UNIVERSITAT JAUME·I

# Lab session 3: Viologen functionalized electrochromic film

**Objective**:
Fabricate and characterize an electrochromic film made with $TiO_2$ functionalized with viologen.

**Materials**
Nanoporous $TiO_2$ Films
bis(2-phosphonoethyl)-4,4_-bipyridinium dichloride (Viologen)
Methanol
3-methoxypropionitrile, (3-MPN)
lithium triflate ($LiCF_3SO_3$)
Platinized FTO Glass or Pt wire.
2 Polimeters
Power source
Laser diode
Photodiode
Nitrogen gas
Fixtures

**Procedure**:
**- $TiO_2$ functionalization**
Immerse the $TiO_2$ electrode into a 0.5mM solution of viologen in metanol and kept it overnight to allow completing the sensitiser uptake
Putt he viologen activated $TiO_2$ electrode and the Platinised glass (or wire) in the small cubete with the flat windows and fix it. Fill with electrolyte made of 0.2M lithium triflate ($LiCF_3SO_3$) in 3-methoxypropionitrile

**- Electrochromic characterization**
Connect the $TiO_2$ to positive (WE) and the Pt to negative (CE).
Illuminate the photodiode with the laser and intercalate the electrochemical cell with $TiO_2$/Viologen-$TiO_2$ sample so that laser beam shines the film.
With the power source modify the potential until the functionalized $TiO_2$ becomes dark blue (approx -2 to -3 V). Take the values of voltage at each 200 mV together with current of the electrochemical cell in stationary conditions and the photocurrent in the diode. Do the same with the bare TiO2 electrode. Rise the potential until it becomes dark blue (aprox. -3 to 4 V)
Measure the surface (S) of the $TiO_2$ film and normalize the current to obtain current density (J=i/S).
Calculate the transmittance through the expression: $T= i_{photo} / i_{photo,\,0}$
where $i_{photo}$ is the current at the photodiode at any of the potentials and $i_{photo,\,0}$ is the photocurrent at initial potential where the $TiO_2$/Viologen-$TiO_2$ sample is still transparent.

**Results**
Make the plot of J-V curve.
Make the plot of photocurrent- voltage and transmittance voltage.

**References:** [1]
1. García-Cañadas, J.; Fabregat-Santiago, F.; Kapla, J.; Bisquert, J.; Garcia-Belmonte, G.; Mora-Seró, I.; Edwards, M. O. M. Dynamic behaviour of viologen-activated nanostructured TiO2: Correlation between kinetics of charging and colouration. *Electrochimica Acta* **2004,** *49*, 745-752.