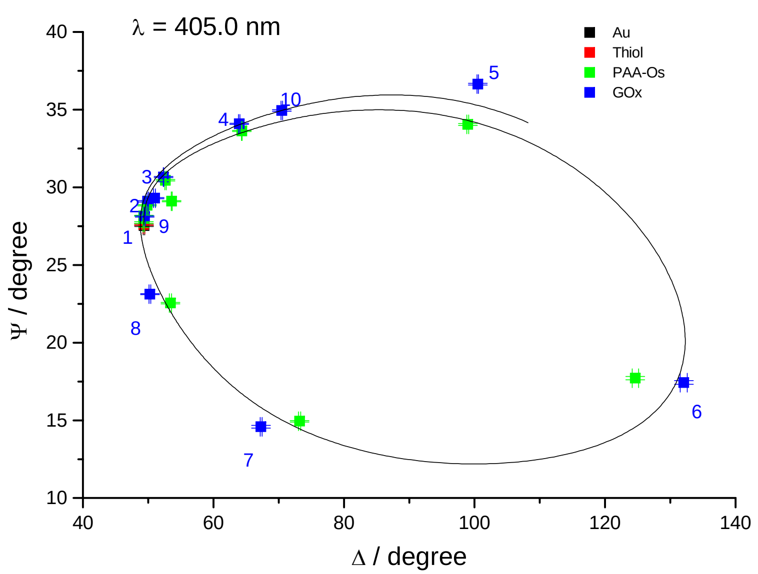
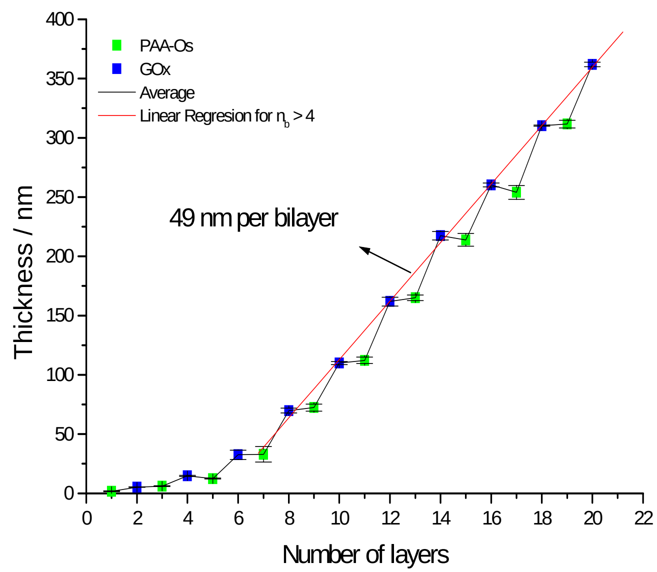
0
200
400
600
800
Å


Height Distribution
N = 80
30
25
20
15
10
5
0
GOx molecule number
1,0
2,0
3,0
4,0
5,0
6,0
7,0
Height in nm


(a)
Au
(b)
Au
(c)
Au

ELIPSOMETRY





Effect of the Polycation Nature on the Structure of Layer-by-Layer Electrostatically Self-Assembled Multilayers of Polyphenol Oxidase

E.S. Forzani, M. Lopez Teijelo, F. Nart, E.J. Calvo,V.M. Solis

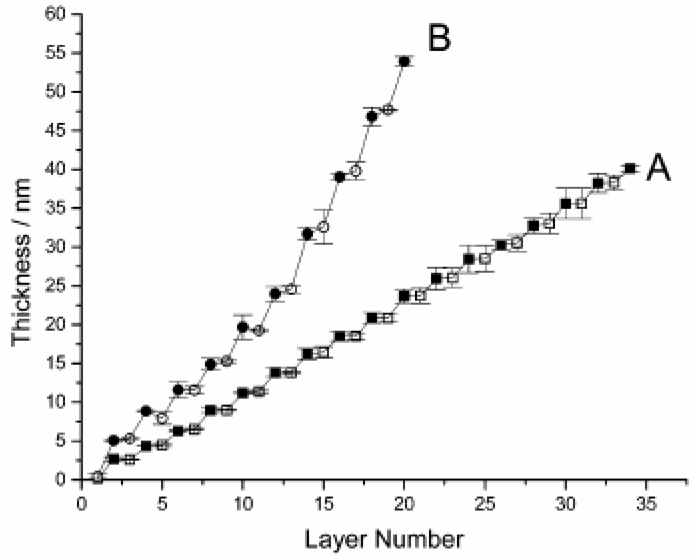
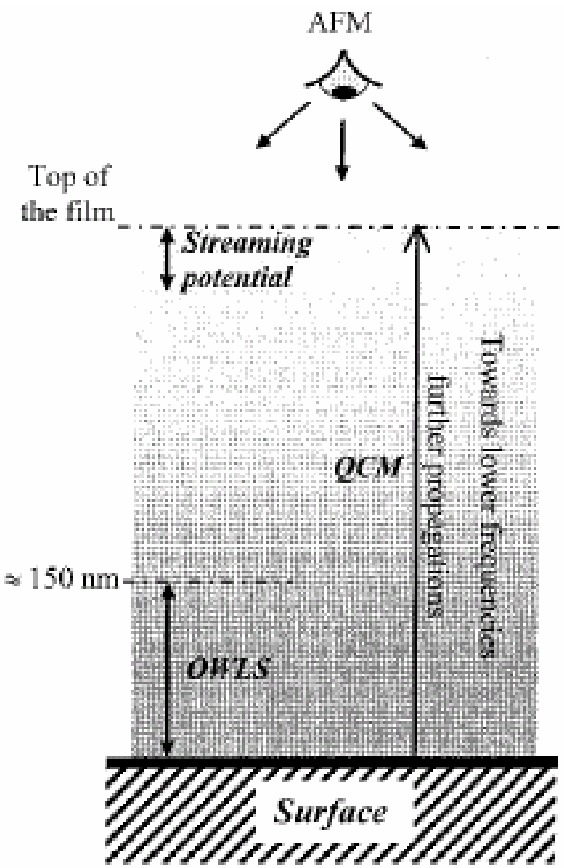
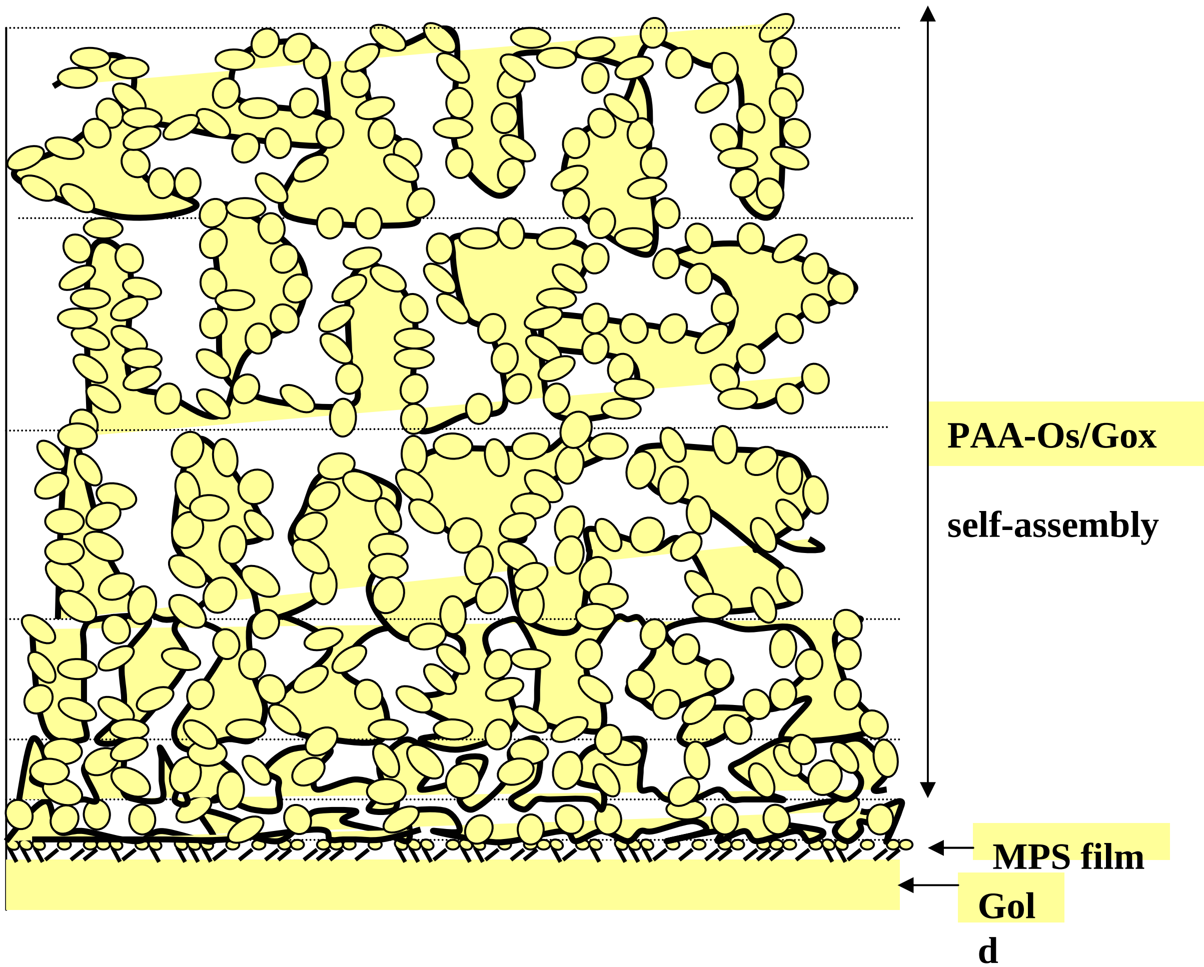


Figure 2. Plots of average thickness vs number of layers (*N*) for multilayer self-assembled systems composed of (A) PAH/PPO and (B) PDDA/PPO. (Open symbols): polycation-terminated layers; (filled symbols) PPO-terminated layers. Error bars represent thickness standard deviation values measured at 546.1 and 632.8 nm.

Biomacromolecules, Vol. 4, No. 4, 2003

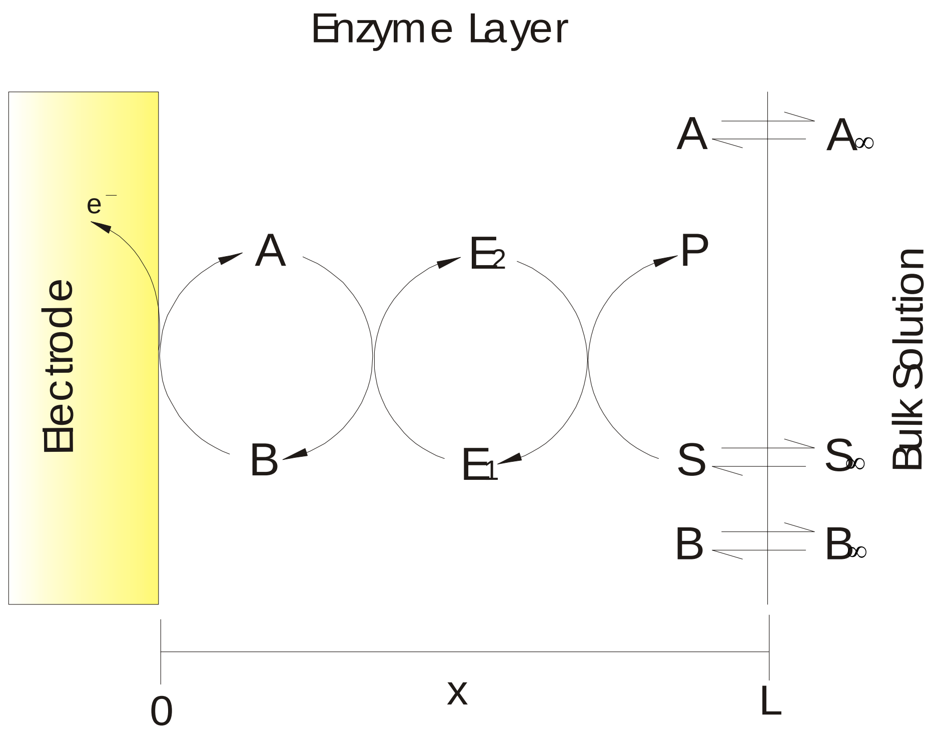
Film thickness is technique dependent





PAA-Os/Gox
self-assembly
MPS film
Gol
d

MODELLING



Diffusion-enzyme kinetic Problem

$$\frac{\partial[Os_{III}]}{\partial t} = D_{e^-}\frac{\partial^2[Os_{III}]}{\partial x^2} - k[E_{red}][Os_{III}]$$

$$\frac{\partial[S]}{\partial t} = D_S\frac{\partial^2[S]}{\partial x^2} - \frac{k_{cat}[E_{ox}][S]}{K_M + [S]}$$

$$\frac{\partial[E_{ox}]}{\partial t} = k[E_{red}][Os_{III}] - \frac{k_{cat}[E_{ox}][S]}{K_M + [S]}$$

Steady State

$$\frac{\partial[E_{ox}]}{\partial t} = \frac{\partial[A]}{\partial t} = \frac{\partial[S]}{\partial t} = 0$$

Diffusion-enzyme kinetic Problem

$$\frac{\partial[Os_{III}]}{\partial t}=D_{e^-}\frac{\partial^2[Os_{III}]}{\partial x^2}-k[E_{red}][Os_{III}]$$

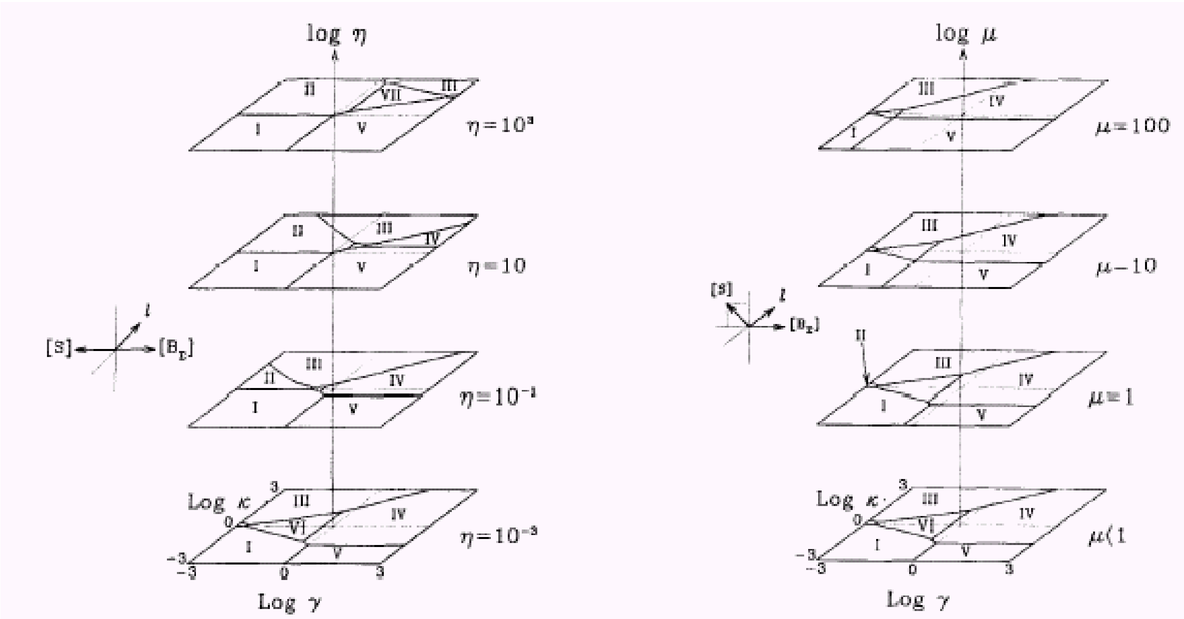
$$\frac{\partial[S]}{\partial t}=D_S\frac{\partial^2[S]}{\partial x^2}-\frac{k_{cat}[E_{ox}][S]}{K_M+[S]}$$

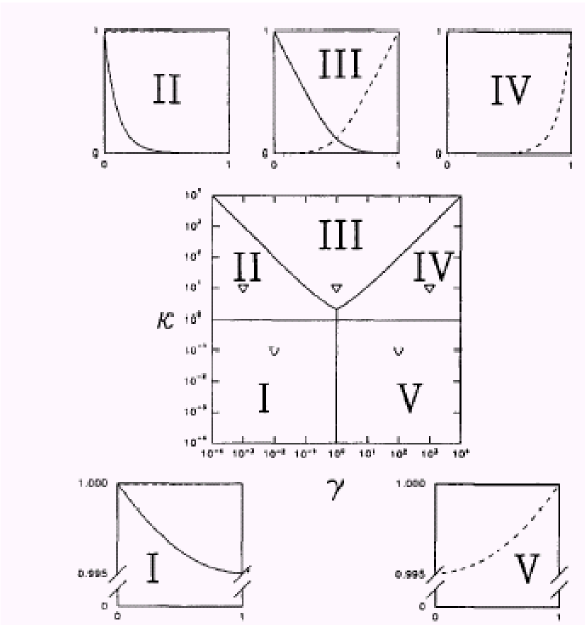
$$\frac{\partial[E_{ox}]}{\partial t}=k[E_{red}][Os_{III}]-\frac{k_{cat}[E_{ox}][S]}{K_M+[S]}$$

$$D_A\ \frac{d^2[Os_{(III)}]}{dx^2}=\frac{kk_{cat}[Os_{(III)}][S][E]}{k[Os_{(III)}](K_M+[S])+k_{cat}[S]}$$

$$D_S\ \frac{d^2[S]}{dx^2}=\frac{kk_{cat}[Os_{(III)}][S][E]}{k[Os_{(III)}](K_M+[S])+k_{cat}[S]}$$

KINETIC CASE DIAGRAM

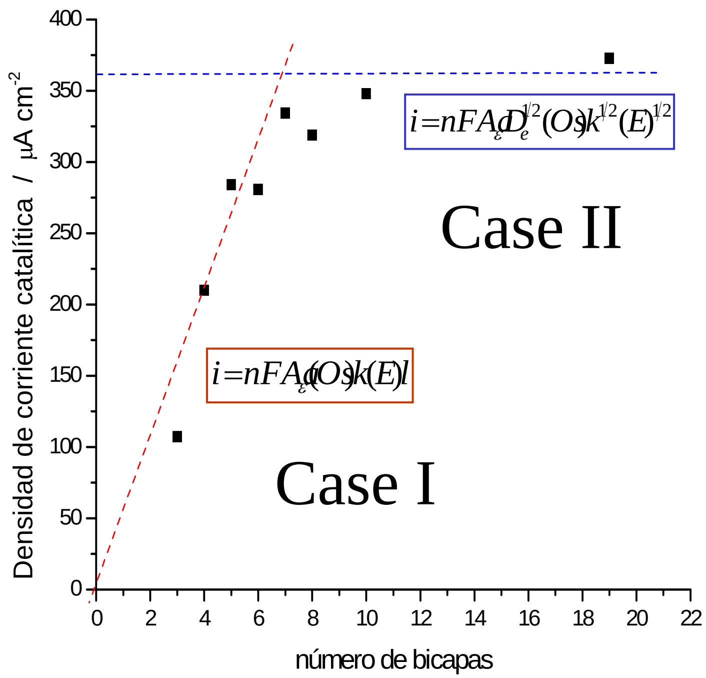




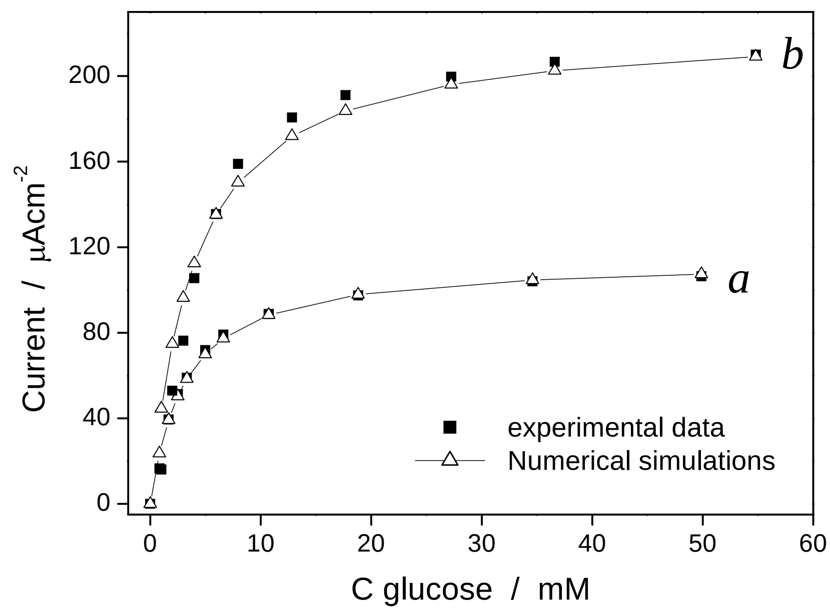
P.N. Bartlett, et al, J. Electroanalytical Chemistry, 397 (1995), 61

LIMITING KINETIC CASES

CASE	Current
I	$I = nFa_\varepsilon[A]k_A[e_\Sigma]\ell$
II	$I = nFAa_\varepsilon D_e^{1/2}(Os)k^{1/2}(E)^{1/2}$
III	$I = nF\left(D_A[A]a_\varepsilon + D_S K_S[S]\right)/\,\ell$
IV	$I = nFAK_S[S]\left\{\dfrac{k_{cat}[e_\Sigma]D_S}{K_M + \frac{1}{2}K_S[S]}\right\}^{1/2}$
V	$I = \dfrac{nFA\ell[e_\Sigma]k_{cat}K_S[S]}{K_M + K_S[S]}$
VI	$I = nFA\left(2a_\varepsilon[A]k_A[e_\Sigma]_S D_S K_S[S]\right)^{1/2}$
VII	$I = nFA\left\{\dfrac{2a_\varepsilon D_A[A]k_{cat}[e_\Sigma]K_S[S]}{K_M + K_S[S]}\right\}^{1/2}$



Densidad de corriente catalítica / μA cm-2
número de bicapas
i=nFAεDe1/2(Os)k1/2(E)1/2
i=nFAε(Os)k(E)l
Case II
Case I



Current / μAcm-2
C glucose / mM
b
a
experimental data
Numerical simulations

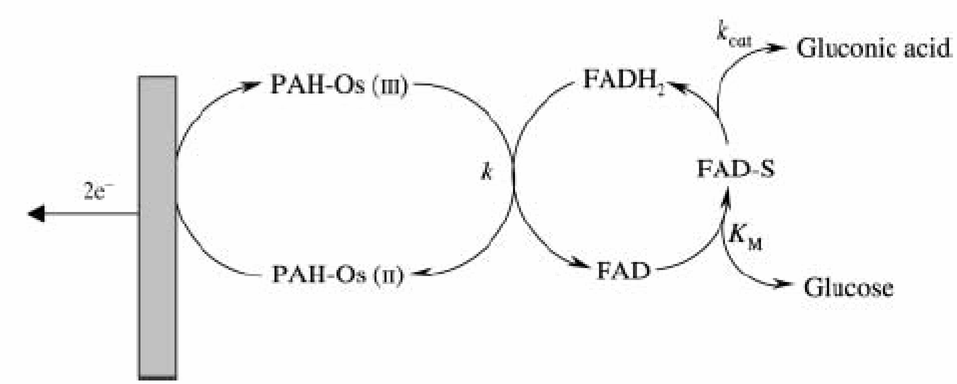


Figure 1. Reaction scheme of the electrochemical oxidation of β-D-glucose catalyzed by GOx and PAH-Os.

$$I_{cat} = \frac{2Fk_{cat}\Gamma_{enz}}{1 + \frac{k_{cat}}{k[\mathrm{Os(III)}]} + \frac{K_M}{[S]}}$$

J. Hodak, **R. Etchenique**, **E.J. Calvo**, **K. Singhal**, **P.N. Bartlett**.Langmuir, 1997, 13, 2716.

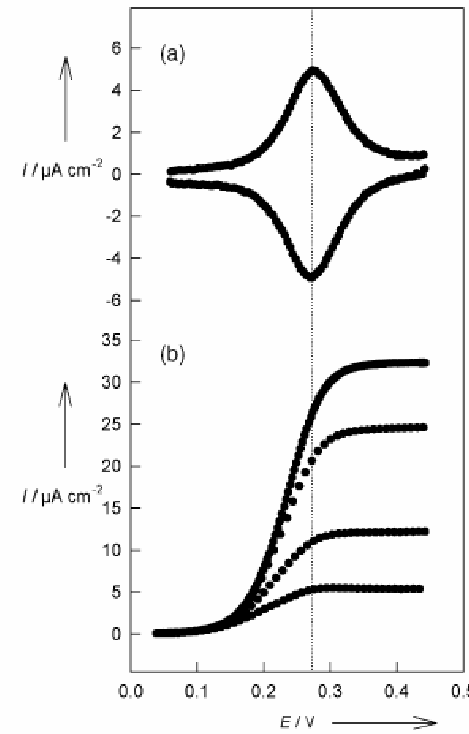
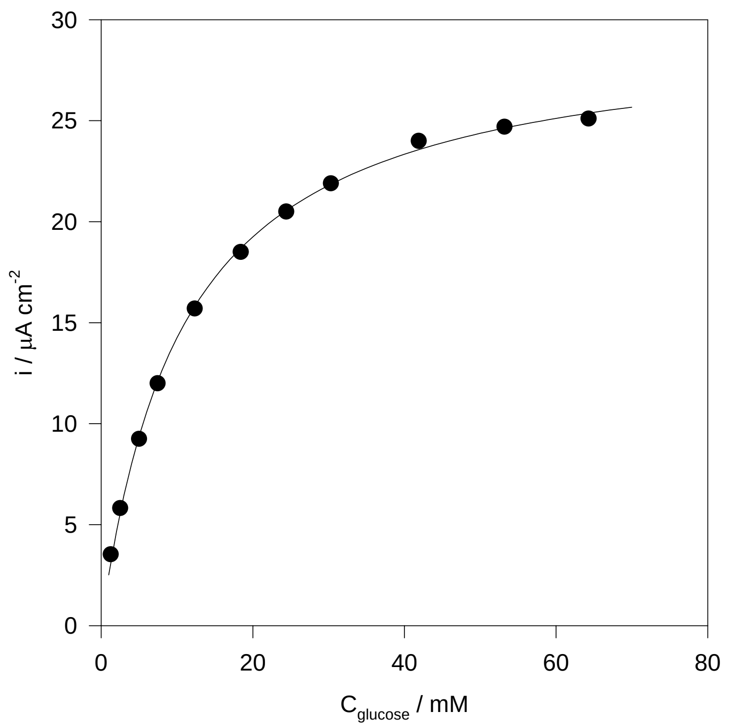


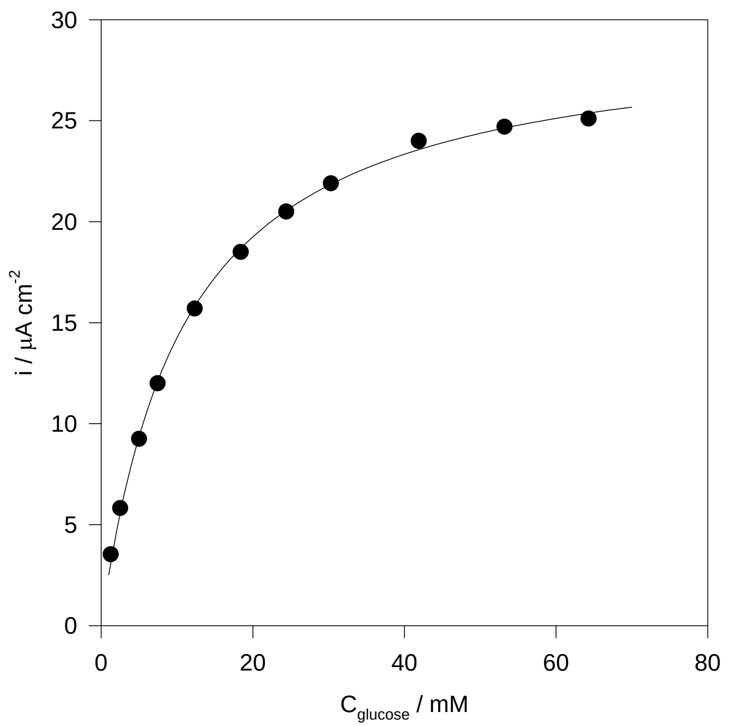
Figure 3. (a) Cyclic voltammetry at 50 mV s^{-1} in the absence of glucose for a PAH/GOx/PAH-Os electrode in 0.1 M Tris(hydroxymethyl)aminomethane (Tris) buffer, pH = 7.5 and 0.2 M KNO_3. (b) Dependence of the steady-state glucose catalytic oxidation current density on potential for the same electrode in 0.1 M Tris buffer, pH = 7.5, 0.2 M KNO_3 and increasing glucose concentration (2.5, 7.5, 25 and 60 mM) at 5 mV s^{-1}.

$$i_{cat} = \frac{2F\, k_{cat}\, \Gamma_E}{1 + \dfrac{k_{cat}}{k\,[Os(III)]} + \dfrac{K_{MS}}{[S]}}$$



From enzyme kinetics: k[Os] and Γ_E

$$i_{cat} = \frac{2F\,k_{cat}\,\Gamma_E}{1 + \dfrac{k_{cat}}{k\,[Os(III)]} + \dfrac{K_{MS}}{[S]}}$$



<u>From enzyme kinetics:</u> k[Os] and Γ_E

<u>From cyclic voltammetry:</u> q

<u>From ellipsometry:</u> d_f

Then, [Os] = q /(FAd_f) and then we can evaluate the wiring efficiency, k

Supramolecular Architectures of Electrostatic Self-Assembled Glucose Oxidase Enzyme Electrodes

Ernesto J. Calvo*[a] and Alejandro Wolosiuk[b]

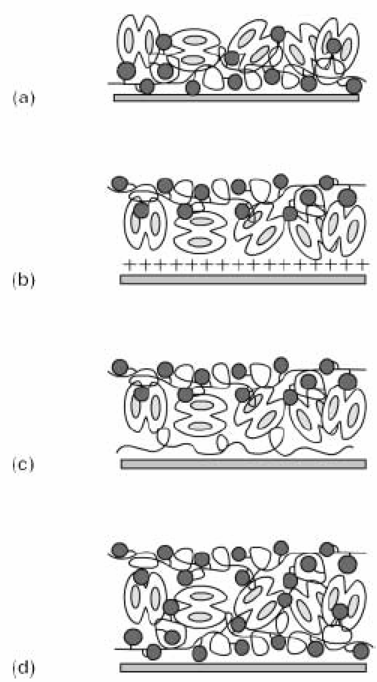


Figure 2. Different enzyme monolayers on electrostatically self-assembled electrodes: (a) PAH-Os/GOx, (b) cysteamine/GOx/PAH-Os, (c) PAH/GOx/PAH-Os, and (d) PAH-Os/GOx/PAH-Os.

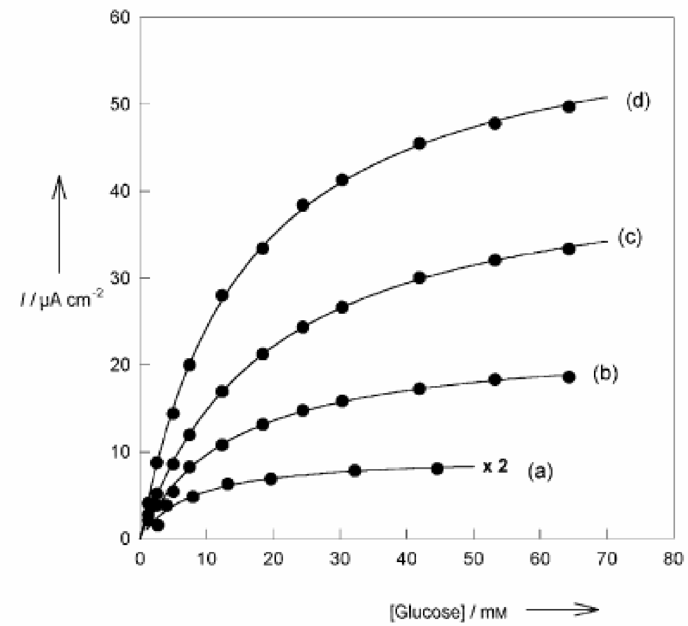


Figure 4. Typical catalytic current response for β-D-glucose concentration for self-assembled nanostructured thin films based on different architectures: (a) PAH/Os/GOx, (b) cysteamine/GOx/PAH-Os, (c) PAH/GOx/PAH-Os, (d) $(PAH\text{-}Os)_2/(GOx)_1$. All measurements were performed in 0.1 M Tris buffer of pH = 7.5, 0.2 M KNO_3.

ChemPhysChem 2004, 5, 235 – 239

ENZYME MULTILAYER

RESEARCH PAPER

PCCP

www.rsc.org/pccp

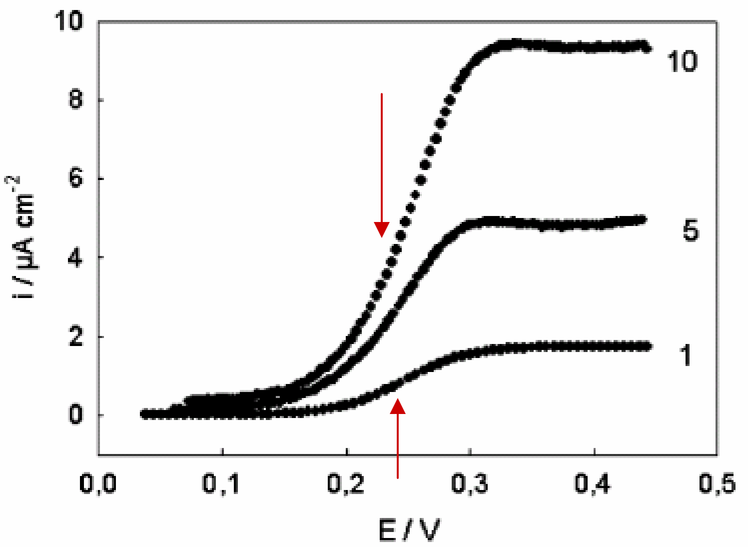
Supramolecular multilayer structures of wired redox enzyme electrodes†

Ernesto J. Calvo,*[a] Claudia B. Danilowicz[b] and Alejandro Wolosiuk[c]

[a] *INQUIMAE, Departamento de Química Inorgánica, Analítica y Química Física, Facultad de Ciencias Exactas y Naturales, Pabellón 2 Ciudad Universitaria, AR-1428 Buenos Aires, ArgentinaE-mail: calvo@qi.fcen.uba.ar; Fax: 5411 45763341; Tel: 5411 45763378*

[b] *Physics Department, Harvard University, 17 Oxford St., Cambridge, MA 02 138, USA. E-mail: claudia@atom.harvard.edu*

[c] *Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, IL 61801, USAE-mail: alew@uiuc.edu*



$(PAH\text{-}Os)_{10}(GOx)_{10}$

$(PAH\text{-}Os)_{5}(GOx)_{5}$

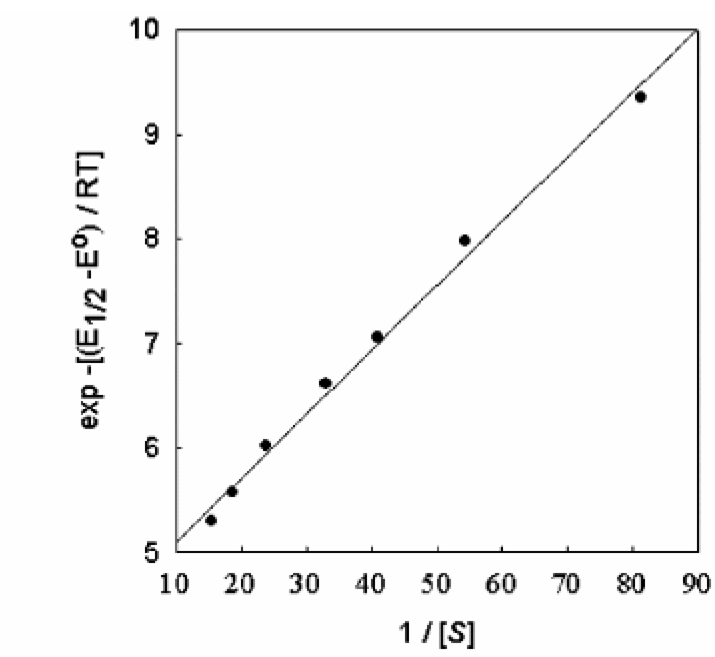
$(PAH\text{-}Os)_{1}(GOx)_{1}$

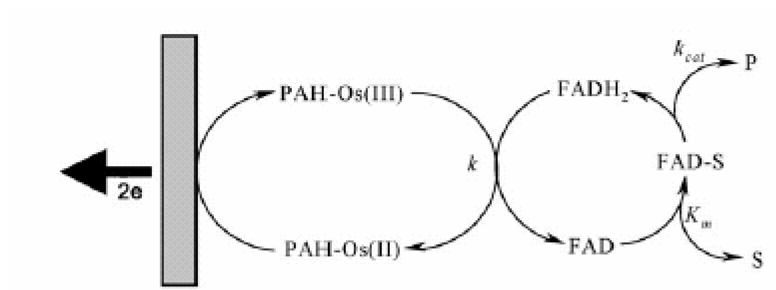
Phys. Chem. Chem. Phys., 2005, 7, 1800–1806

Pelicula delgada $l^2 << D_s K_{MS}/(kcat\,[E_\Sigma])$

$$i_{cat} = \frac{2F\,k_{cat}\,\Gamma_E}{1+\frac{k_{cat}}{k[Os^{III}]}+\frac{K_{MS}}{[S]}}$$

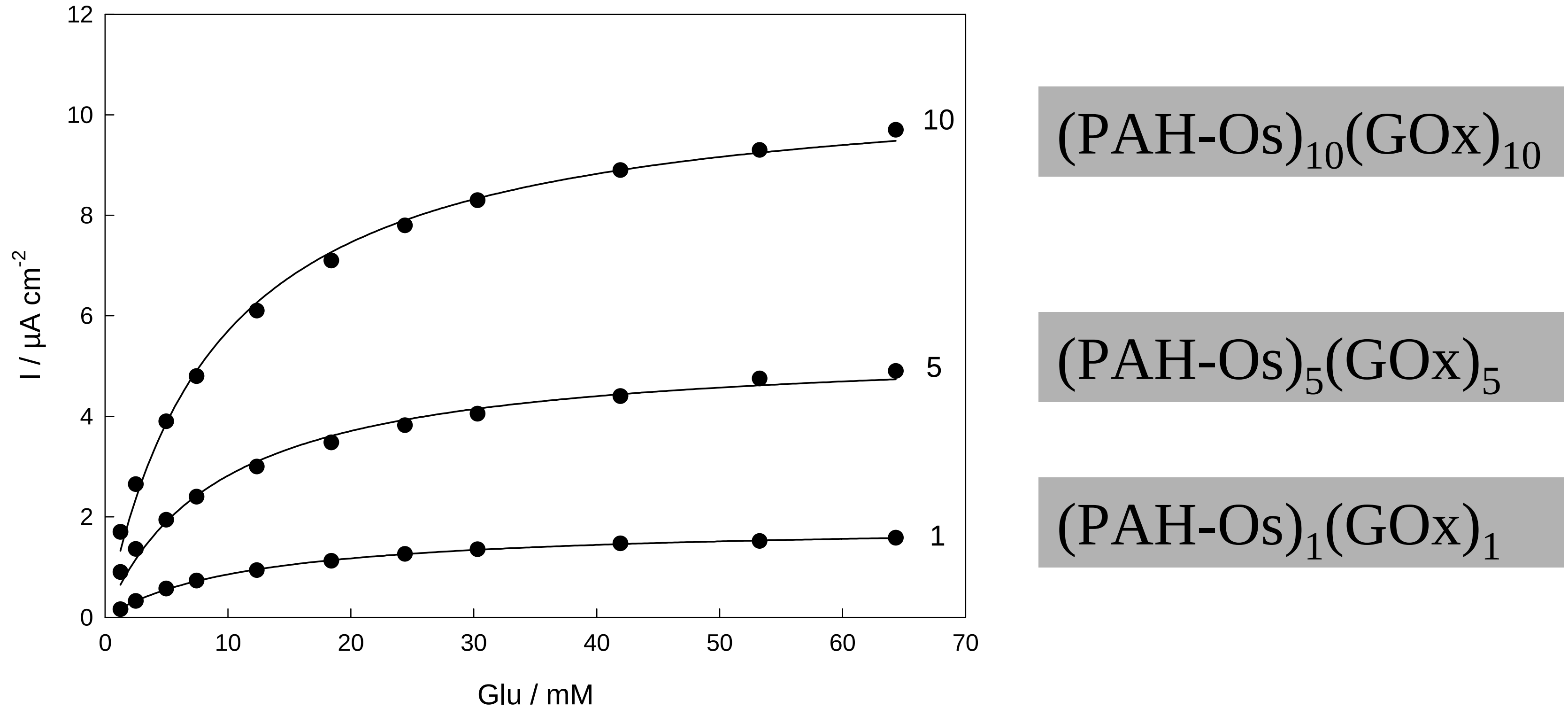
Case I Case V





$$\exp[-(E_{1/2}-E^0)F/RT] = \frac{k[Os_{TOT}]K_M}{k_{cat}[S]^*} + \frac{k[Os_{TOT}]}{k_{cat}} - 1$$

Limit Case I/ Case V


I / µA cm^{-2}
Glu / mM
10
5
1
(PAH-Os)$_{10}$(GOx)$_{10}$
(PAH-Os)$_5$(GOx)$_5$
(PAH-Os)$_1$(GOx)$_1$

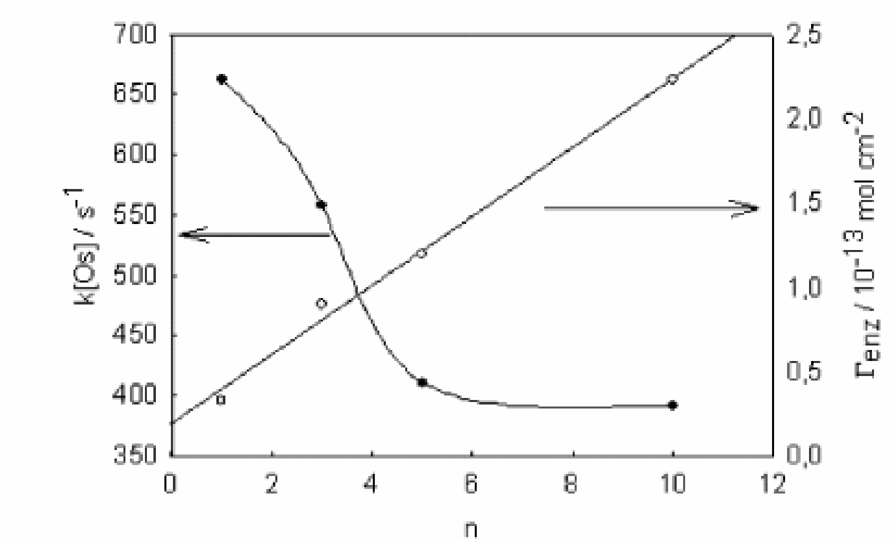
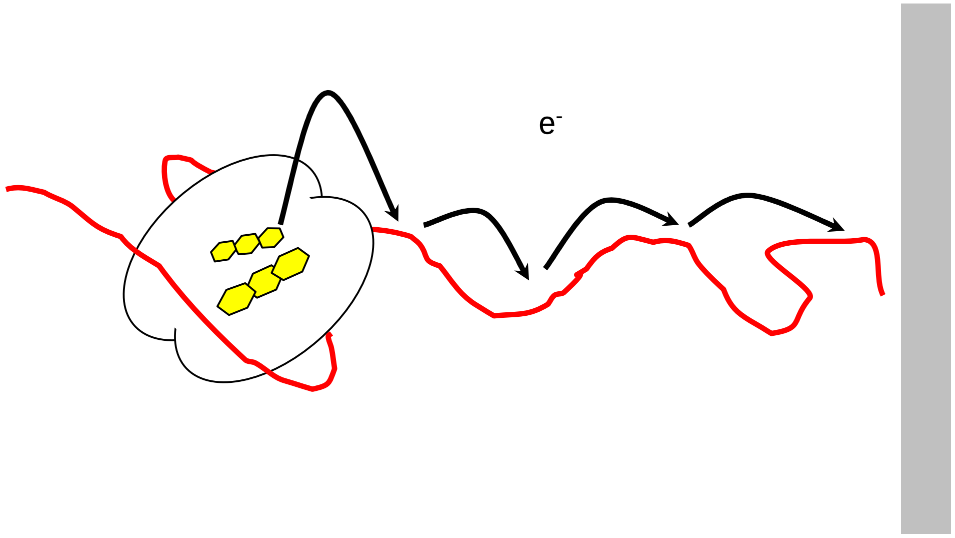


Table 1 Kinetic data for (PAH-Os)(GOx) multilayers

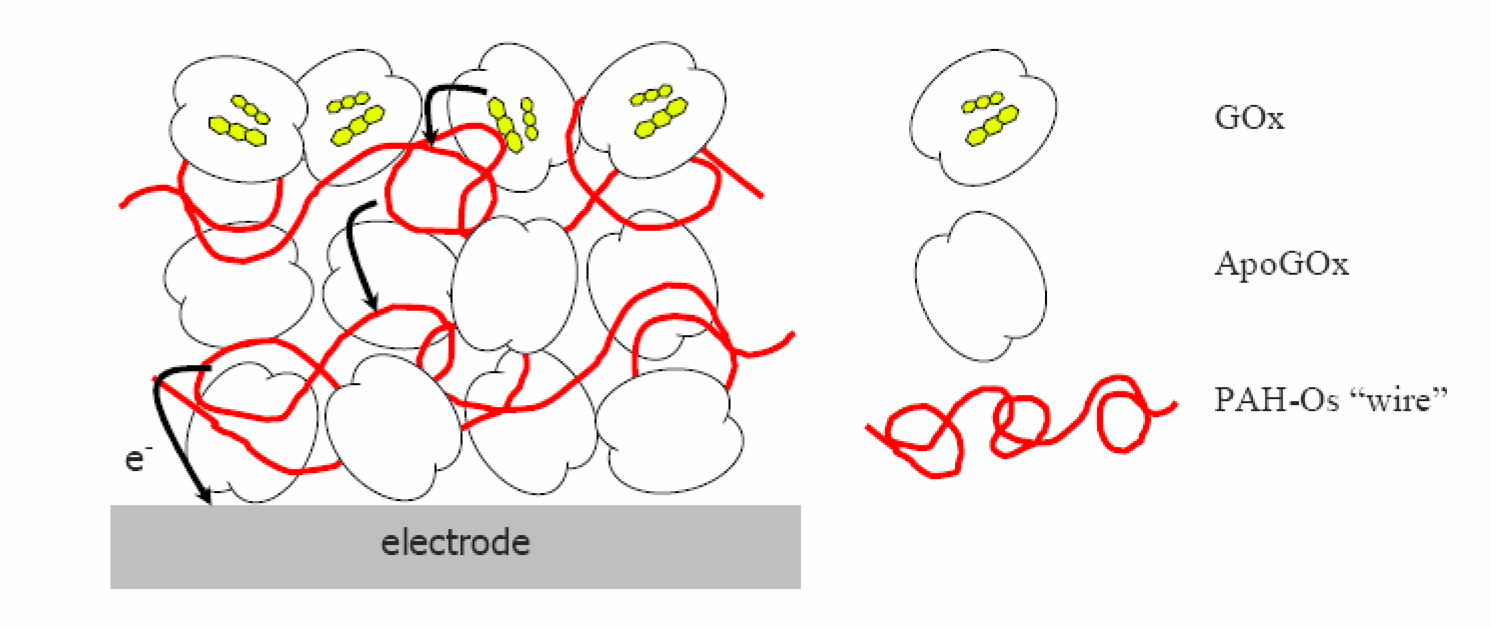
Dipping cycles	q/μC cm^{-2}	d_f/nm	[Os]/M	Γ_E/10^{-13} mol cm^{-2}	k[Os]/s^{-1}	K/mol dm^{-3} s^{-1} × 10^4
1	6.0	8.1	0.077	0.33	662	0.86
3	9.3	28.0	0.034	0.90	558	1.60
5	11.7	46.4	0.026	1.20	410	1.60
10	17.2	92.7	0.019	2.23	391	2.00

CABLE MOLECULAR



EFECT OF THE DISTANCE BETWEEN ENZYME AND ELECTRODE

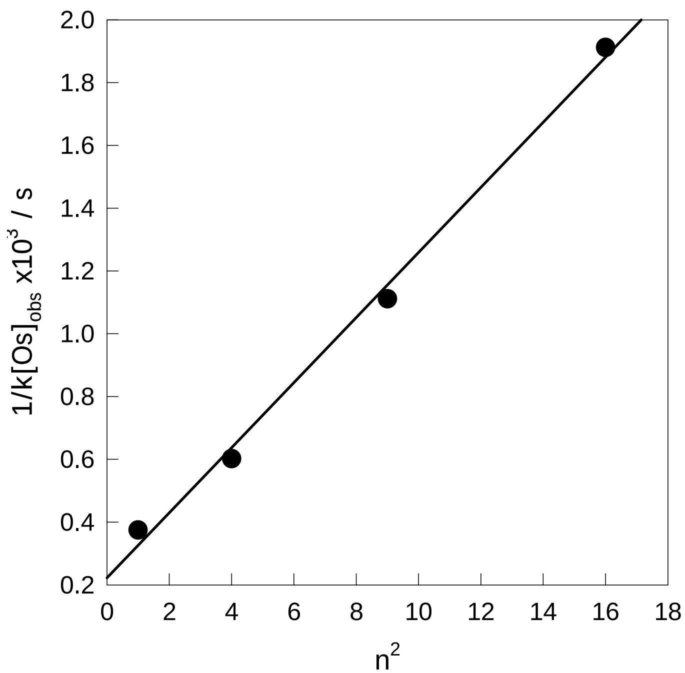
LEGO CHEMISTRY

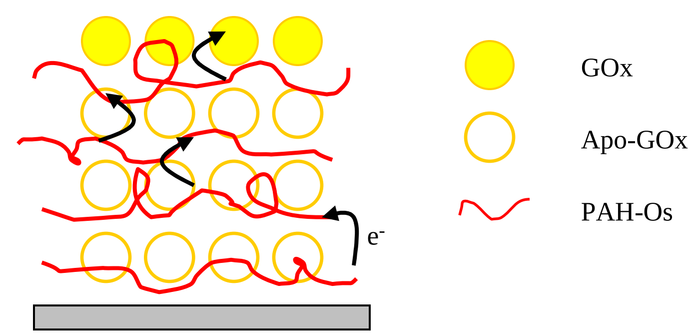


Propagación de carga en la multicapa enzimatica

$$\frac{1}{k_M(obs)} = \frac{1}{k_M} + \frac{1}{k_D}$$

Podemos epresar k_D como la inversa del tiempo característico de difusión dado por la ecuación de Einstein $\tau = \Delta x^2/2D_e$
Con $\Delta x = n \cdot d_{layer}$, n es el numero de capa y d_{layer} es la distacia promedio entre capas , D_e es el coeficiente de difusión por electron-hopping.

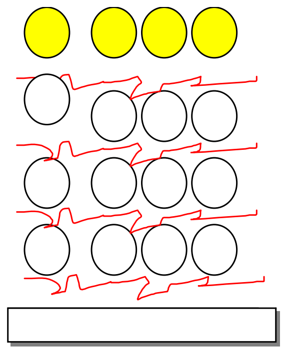




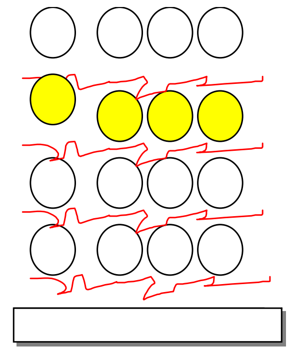
$$D_e = 1.2\,10^{-9}\ cm^2 s^{-1}$$

$$(D_{solucion} \sim 10\text{-}6\ cm2\ s\text{-}1)$$

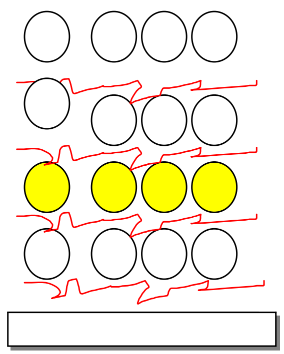
E.J. Calvo, C. Danilowicz, A. Wolosiuk, J. Am. Chem. Soc., 124, (2002), 2452



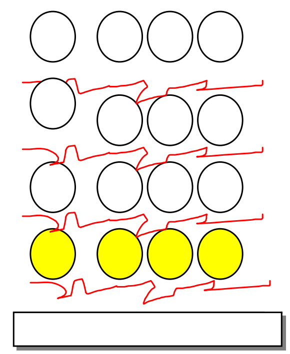
k = 1.26 x 10^3 M^{-1} s^{-1}



k = 2.30 x 10^3 M^{-1} s^{-1}



k = 4.05 x 10^3 M^{-1} s^{-1}



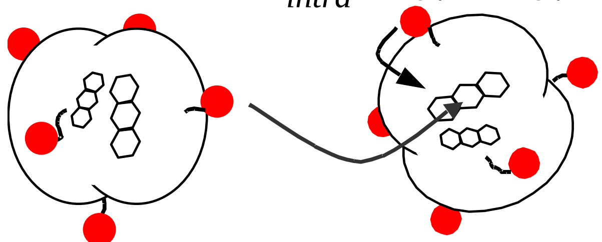
k = 6.31 x 10^3 M^{-1} s^{-1}

REDOX MEDIATION IN SOLUTION, INTRA AND INTERMOLECULAR

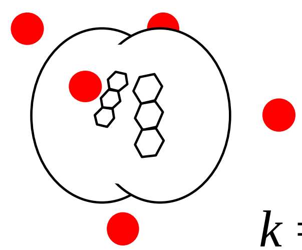
Soluble Redox Mediator, soluble mediator and integrated intra and intermolecular

$$k_{autoens} \sim 5 - 9\ 10^{3}\ M^{-1}\ s^{-1}$$

$k_{intra} = 0.2 – 0.4\ s^{-1}$

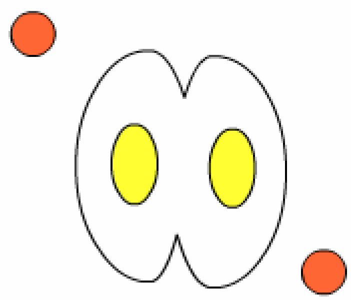


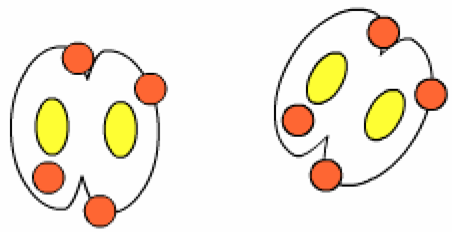
$k_{inter} = 8.3\ 10^{3}\ M^{-1}\ s^{-1}$

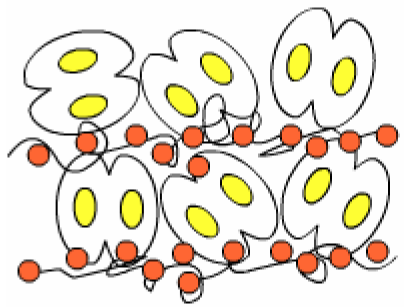


$k = 3.7\ 10^{5}\ M^{-1}\ s^{-}$

Danilowicz, C.; Cortón, E.; Battaglini, F. *J.Electroanal.Chem.* **1998**, *445*, 49.
Battaglini, F.; Bartlett, P.N.; Wang, J.H. *Anal.Chem.* **2000**, *72*, 502.







k = 3.7 10^5 M^{-1} s^{-1}

k_{inter} = 8.3 10^3 M^{-1} s^{-1}

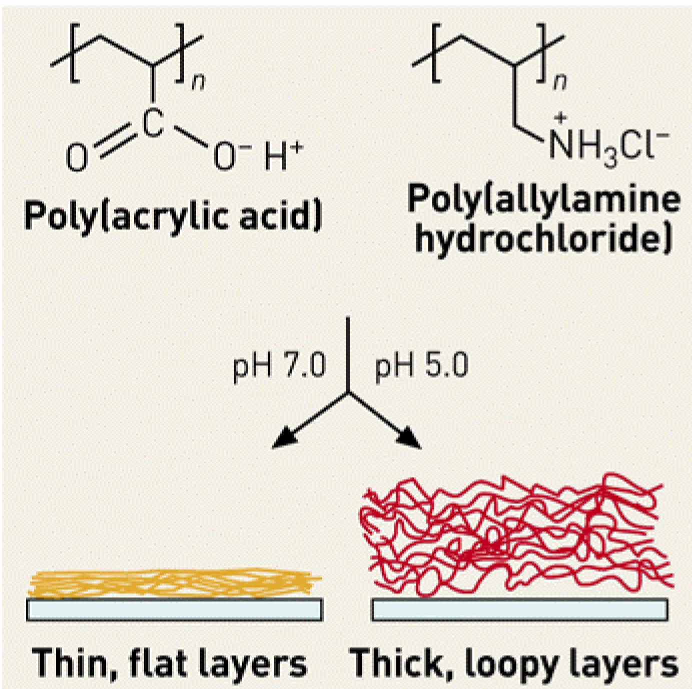
k_{intra} = 0.2 – 0.4 s^{-1}

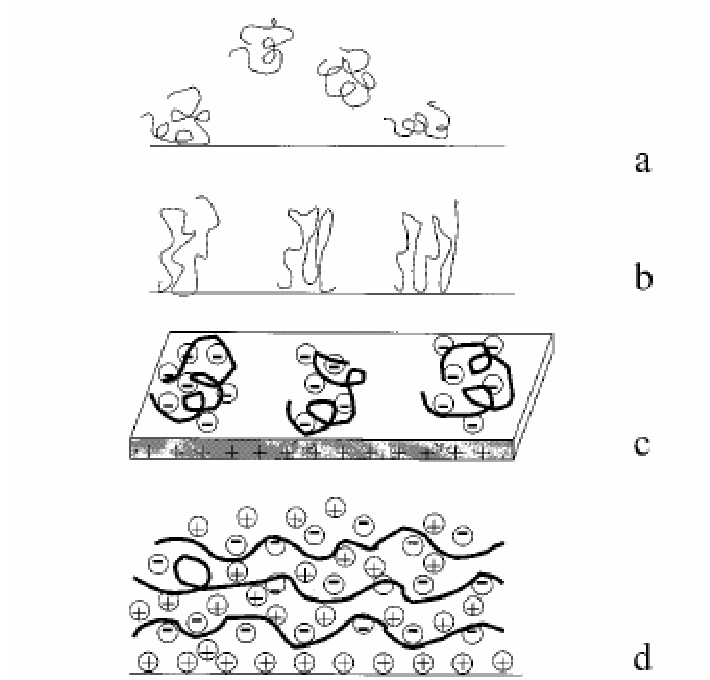
k ~ 5 - 9 10^3 M^{-1} s^{-1}

Danilowicz, C.; Cortón, E.; Battaglini, F. *J.Electroanal.Chem.* **1998**, *445*, 49.
Battaglini, F.; Bartlett, P.N.; Wang, J.H. *Anal.Chem.* **2000**, *72*, 502.

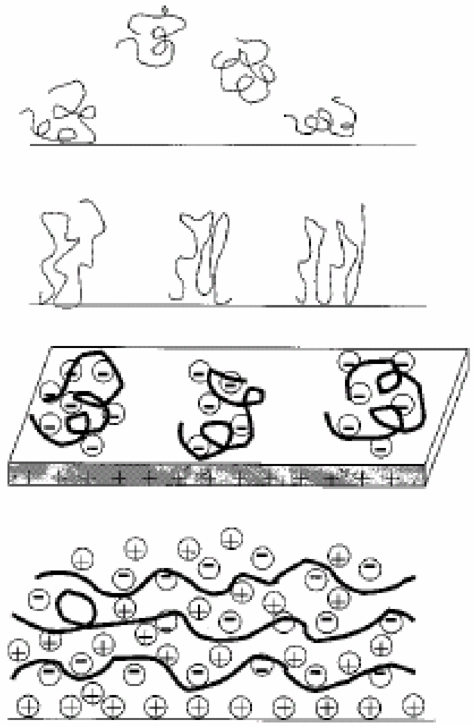
EFFECT OF FILM STRUCTURE AND THICKNESS

We can regulate the thickness and structure in multilayer enzyme films by adjusting the charge density through the adsorption solution pH.





S.S. Shirartori, M.F. Rubner, Macromolecules, 2000, 33, 4213



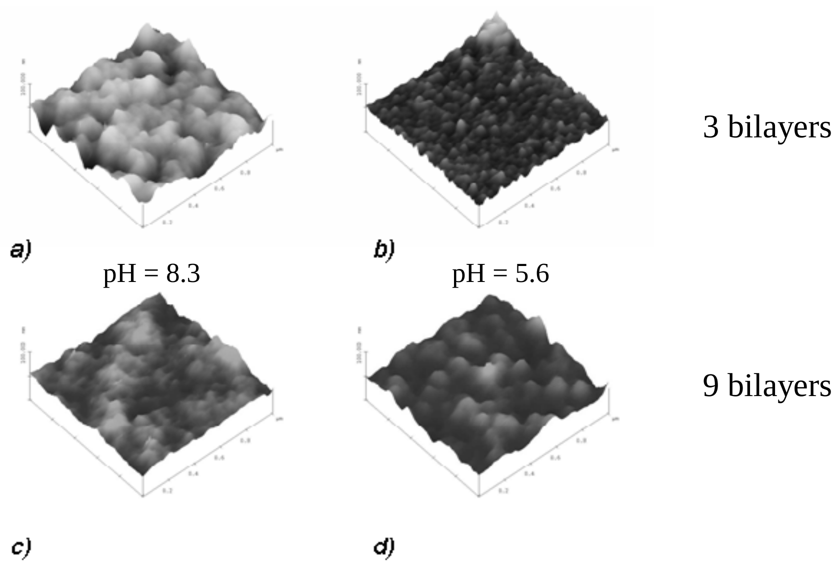
a

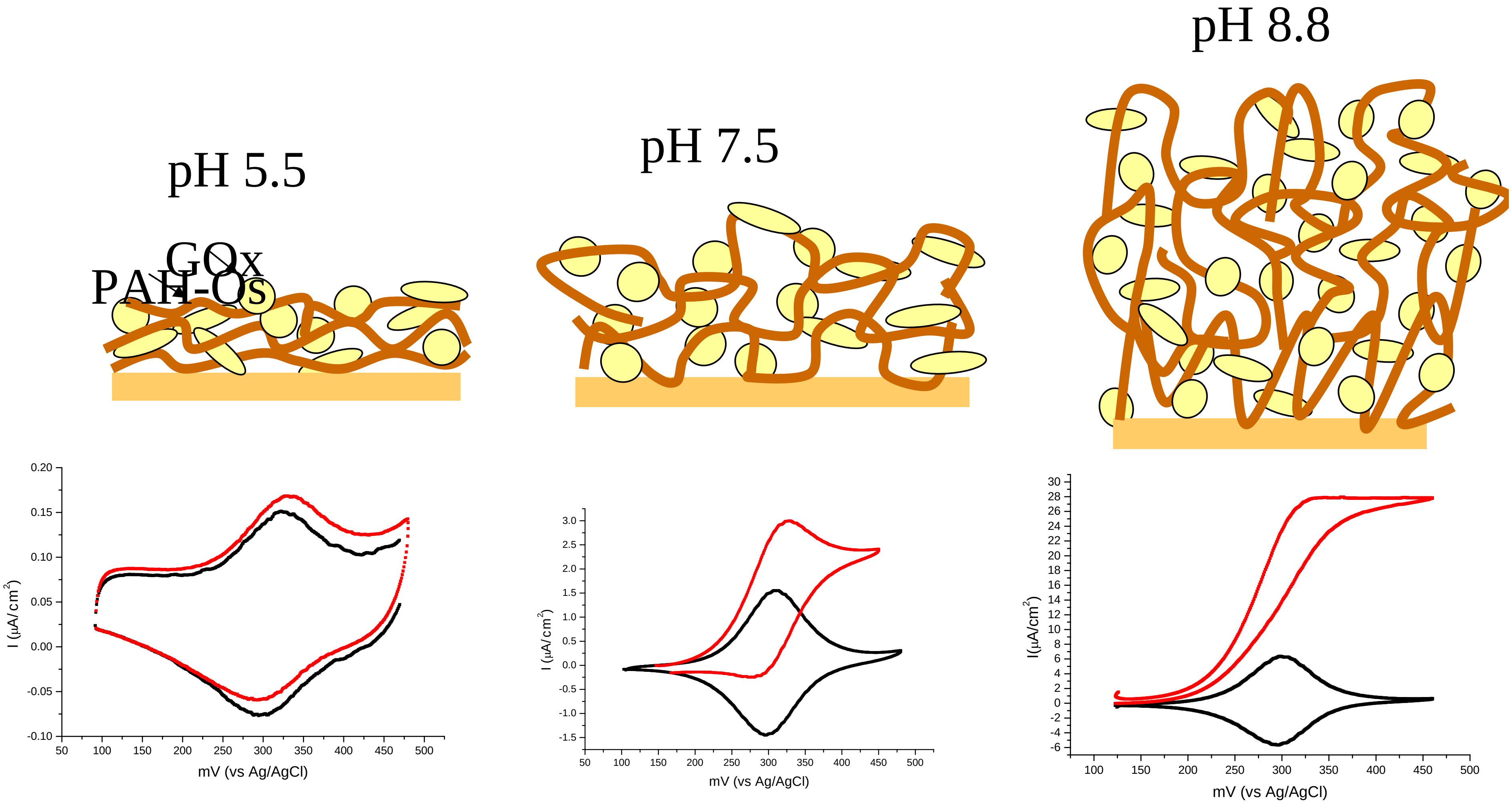
b

c

d

Tapping mode AFM
Au/MPS/(PAH-Os/GOx)$_n$ under water

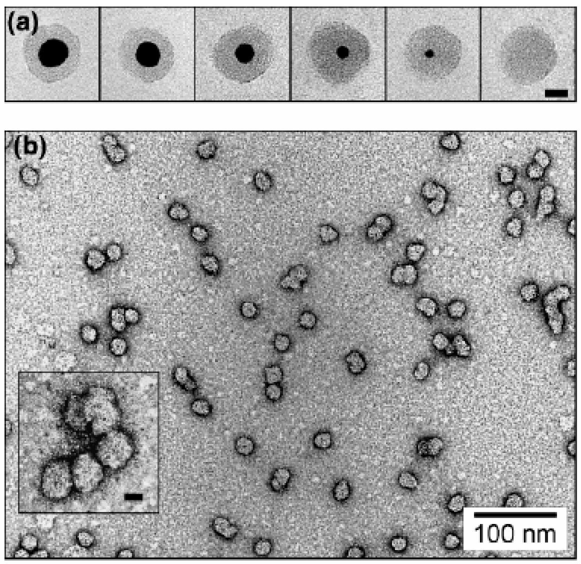




V. Flexer, E. Forzani, E.J. Calvo, Anal. Chem., 78, 2006, 399-407.

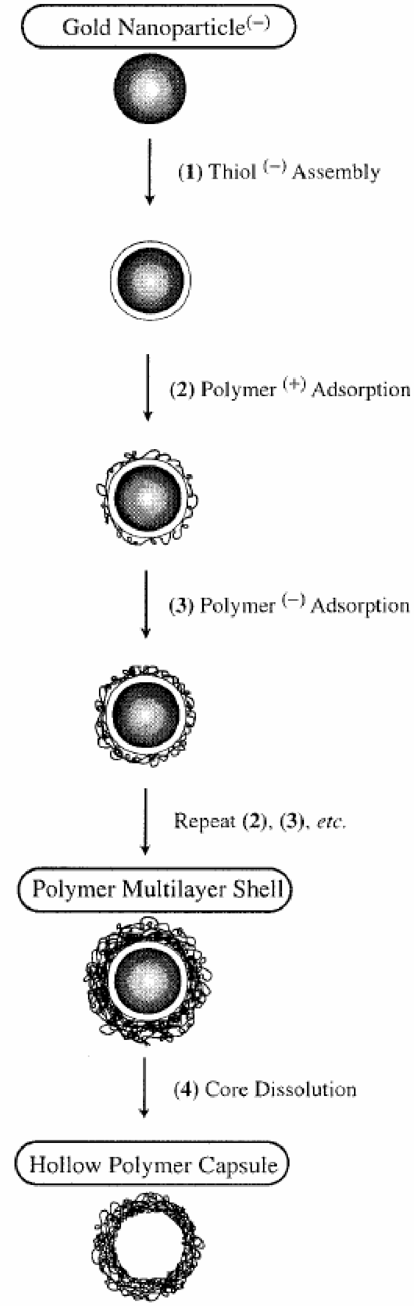
Not only flat surfaces

Polyelectrolyte Coating of Metal Nanoparticles, D.I. Gittins, F. Caruso, J. Phys. Chem. B, 2001, 105, 6846-6852.



G. Schneider, G. Decher, Nanoletters 2004, 4, 1833-1839

SCHEME 1: Schematic Diagram Illustrating the Layer-by-Layer Polymer Deposition Process Applied to Gold Nanoparticles.

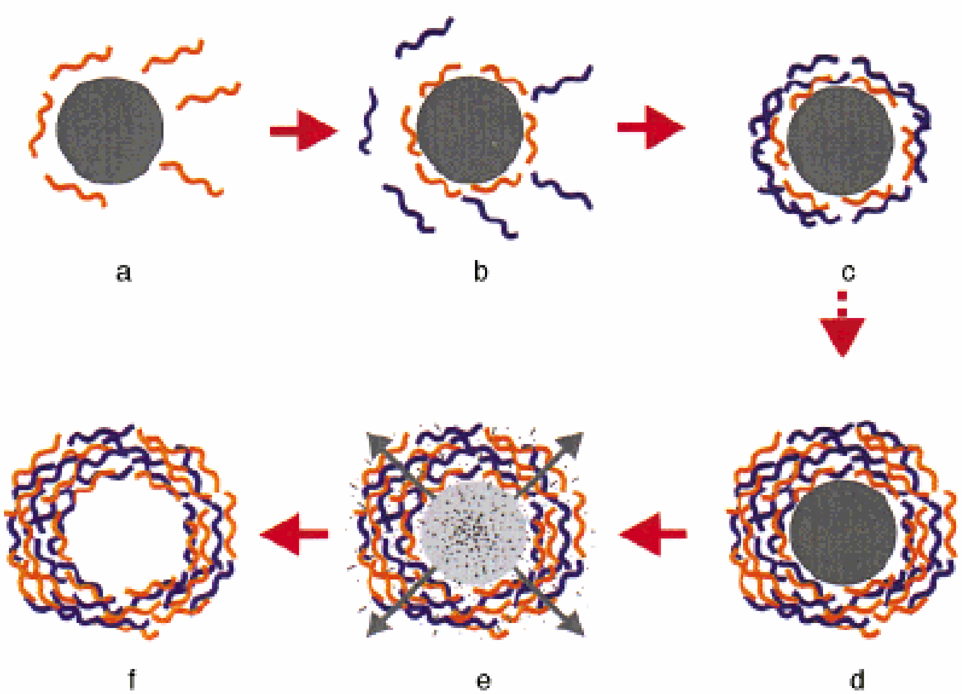


COMMUNICATIONS

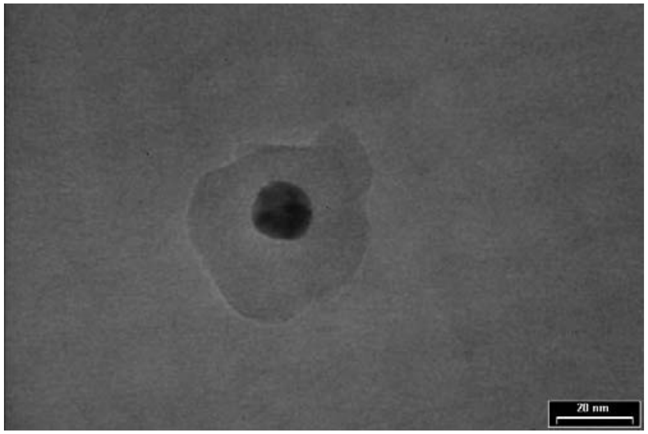
Novel Hollow Polymer Shells by Colloid-Templated Assembly of Polyelectrolytes**

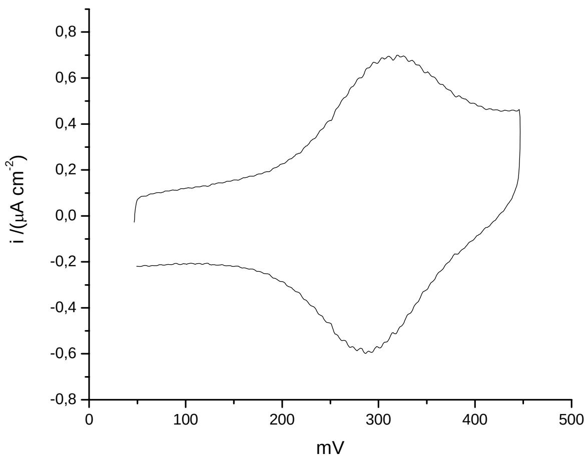
Edwin Donath,* Gleb B. Sukhorukov, Frank Caruso, Sean A. Davis, and Helmuth Möhwald

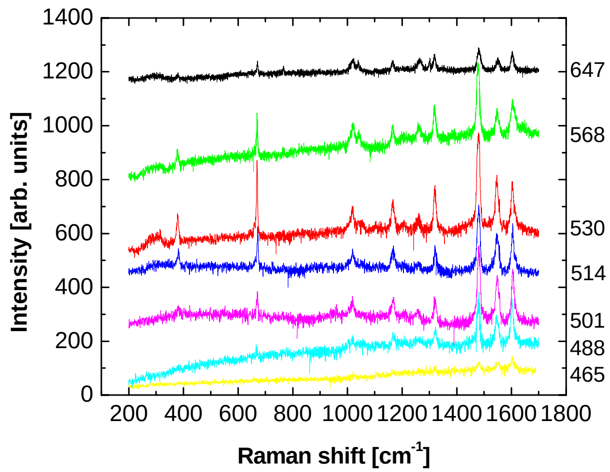
Angew. Chem. Int. Ed. 1998, 37, No. 16, 2202



Au-NP/$(PAH\text{-}Os)_3(PVS)_3$

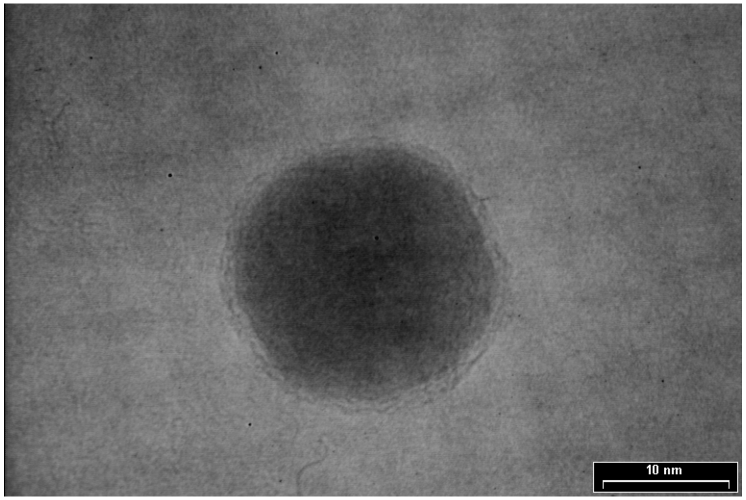


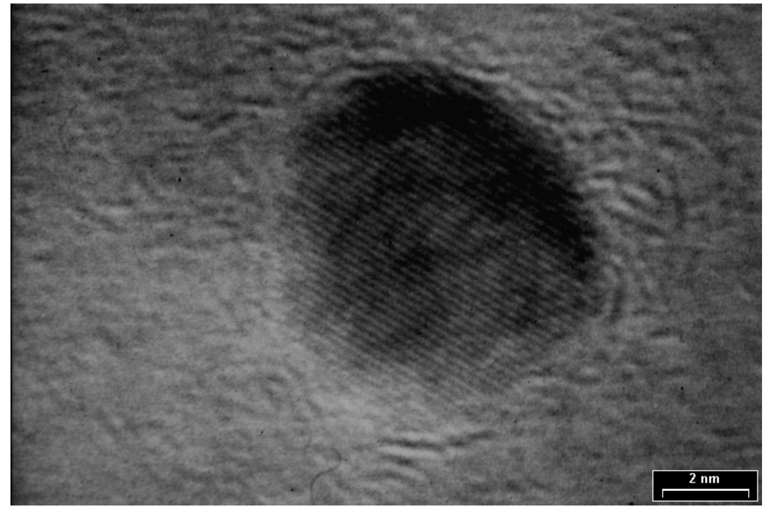


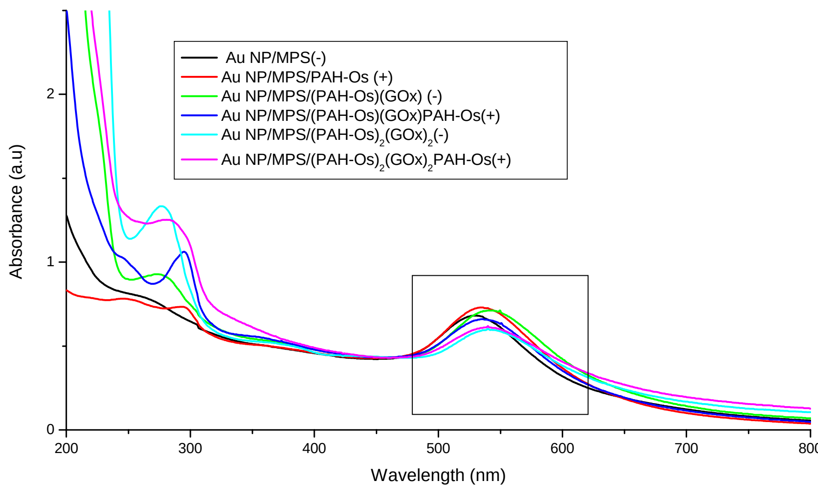


P. Scodeller, N. Tognalli, H. Troiani, A. Fainstein, E.J. Calvo, Unpublished results

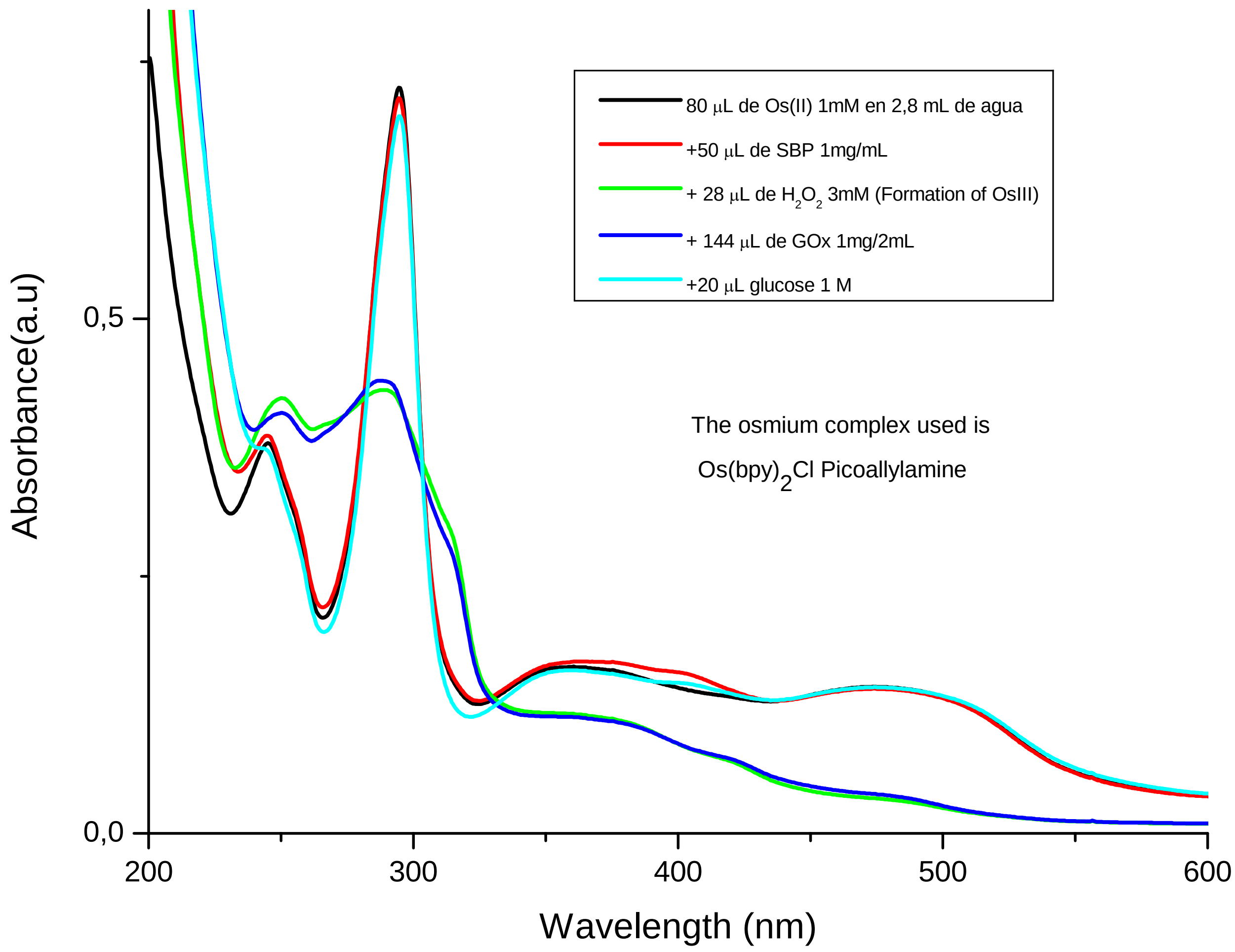
Au-NP/(PAH-Os)$_3$(GOx)$_3$



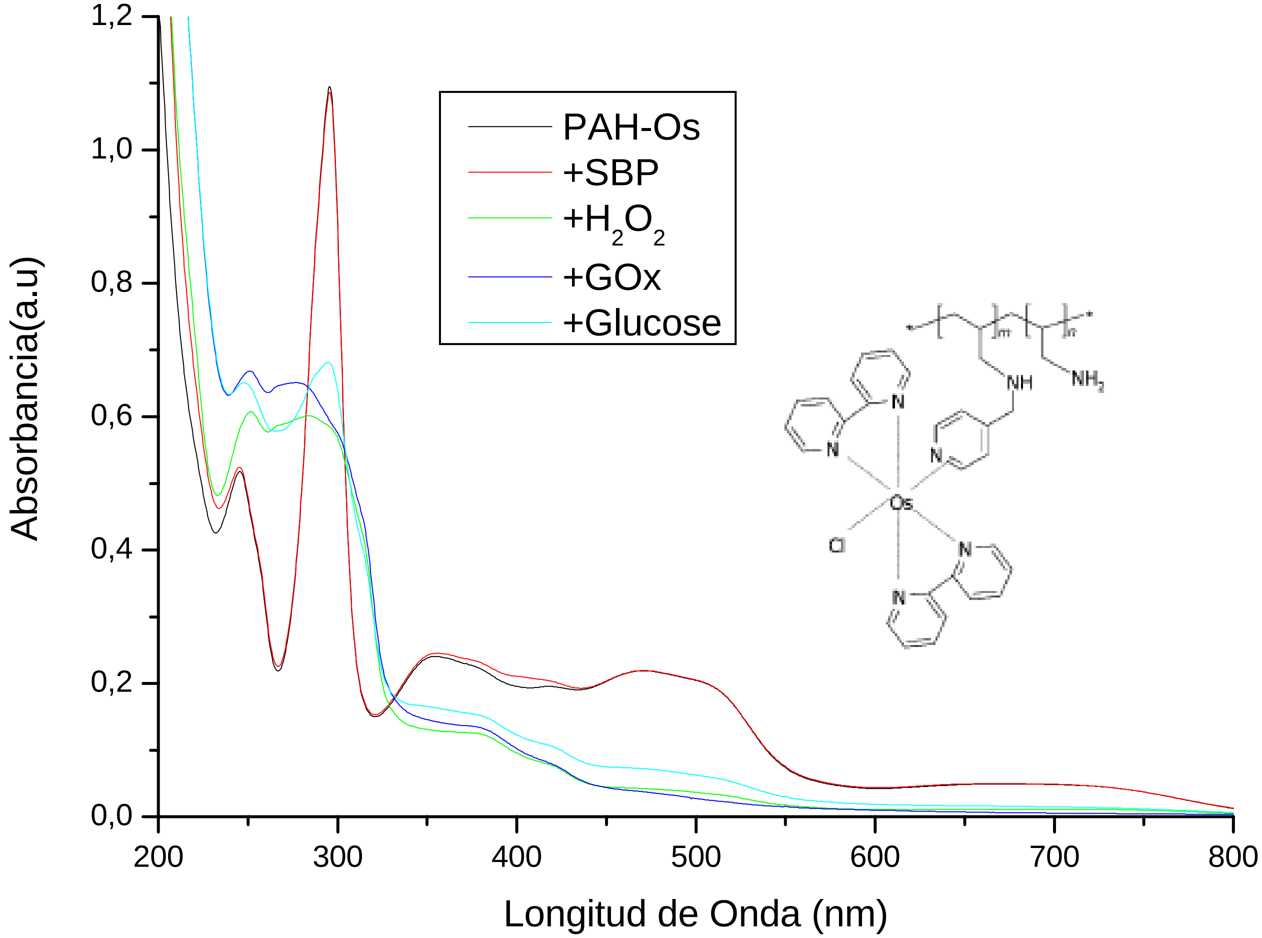




P. Scodeller, N. Tognalli, H. Troiani, A. Fainstein, E.J. Calvo, Unpublished results

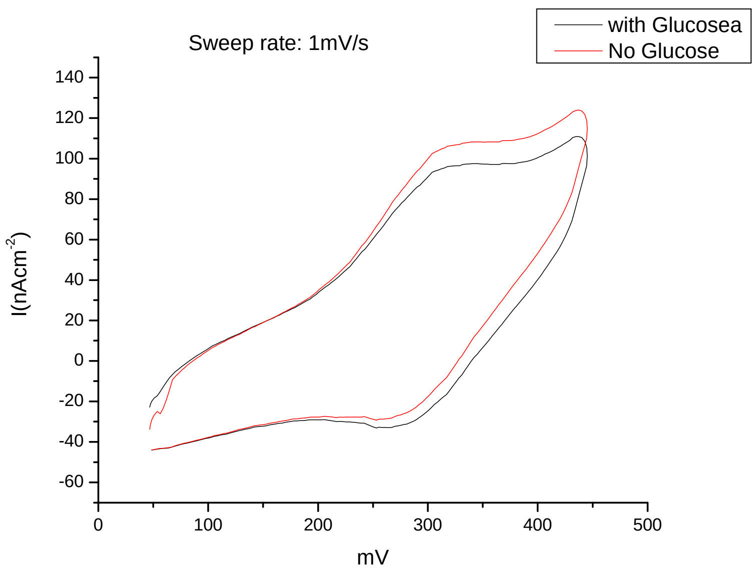


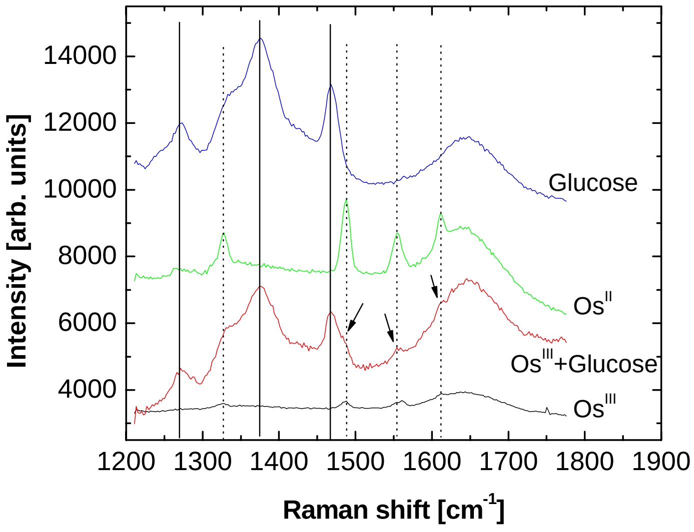
80 μL de Os(II) 1mM en 2,8 mL de agua
+50 μL de SBP 1mg/mL
+ 28 μL de H_2O_2 3mM (Formation of OsIII)
+ 144 μL de GOx 1mg/2mL
+20 μL glucose 1 M
The osmium complex used is
$Os(bpy)_2Cl$ Picoallylamine
Absorbance(a.u)
0,5
0,0
200
300
400
500
600
Wavelength (nm)



1,2
1,0
0,8
0,6
0,4
0,2
0,0
200
300
400
500
600
700
800
Absorbancia(a.u)
Longitud de Onda (nm)
PAH-Os
+SBP
+H_2O_2
+GOx
+Glucose
N
NH
NH_2
Os
Cl

Molecular recognition and Signal generation





Electrocatalysis

Resonant Raman