SUPPORTING INFORMATION FOR:

Characterization and Performance of Electrostatically Adsorbed Ru-Hbpp Water Oxidation Catalysts


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Fax: 34 93 581 3101; Tel: 34 93 586 8295; Email: xavier.sala@uab.cat
**Figure S1.** 1D and 2D NMR spectra (400 MHz, 298 K, acetonitrile-d$_3$) for L1$^+$: (a) schematic representation, (b) $^1$H NMR, (c) COSY, (d) $^{13}$C-$^1$H NMR, (e) HSQC NMR (aromatic region), (f) HMBC NMR (aromatic region).
Figure S2. Mercury plot of the unit cell of $1^+$ (counteranions have been omitted for the sake of clarity).
**Figure S3.** Mercury plot of the unit cell of $2^{4+}$ (counteranions have been omitted for the sake of clarity).

**Figure S4.** 1D and 2D NMR spectra (400 MHz, 298 K, acetone-$d_6$) for complex $2^{4+}$: (a) $^1$H NMR, (b) COSY, (c) $^{13}$C-$^1$H NMR, (d) HSQC NMR (aromatic region), (e) HMBC NMR (aromatic region).

a)
Figure S5. 1D and 2D NMR spectra (250 MHz, 298 K, acetone-\textit{d}_6) for complex 34+: (a) $^1$H NMR with resonance assignment, (b) COSY, (c) $^{13}$C-{$^1$H} NMR, (d) HSQC NMR (aromatic region) (e) HMBC NMR (aromatic region).
Figure S6. Cyclic voltammogram for complex 1⁺ in 0.1 M n-Bu₄NPF₆ in acetonitrile at 100 mV/s scan rate. Glassy carbon electrode is used as working electrode and the potential is measured vs. SSCE.
Figure S7. Cyclic voltammogram for [RuIIICl3(trpy)] in 0.1 M n-Bu4NPF6 in acetonitrile at 100 mV/s scan rate. Glassy carbon electrode is used as working electrode and the potential is measured vs. SSCE.

Figure S8. Cyclic voltammogram for the acetato-bridged complex 34+ in 0.1 M n-Bu4NPF6 in acetone at 100 mV/s scan rate. Glassy carbon electrode is used as working electrode and the potential is measured vs. SSCE.
Figure S9. UV-Vis spectrum of washing acetone for Silica-$2^{4+}/3^{4+}$.

Figure S10. Diffuse reflectance UV-vis spectra of Silica (black line) and Silica-$2^{4+}$ (orange line).
**Figure S11.** UV-vis spectra of a FTO-TiO$_2$-2$^{4+}$ film with (red line) and without (purple line) previous overnight activation at pH=12.

**Figure S12.** Cyclic voltammograms for FTO-TiO$_2$-2$^{4+}$ in 0.1 M $n$-Bu$_4$NPF$_6$ in CH$_2$Cl$_2$ at 50 (a) and 100 (b) mV/s scan rate. FTO is used as working electrode and the potential is measured vs. SSCE.
Figure S13. UV-vis monitoring (aqueous face) of the stability of the generated FTO-TiO₂-2⁺ films when soaked at pH = 1 (a), pH = 7 (b) and pH = 12 (c) aqueous solutions.
Figure S14. UV-vis spectra of a FTO-Nafion film (blue line) and FTO-Nafion-24+ (red line).
**Figure S15.** Top: Chemically triggered water oxidation with $4^{5+}$ (1 mM) at pH = 1.0 in 0.1 M triflic acid solution in the presence of $(\text{NH}_4)_2\text{Ce}^{IV}(\text{NO}_3)_6$ (100 mM). (a) Manometric measurement. (b) On-line mass spectrometry ($\text{O}_2$ evolution, red line; $\text{CO}_2$ evolution, blue line). Bottom: Chemically triggered water oxidation with Silica-$4^{5+}$ (0.650 g, $1.82 \times 10^{-3}$ mmol/g) at pH = 1.0 in 0.1 M triflic acid solution in the presence of $(\text{NH}_4)_2\text{Ce}^{IV}(\text{NO}_3)_6$ (100 mM). (c) manometric measurement (d) on-line Mass Spectrometry ($\text{O}_2$ evolution, red line; $\text{CO}_2$ evolution, blue line).
Figure S16. Chemically triggered water oxidation with FTO-Nafion-4^5+ (1 mM) at pH =1.0 in 0.1 M triflic acid solution in the presence of (NH₄)₂Ce^{IV}(NO₃)₆ (100 mM).
Figure S17. Cyclic voltammetry for FTO-TiO$_2$-2$^{4+}$ before (a) and after (b) CPE at pH = 12 (NaOH 0.01M in water). Potential measured vs. Ag/AgCl.
**Figure S18.** Controlled Potential Electrolysis (CPE) of FTO-TiO$_2$-4$^{5+}$. $E = 1.1$ V for 8h. FTO used as working electrode. Potential measured vs. Ag/AgCl.

![Graph showing electrolysis data](image1)

**Figure S19.** Clark electrode profile during the CPE of FTO-TiO$_2$-4$^{5+}$. $E = 1.1$ V for 8h.

![Graph showing electrode profile](image2)
**Figure S20.** UV-vis spectra of the reaction vessel solution after CPE of FTO-TiO$_2$$^+$.

![UV-vis spectra](image)

**Figure S21.** Zoom of experimental (right side) and theoretical (left side) mass spectra for complex 1$^+$. 

![Zoomed mass spectra](image)
Figure S22. Mass spectrum for complex $2^{4+}$.

Figure S23. Zoom of experimental (right side) and theoretical (left side) mass spectra for complex $2^{4+}$.
**Figure S24.** Mass spectrum for complex 3^{4+}.

![Mass spectrum for complex 3^{4+}](image1)

**Figure S25.** Zoom of experimental (right side) and theoretical (left side) mass spectra for complex 3^{4+}.

![Zoom of experimental (right side) and theoretical (left side) mass spectra for complex 3^{4+}](image2)
Figure S26. Calcination ramps for the FTO-TiO₂ preparation.

Scheme S1. Procedure for the production of Nafion films over FTO electrodes and the subsequent attachment of ²⁺ and ³⁺ to prepare FTO-Nafion-²⁺ and FTO-Nafion-³⁺. (1-2) Deposition and drying of the Nafion solution. (3-5) Attachment of the catalyst onto the FTO-Nafion surface.
**Scheme S2.** Schematic representation of the proposed interaction between a FTO-Nafion support and catalyst $2^{4+}$.

**Scheme S3.** Representation of activated (red color) and non-activated (green color) methylenic moieties. Top: $\{\text{[Ru}^{II}(\text{H}_2\text{O})(\text{trpy})\}_2(\mu\text{-bpp-Bz})\}_3^{3+}$ and $\text{TiO}_2\text{-}[\text{[Ru}^{II}(\text{H}_2\text{O})\text{ (trpy)}\}_2(\mu\text{-bpp-Ra})\}_3^{3+}$. Bottom: $4^{5+}$ and $\text{SiO}_2\cdot4^{5+}$. 

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Scheme S4. Electrochemical cell formed by an anode (compartment A), which contains a FTO-TiO$_2$.2$^{4+}$ film and a Clark electrode and where the water oxidation reaction occurs; a cathode (compartment C), which contains the counter and the reference electrodes and where the reduction of protons to hydrogen takes place, and frit (membrane B) permeable to protons.
**Scheme S5.** Schematic representation of the proposed exchange of the chlorido-bridged ion for two water molecules in FTO-Nafion-2⁴⁺.

![Scheme S5](image)

**Table S1.** Crystallographic data for complex 1⁺.

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Table S2. Crystallographic data for complex 2⁺.
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Crystal system Monoclinic
Space group P2₁/n
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b = 52.120(3) Å  b = 91.920(2) °.
c = 28.8286(14) Å  g = 90.00 °.
Volume 39083(4) Å³
Z 4
Density (calculated) 1.252 Mg/m³
Absorption coefficient 0.486 mm⁻¹
F(000) 14688
Crystal size 0.35 x 0.15 x 0.15 mm³
Theta range for data collection 0.78 to 24.78 °.
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Reflections collected 480821
Independent reflections 65635 [R(int) = 0.1010 ]
Completeness to theta ~24.78 ° 0.977 %
Absorption correction Empirical
Max. and min. transmission 0.9307 and 0.8483
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 65635 / 30 / 3961
Goodness-of-fit on F² 1.033
Final R indices [I>2sigma(I)] R1 = 0.0743 , wR2 = 0.1663
R indices (all data) R1 = 0.1179 , wR2 = 0.1816
Largest diff. peak and hole 1.319 and -0.995 e.Å⁻³
Table S3. Selected interatomic distances (Å) and angles (°) for complex 1⁺.

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Table S4. Selected interatomic distances (Å) and angles (°) for complex 2⁺ (four independent molecules: A, B, C & D).

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**ANGLES**

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| N3B-Ru1B-N5B | 95.27(18) | N5D-Ru1D-N1D | 86.8(2) |
| N2B-Ru1B-N1B | 79.19(17) | N2D-Ru1D-N3D | 79.0(2) |
| N6B-Ru1B-N1B | 102.30(18) | N6D-Ru1D-N3D | 103.1(2) |
| N3B-Ru1B-N1B | 158.28(18) | N5D-Ru1D-N3D | 94.8(2) |
| N5B-Ru1B-N1B | 89.67(18) | N1D-Ru1D-N3D | 158.4(2) |
| N2B-Ru1B-C1B | 90.68(13) | N2D-Ru1D-C1D | 94.59(15) |
| N6B-Ru1B-C1B | 88.08(14) | N6D-Ru1D-C1D | 86.96(14) |
| N3B-Ru1B-C1B | 89.08(13) | N5D-Ru1D-C1D | 164.11(17) |</p>
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