

**MISSION
INNOVATION**

accelerating the clean energy revolution

POA MATERIALI AVANZATI PER L'ENERGIA**PROGETTO IEMAP - Piattaforma Italiana Accelerata per i Materiali per
l'Energia**

D4.28 - Database con le proprietà delle interfacce degli elettrodi sviluppati

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Consiglio Nazionale
delle Ricerche

D4.28 - DATABASE CON LE PROPRIETÀ DELLE INTERFACCE DEGLI ELETTRODI SVILUPPATI

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Report MISSION INNOVATION

Ministero dell'Ambiente e della Sicurezza Energetica - ENEA
Mission Innovation 2021-2024 - III annualità

Progetto: Piattaforma accelerata per i Materiali per l'Energia
Work package: WP4 – Materiali per Fotovoltaico
Linea di attività LA4.12 Realizzazione di dispositivi integrati fotovoltaico-accumulo a 2 terminali
Responsabile del Progetto: Massimo Celino (ENEA)
Responsabile della LA: Dr.ssa Alessandra Sanson (ISSMC-CNR)

WO₃IJP-PEDOT/MIP asymmetric integrated photovoltaic/storage device with gel state LiClO₄ electrolyte

SUMMARY

Interfaces



FILM DEPOSITION ANDE DEVICE ASSEMBLY

Reagents

Substrate: Fluorine doped tin oxide coated glass (FTO, Sigma Aldrich, surface resistivity 7 Ω/sq.) with dimension of 2.5 x 2.5 cm².

Screen printing WO₃-based ink, containing active material between 15 and 20 wt.%, produced by using α-terpineol and cellulose derivatives (Sigma Aldrich).

Inkjet printing ink formulated starting from the Avantama P-10 commercial suspension and using additives, with viscosity and surface tension equal to 6.50 mPa*s and 24.59 mN/m respectively.

3,4 Ethylenedioxythiophene (EDOT, 97% Sigma Aldrich), LiClO₄ (ACS Reagent, Sigma Aldrich), water with MQ grade, oxalic acid di-hydrate (>99.5%, Merck), water MQ grade.

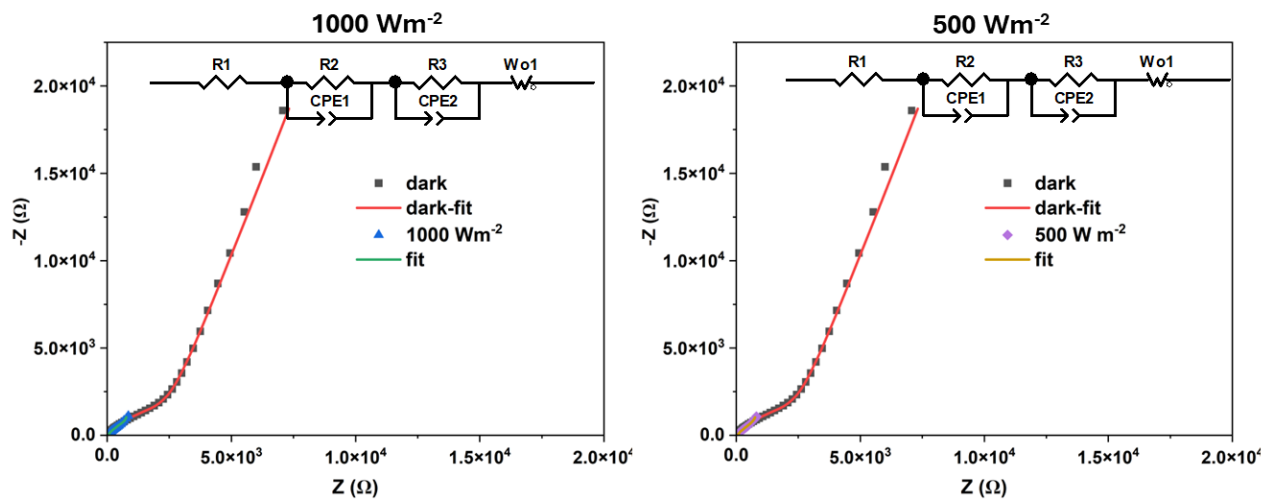
LiClO₄ salt (ACS Reagent, Sigma-Aldrich) 0.1M water MQ solution and poly-vinyl alcohol (PVA) 10% wt.

Procedure

Thick films based on WO₃ were prepared by semi-automatic screen-printing machine, AUREL 900 (Aurel Automation S.p.A., Italia), with speed of 45 mm/s and ten consecutive depositions. A mean thickness close to 22 μm and an active area of 0.25 cm² were achieved. Between each deposition, a drying treatment in IR oven at 80°C was applied. Finally, the thermal consolidation of the films was obtained by treating them at 450°C for 30'. Inkjet printing decoration was performed on the previous films, by using a multiple-deposition techniques station (XCEL, Aurel Automation s.p.a., Italy) equipped with a drop-on-demand inkjet printing head, MD-K-140 (microdrop Technologies GmbH, Germany) that has the possibility to heat up the nozzle. A specific pattern, a wavy line, was realized by printing at 20 mm/s and heating up the nozzle at 45°C. The film drying was realized at 95°C for 90'' on a hot plate while the final consolidation was obtained by treating the samples at 120°C for 60' in a common oven with an oxidative atmosphere. For supporting-electrode preparation, Poly-3,4 Ethylenedioxythiophene (PEDOT) MIP film was deposited by electro-polymerization in a three electrodes cells (working: FTO, reference: SCE, counter: platinum foil) using 3,4 Ethylenedioxythiophene 5 mM in LiClO₄ 0.5M in water MQ and 25 mM of oxalic acid. Applied potential equal to +1.05 V vs SCE with a total amount of charge equal to 0.1 C, active area deposited equal to 0.25 cm². The final device was completed adding PVA gel between two electrodes and sealed with a thermoplastic sealant, epoxy glue and protective UV-filter.

INTERFACE PROPERTIES

Electrochemical Impedance Spectroscopy in frequency range between 1×10^5 Hz e 0.01 Hz with signal amplitude of 10 mV and potential applied equal to 0 V in the dark and under illumination with different intensities (1000 W m^{-2} or 500 W m^{-2} calibrated with a reference cell).



Interfaces Properties

- Electrical resistance under 1000 W m^{-2} of 14Ω .
- Electrical resistance under 500 W m^{-2} of 14Ω .
- Charge transfer resistances at the electrode/electrolyte interfaces under 1000 W m^{-2} illumination equal to 30Ω (R2) and 1440Ω (R3).
- Charge transfer resistances at the electrode/electrolyte interfaces under 500 W m^{-2} illumination equal to 45Ω (R2) and 1404Ω (R3).

WO₃IJP-rGO asymmetric integrated photovoltaic/storage device with water based LiClO₄ electrolyte

SUMMARY

Interfaces



WO₃ AND PEDOT-OA FILM DEPOSITION AND DEVICE ASSEMBLY

Reagents

Substrate: Fluorine doped tin oxide coated glass (FTO, Sigma Aldrich, surface resistivity 7 Ω/sq.) with dimension of 2.5 × 2.5 cm².

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Screen printing WO₃-based ink, containing active material between 15 and 20 wt.%, produced by using α-terpineol and cellulose derivatives (Sigma Aldrich).

Inkjet printing ink formulated starting from the Avantama P-10 commercial suspension and using additives, with viscosity and surface tension equal to 6.50 mPa*s and 24.59 mN/m respectively.

Graphene oxide suspension (GO, 4 mg/mL in water, Sigma Aldrich), Nafion (Nafion[®] 5 wt % in alcohol and water), Na₂SO₄ (ACS Reagent ≥99.5%, Sigma-Aldrich), water MQ grade.

LiClO₄ salt (ACS Reagent, Sigma-Aldrich) 0.1 M water MQ solution.

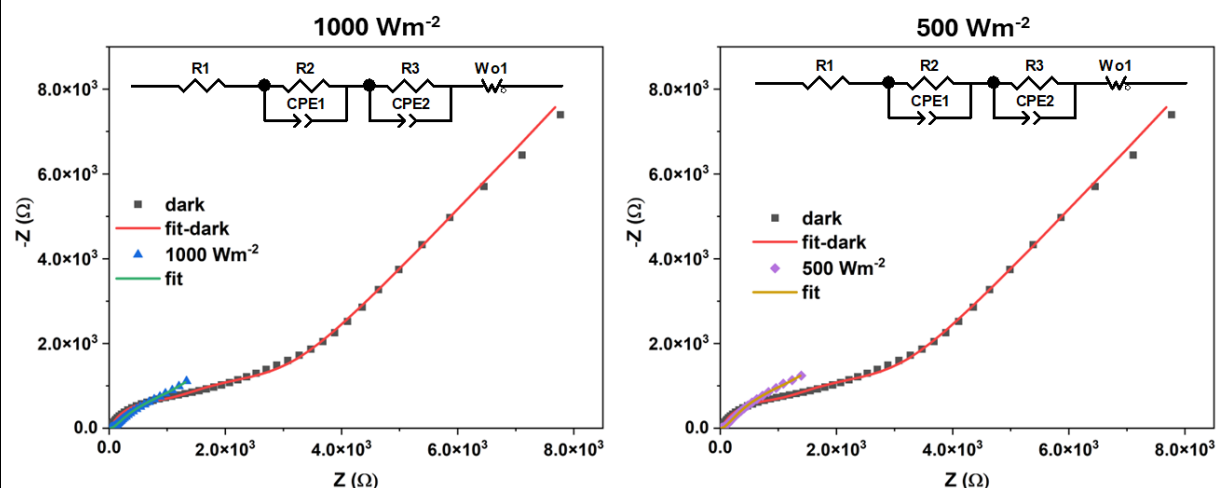
Procedure

Thick films based on WO₃ were prepared by semi-automatic screen-printing machine, AUREL 900 (Aurel Automation S.p.A., Italia), with speed of 45 mm/s and ten consecutive depositions. A mean thickness close to 22 μm and an active area of 0.25 cm² were achieved. Between each deposition, a drying treatment in IR oven at 80°C was applied. Finally, the thermal consolidation of the films was obtained by treating them at 450°C for 30'. Inkjet printing decoration was performed on the previous films, by using a multiple-deposition techniques station (XCEL, Aurel Automation s.p.a., Italy) equipped with a drop-on-demand inkjet printing head, MD-K-140 (microdrop Technologies GmbH, Germany) that has the possibility to heat up the nozzle. A specific pattern, a wavy line, was realized by printing at 20 mm/s and heating up the nozzle at 45°C. The film drying was realized at 95°C for 90'' on a hot plate while the final consolidation was obtained by treating the samples at 120°C for 60' in a common oven with an oxidative atmosphere.

. For supporting-electrode preparation, an ink formulation based on GO 83% vol. e 17 % vol. Nafion was prepared and deposited by drop by drop deposition (150 μL) on FTO substrate. After that, this film was reduced electrochemically in a three electrodes cells (working: FTO, reference: SCE, counter: platinum foil) using Na₂SO₄ 0.1 M water solution using cyclic voltammetry method. The potential range was set between +0.750V and -1.4 V vs SCE with a scan rate of 50 mV sec⁻¹ for 50 cycles. Active area deposited was equal to 0.25 cm². The final device was completed adding liquid electrolyte between two electrodes (by holes on supporting electrode) and sealed with a thermoplastic sealant, epoxy glue.

INTERFACE PROPERTIES

Electrochemical Impedance Spectroscopy in frequency range between 1×10^5 Hz e 0.01 Hz with signal amplitude of 10 mV and potential applied equal to 0 V in the dark and under illumination with different intensities (1000 W m^{-2} or 500 W m^{-2} calibrated with a reference cell).



Interfaces Properties

- Electrical resistance under 1000 W m^{-2} of $20 \text{ } \Omega$.
- Electrical resistance under 500 W m^{-2} of $13 \text{ } \Omega$.
- Charge transfer resistances at the electrode/electrolyte interfaces at 1000 W m^{-2} equal to $33 \text{ } \Omega$ (R2) and $333 \text{ } \Omega$ (R3).
- Charge transfer resistances at the electrode/electrolyte interfaces under 500 W m^{-2} illumination equal to $38 \text{ } \Omega$ (R2) and $440 \text{ } \Omega$ (R3).

WO₃IJP-rGO asymmetric integrated photovoltaic/storage device with acetonitrile based LiClO₄ electrolyte

SUMMARY

Interfaces



WO₃ AND PEDOT-OA FILM DEPOSITION AND DEVICE ASSEMBLY

Reagents

Substrate: Fluorine doped tin oxide coated glass (FTO, Sigma Aldrich, surface resistivity 7 Ω/sq.) with dimension of 2.5 × 2.5 cm².

Substrate: Fluorine doped tin oxide coated glass (FTO, Sigma Aldrich, surface resistivity 7 Ω/sq.) with dimension of 2.5x2.5 cm².

Screen printing WO₃-based ink, containing active material between 15 and 20 wt.%, produced by using α-terpineol and cellulose derivatives (Sigma Aldrich).

Inkjet printing ink formulated starting from the Avantama P-10 commercial suspension and using additives, with viscosity and surface tension equal to 6.50 mPa*s and 24.59 mN/m respectively.

Graphene oxide suspension (GO, 4 mg/mL in water, Sigma Aldrich), Nafion (Nafion[®] 5 wt % in alcohol and water), Na₂SO₄ (ACS Reagent ≥99.5%, Sigma-Aldrich), water MQ grade.

LiClO₄ salt (ACS Reagent, Sigma-Aldrich) 0.1M acetonitrile-based solution

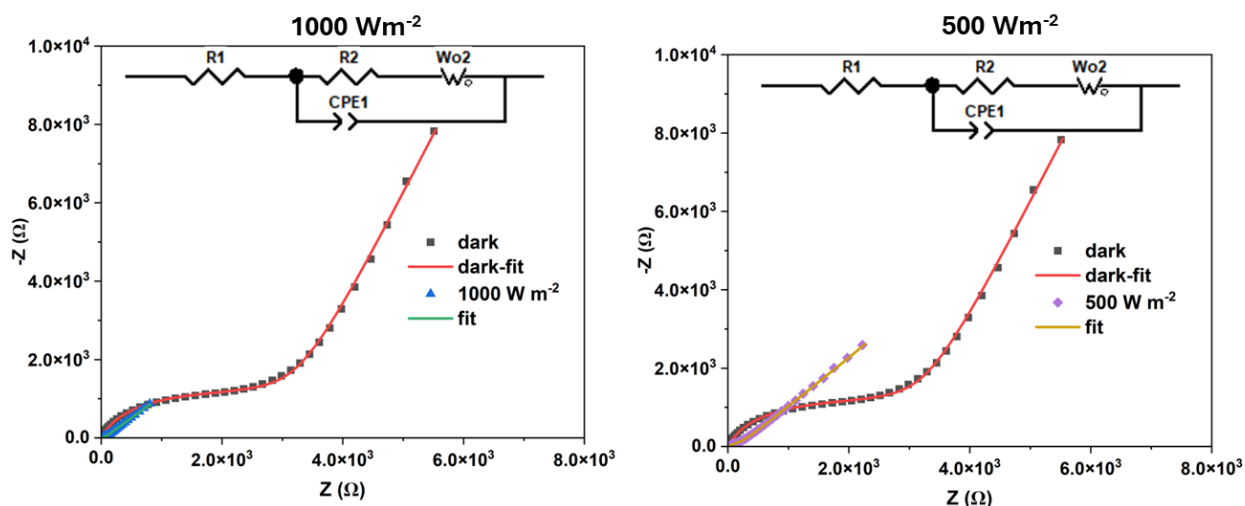
Procedure

Thick films based on WO₃ were prepared by semi-automatic screen-printing machine, AUREL 900 (Aurel Automation S.p.A., Italia), with speed of 45 mm/s and ten consecutive depositions. A mean thickness close to 22 μm and an active area of 0.25 cm² were achieved. Between each deposition, a drying treatment in IR oven at 80°C was applied. Finally, the thermal consolidation of the films was obtained by treating them at 450°C for 30'. Inkjet printing decoration was performed on the previous films, by using a multiple-deposition techniques station (XCEL, Aurel Automation s.p.a., Italy) equipped with a drop-on-demand inkjet printing head, MD-K-140 (microdrop Technologies GmbH, Germany) that has the possibility to heat up the nozzle. A specific pattern, a wavy line, was realized by printing at 20 mm/s and heating up the nozzle at 45°C. The film drying was realized at 95°C for 90'' on a hot plate while the final consolidation was obtained by treating the samples at 120°C for 60' in a common oven with an oxidative atmosphere.

For supporting-electrode preparation, an ink formulation based on GO 83% vol. e 17 % vol. Nafion was prepared and deposited by drop-by- drop deposition (150 μL) on FTO substrate. After that, this film was reduced electrochemically in a three electrode cells (working: FTO, reference: SCE, counter: platinum foil) using Na₂SO₄ 0.1 M water solution using cyclic voltammetry method. The potential range was set between +0.750 V and -1.4 V vs SCE with a scan rate of 50 mV s⁻¹ for 50 cycles. Active area deposited was equal to 0.25 cm². The final device was completed adding liquid electrolyte between two electrodes (by holes on supporting electrode) and sealed with a thermoplastic sealant, epoxy glue.

INTERFACE PROPERTIES

Electrochemical Impedance Spectroscopy in frequency range between 1×10^5 Hz e 0.01 Hz with signal amplitude of 10 mV and potential applied equal to 0V in the dark and under illumination with different intensities (1000 W m^{-2} or 500 W m^{-2} calibrated with a reference cell).



Interfaces Properties

- Electrical resistance under 1000 W m^{-2} of $11 \text{ } \Omega$.
- Electrical resistance under 500 W m^{-2} of $17 \text{ } \Omega$.
- Charge transfer resistance at the electrode/electrolyte interface under 1000 W m^{-2} illumination equal to $274 \text{ } \Omega$.
- Charge transfer resistance at the electrode/electrolyte interface under 500 W m^{-2} illumination equal to $305 \text{ } \Omega$.

TiO₂IJP-N3-PEDOT-PEDOT/MIP asymmetric integrated photovoltaic/storage device with gel state LiClO₄ electrolyte

SUMMARY

Interfaces



FILM DEPOSITION ANDE DEVICE ASSEMBLY

Reagents

Substrate: Fluorine doped tin oxide coated glass (FTO, Sigma Aldrich, surface resistivity 7 Ω /sq.) with dimension of 2.5 x 2.5 cm².

Screen printing TiO₂-based ink 18NR-T (Greatcell Solar Materials), with viscosity between 40-55 Pa*s.

Inkjet printing ink formulated starting from 18NR-T paste and including some additives, with viscosity and surface tension equal to 37.66 mPa*s and 32.55 mN/m respectively.

Dye N3 (*cis*-Bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II), Sigma Aldrich) 0.3 mM in Absolute Ethanol.

3,4 Ethylenedioxythiophene (EDOT, 97% Sigma Aldrich), LiClO₄ (ACS Reagent, Sigma Aldrich), water with MQ grade, oxalic acid di-hydrate (>99.5%, Merck), water MQ grade.

0.1M water MQ solution and poly-vinyl alcohol (PVA) 10% wt.

Procedure

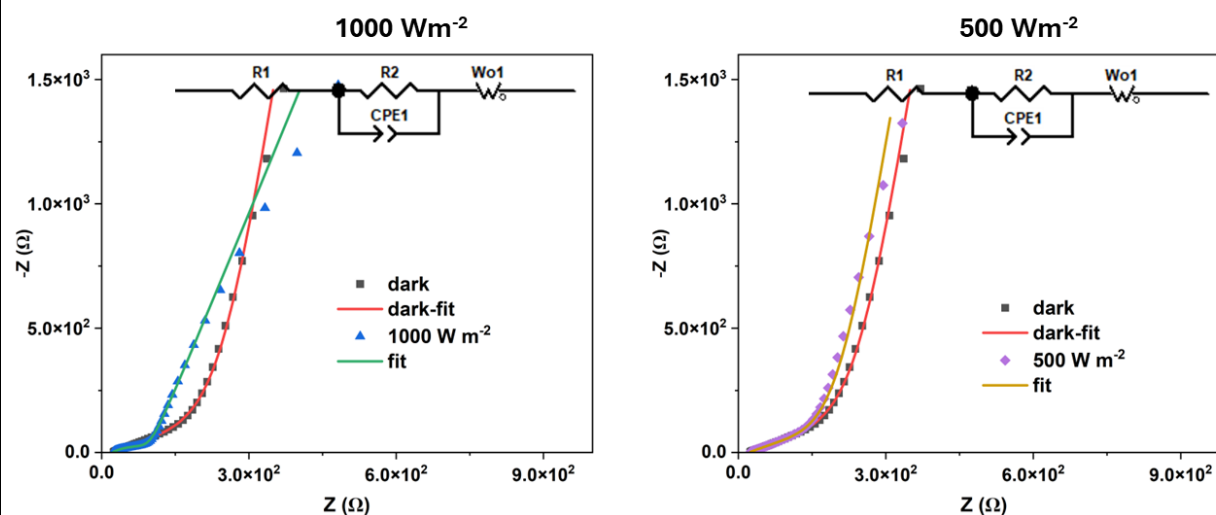
Thick films based on TiO₂ were prepared by semi-automatic screen-printing machine, AUREL 900 (Aurel Automation S.p.A., Italia), with speed of 90 mm/s and three consecutive depositions. A mean thickness of 11.84 μ m and an active area of 0.25 cm² were achieved. Between each deposition, a drying treatment in IR oven at 80°C was applied. Finally, the thermal consolidation of the films was obtained by treating them at 450°C for 30'.

Inkjet printing was performed on the previous films, by using a multiple-deposition techniques station (XCEL, Aurel Automation s.p.a., Italy) equipped with a drop-on-demand inkjet printing head, MD-K-140 (microdrop Technologies GmbH, Germany) that has the possibility to heat up the nozzle. A specific pattern, a wavy line, was realized by printing at 40 mm/s and heating up the nozzle at 35°C. The film drying was realized at 85°C for 60 seconds on a hot plate while the final consolidation was obtained treating the samples at 450°C for 30' in a common oven.

The as obtained film was sensitized overnight in 0.3 mM N3 dye solution and the excess of dye was removed by absolute ethanol. On top of this film, Poly-3,4 Ethylenedioxythiophene (PEDOT) film was deposited by electro-polymerization in a three electrodes cells (working: FTO, reference: SCE, counter: platinum foil) using 3,4 Ethylenedioxythiophene 5 mM in LiClO₄ 0.5M in water MQ with applied potential equal to +1.05 V vs SCE and with a total amount of charge equal to 0.1 C. For supporting-electrode preparation, Poly-3,4 Ethylenedioxythiophene (PEDOT) MIP film was deposited by electro-polymerization in a three electrodes cells (working: FTO, reference: SCE, counter: platinum foil) using 3,4 Ethylenedioxythiophene 5 mM in LiClO₄ 0.5M in water MQ and 25 mM of oxalic acid. Applied potential equal to +1.05 V vs SCE with a total amount of charge equal to 0.1 C, active area deposited equal to 0.25 cm². The final device was completed adding PVA gel between two electrodes and sealed with a thermoplastic sealant, epoxy glue and protective UV-filter.

INTERFACE PROPERTIES

Electrochemical Impedance Spectroscopy in frequency range between 1×10^5 Hz e 0.01 Hz with signal amplitude of 10 mV and potential applied equal to 0V in the dark and under illumination with different intensities (1000 W m^{-2} or 500 W m^{-2} calibrated with a reference cell).



Interfaces Properties

- Electrical resistance under 1000 W m^{-2} of 22Ω .
- Electrical resistance under 500 W m^{-2} of 22Ω .
- Charge transfer resistance at the electrode/electrolyte interface under 1000 W m^{-2} illumination equal to 76Ω .
- Charge transfer resistance electrode/electrolyte interface at 500 W m^{-2} equal to 224Ω .

TiO₂IJP-AD418-PEDOT-PEDOT/MIP asymmetric integrated photovoltaic/storage device with gel state LiClO₄ electrolyte

SUMMARY

Interfaces



FILM DEPOSITION ANDE DEVICE ASSEMBLY

Reagents

Substrate: Fluorine doped tin oxide coated glass (FTO, Sigma Aldrich, surface resistivity 7 Ω /sq.) with dimension of 2.5x2.5 cm².

Screen printing TiO₂-based ink 18NR-T (Greatcell Solar Materials), with viscosity between 40-55 Pa*s.

Inkjet printing ink formulated starting from 18NR-T paste and including some additives, with viscosity and surface tension equal to 37.66 mPa*s and 32.55 mN/m respectively.

Dye AD418 (molecular formula C₄₃H₄₄N₂O₄S₂) 0.3 mM in Ethanol:THF.

3,4 Ethylenedioxythiophene (EDOT, 97% Sigma Aldrich), LiClO₄ (ACS Reagent, Sigma Aldrich), water with MQ grade, oxalic acid di-hydrate (>99.5%, Merck), water MQ grade.

0.1M water MQ solution and poly-vinyl alcohol (PVA) 10% wt.

Procedure

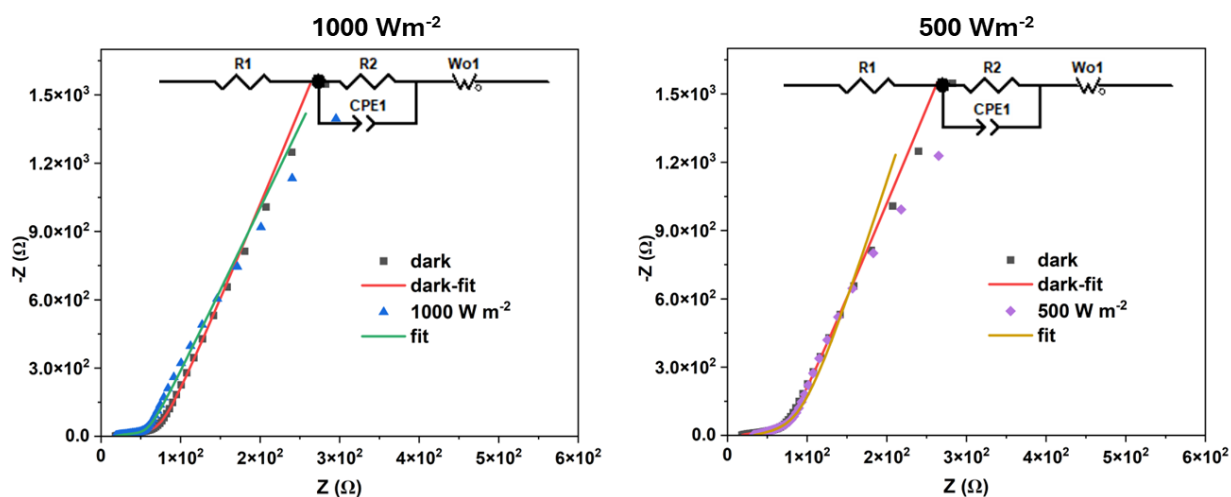
Thick films based on TiO₂ were prepared by semi-automatic screen-printing machine, AUREL 900 (Aurel Automation S.p.A., Italia), with speed of 90 mm/s and three consecutive depositions. A mean thickness of 11.84 μ m and an active area of 0.25 cm² were achieved. Between each deposition, a drying treatment in IR oven at 80°C was applied. Finally, the thermal consolidation of the films was obtained by treating them at 450°C for 30'.

Inkjet printing was performed on the previous films, by using a multiple-deposition techniques station (XCEL, Aurel Automation s.p.a., Italy) equipped with a drop-on-demand inkjet printing head, MD-K-140 (microdrop Technologies GmbH, Germany) that has the possibility to heat up the nozzle. A specific pattern, a wavy line, was realized by printing at 40 mm/s and heating up the nozzle at 35°C. The film drying was realized at 85°C for 60 seconds on a hot plate while the final consolidation was obtained treating the samples at 450°C for 30' in a common oven.

The as obtained film was sensitized overnight in 0.3 mM AD418 dye solution and the excess of dye was removed by absolute ethanol. On top of this film, Poly-3,4 Ethylenedioxythiophene (PEDOT) film was deposited by electro-polymerization in a three electrodes cells (working: FTO, reference: SCE, counter: platinum foil) using 3,4-Ethylenedioxythiophene 5 mM in LiClO₄ 0.5M in water MQ with applied potential equal to +1.05 V vs SCE and with a total amount of charge equal to 0.1 C. For supporting-electrode preparation, Poly-3,4-Ethylenedioxythiophene (PEDOT) MIP film was deposited by electro-polymerization in a three electrodes cells (working: FTO, reference: SCE, counter: platinum foil) using 3,4 Ethylenedioxythiophene 5 mM in LiClO₄ 0.5M in water MQ and 25 mM of oxalic acid. Applied potential equal to +1.05 V vs SCE with a total amount of charge equal to 0.1 C, active area deposited equal to 0.25 cm². The final device was completed adding PVA gel between two electrodes and sealed with a thermoplastic sealant, epoxy glue and protective UV-filter.

INTERFACE PROPERTIES

Electrochemical Impedance Spectroscopy in frequency range between 1×10^5 Hz e 0.01 Hz with signal amplitude of 10 mV and potential applied equal to 0V in the dark and under illumination with different intensities (1000 W m^{-2} or 500 W m^{-2} calibrated with a reference cell).



Interfaces Properties

- Electrical resistance under 1000 W m^{-2} of 17Ω .
- Electrical resistance under 500 W m^{-2} of 33Ω .
- Charge transfer resistance at the electrode/electrolyte interface under 1000 W m^{-2} illumination equal to 47Ω .
- Charge transfer resistance at the electrode/electrolyte interface under 500 W m^{-2} illumination equal to 77Ω .