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# **Plenary conferences**

# CHALLENGES FOR A 21<sup>st</sup> CENTURY'S ELECTROCHEMISTRY

## Invited Speaker 1:



Prof. Farouk TEDJAR

Energy Research Institute, Nanyang Technological University of Singapore

### Abstract:

Today world population increase and related economy expands are accompanied with important increase of substantial need for natural resources. Consequently, in order to sustain same level of economic activities a great amount of energy and resource are consumed [1]. Within all resources and just after water and energy we have the metals in third position. Energy issues and climate changes are now asking urgent solution and one of the solutions is large growing of renewable energy part in the consumption models facing a demonstrated scarcity and important potential sources of negative environment impact in other side. [2]. In addition to those environment impacts, the sector of strategic and critical metals is now entering in sustainability problems due to scarcity of some metals. In the meantime, accurate assessment of mineral resources must include not only the resources available in the ground as ores but also those that become available as waste and recovered through recycling. This means that continuation of metal production/ supply is closely linked to access to secondary sources to enhance resource conservations. This is why the concept of “*Urban Mine*” was introduced [3]. Acceptability of the new segment of metal production, need to comply with a better sustainability and in particular for the impact on climate change and resource conservation. However to continue in direction of real sustainability, production of materials from primary as well as secondary sources (end-of-life equipment), involved processes need to address the following challenges:

- ✓ Lowest energy consumption ;
- ✓ Low water consumption and waste water release;
- ✓ Low CO<sub>2</sub> emission (as well as greenhouse gases).

In particular, access to “Urban Ores” can also prevent the Geopolitical consideration in terms of availability of some strategic /critical metals owned by few countries for more than 50% of known resources/deposits [4]. In almost all those segments electrochemistry plays a central role in:

- ✓ Extraction and production of raw materials mainly by electro-winning;
- ✓ Construction of equipment for electrochemical storage;
- ✓ Recycling end-of-life equipment.

Nevertheless, traditional electrochemistry presents several dark sides either by hazardous materials such as:

- ✓ Mercury for batteries and cathode in electro-winning processes;
- ✓ Cadmium, lead and cobalt as electrode materials (all 3 highly toxic);
- ✓ Chlorine and fluorine evolution from some production processes.

Today answers are expected on behalf of the community of electrochemists to arise a new electrochemistry of 21<sup>th</sup> century. Important programs were launched early of 1995 and some examples of representative studies will be presented [8-12].

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[4]: US Department of Interior, US Geological Survey, Global Flows of Metals (September 2017).

[5]: European Patent EP0510239B1, Hiroshi Yoshizawa, Panasonic Corp.

[6]: US patent 3824131A, "Negative electrode nickel alloy hydride phases".

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[11]: M. Gras, Recycling rare earths using ionic liquid, PhD Thesis, Grenoble October 2018

[12]: M. Gras, E. Chainet, F. Tedjar, Separation of Rare Earths in ionic liquids, Separation Technologies 178 (2017) 169-177.

# PLASMONIC HOT-ELECTRON SOLAR CELLS AND PHOTO-CATALYSIS

## Invited Speaker 2:



Prof. Jean-Michel NUNZI

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### Abstract:

There currently exists a significant demand for IR broadband photoresponsive devices for applications ranging from photovoltaics and renewable energy to photodetection for military and civilian purposes. When considering the effectiveness of those photosensitive devices, several factors must be considered including photoresponsivity, fabrication process, and cost. Moreover, the spatial resolution of IR photodetectors can be significantly improved by simultaneously sensing the intensity and polarization of the incident light [1].

Photodetection through conventional procedures is based on light absorption by a material with a matching bandgap. However, this approach limits the range of wavelengths that can be detected, it is not sensitive to polarization, and cannot be used accurately in the infrared range because of thermal noise [2]. Recent approaches have attempted to circumvent these limitations, optical rectification is one of them [3].

Metal–semiconductor Schottky junctions have been reported as the most efficient structures to collect hot electrons [4] and generate a signal in photodetectors. However, previously reported photodetectors based on this methodology can be very costly to fabricate and are not suitable for large-scale fabrication. Herein, we demonstrate that ITO-Au nanostructures can indeed be used to fabricate a NIR photodetector [5] using the rectification effect induced by dipole orientation in a thin-film [6].

We review optical rectification principles and experiments and report on a hot electron-based photodetector device that is sensitive in the NIR range, sensitive to

polarization, as well as easy and cost-effective to fabricate. The approach developed herein represents a significant milestone towards the development of energy conversion devices based on hot electrons and plasmonics, which could be beneficial in integrated optoelectronics. The concept can be extended to photocatalysis in which specific photo-electrochemical reactions can be triggered by hot-electron ejection [7].

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# **ELECTROCHEMICAL MODIFICATION OF SURFACES: ORGANOPHOSPHONIC ACIDS GRAFTING ON NITINOL**

## **Invited Speaker 3:**



Prof. Zineb MEKHALIF

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### **Abstract:**

Modification of surfaces with thin or thick layers, organic, inorganic or hybrids is nowadays an elegant and efficient approach to provide new functionalities to materials making them attractive in many domains of applications.

Spontaneous adsorption on metallic and metal oxides surfaces of molecules based respectively on organothiols and organophosphonics has been studied for years to elaborate self-assembled monolayers and have been shown to confer to the modified surfaces improved or new properties such as corrosion protection, lubrication, controlled wetting properties, *etc.* The interface properties as well as the organization of the molecules have a direct impact on the macroscopic induced properties.

Electrochemical modifications of surfaces through oxidation or reduction are largely applied to elaborate organic monolayers, multilayers or polymers on surfaces. An important approach is the electrochemical reduction of aryl dicationic molecules efficient to modify metals and oxides. More recently, the electro-assisted adsorption of molecules such as organothiols and organophosphonic acids has shown important promises in the field of surface modifications.

In this presentation, the focus will be put on the electro-assisted modification of nitinol, a nearly equiatomic NiTi alloy, with organophosphonic acid derivatives.



# LA TECHNIQUE DE L'ELECTROCHIMIE ENTRE LA SYNTHESE ET LA CARACTERISATION DES SYSTEMES

**Invited Speaker 4:**



Prof. Radhouane CHTOUROU

## **Abstract :**

Au cours de cette présentation, nous allons parler de la technique électrochimie entre la synthèse des couches minces, des nanostructures et aussi des techniques de mesure de photo-électrochimie, des mesures photovoltaïques et les mesures d'impédance pour des systèmes en régime de fonctionnement. Cette présentation sera appuyée par des exemples de mesures réels sur différents types de système.

# **Oral presentations**

## Topic 1: Batteries and Energy storage.

[T1-OP 01]

### IMPROVEMENT OF BATTERY/FUEL CELL HYBRID SYSTEM LIFE TIME USING OPTIMAL SIZING AND ENERGY MANAGEMENT STRATEGY FOR AUTOMOTIVE APPLICATION

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#### Abstract:

Electric vehicles (EVs) have particular limitations which make them less challenger to thermal ones and handicap their commercialization. The main problems as the autonomy, the robustness and the cost depend directly on the energy storage problematic issues.

In this context, this paper deals with the improvement of battery/fuel cell hybrid system life time using optimal sizing and energy management strategy. It focuses on optimal sizing and energy management strategy for an Energy Storage System (ESS) composed by a Fuel Cell (FC) as main source and lithium ion batteries as an assistant one to supply a light weight vehicle.

The hybrid source life time is studied as an energy storage system problematic. Due to difficulties in evaluating the battery, and fuel cell lifetime, some key parameters are selected to evaluate each source aging. The FC stack degradation can be determined mainly by current dynamics, coolant temperature, membrane humidity and idle current as main stress factors of the fuel cell stack aging [1]. It was concluded that the current (or power) dynamics have a major influence. The Root Mean Square (RMS) power is used in this work to know how often fuel cell is solicited. Several parameters as temperature, DOD, the State Of Charge (SOC), cycles number and current dynamics are stress factors for the battery aging [1]. In this work, it is assumed that temperature and DOD are fixed. Only the Root Mean Square (RMS) power (image of current dynamics) and absolute energy are used to know how often batteries are solicited.

From the simulation results, the sizing of the energy storage system is affected directly by the choice of the battery's technology and the energy management strategy. Otherwise, the whole system life time is affected by the used technologies and the energy management strategies too. It is noted that the lithium Ion UHP technology provides the optimum weight, volume cost. In addition, it guarantees a longer life time of the fuel cell system due to its high power density.

**Keywords:** Battery, Electric vehicles, Fuel Cell, life time

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[T1-OP 02]

**EFFET DU BISMUTH SUR LA CAPACITE DE DECHARGE DE LA  
BATTERIE AU PLOMB.**

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**Résumé :**

La masse du bioxyde de Plomb actif des plaques de batterie Pb-acide est un système gel-cristal avec une conductivité d'électrons et de protons des zones de gel hydraté.

Cette étude tente de discuter l'influence du Bismuth, sur la stœchiométrie, la structure et la composition de la phase du matériau actif positif (PAM) du PbO<sub>2</sub> des batteries Pb-acide.

Les échantillons de PAM dopés ont été caractérisés par diffraction des rayons X (DRX), analyse thermique gravimétrique (ATG).

Les résultats de la diffraction aux rayons X ont montré que l'ajout du Bismuth n'influe pas sur la phase de notre PbO<sub>2</sub>, mais modifie la taille de ses particules.

Les analyses thermiques ont été réalisées dans la zone de températures comprise entre 25 et 370°C qui correspond à la plage de température de déshydratation de la partie gel du bioxyde de plomb.

Les pertes en poids mesurées en fonction de la température de chauffe ont montré que le dopage de PbO<sub>2</sub> modifie complètement la disposition des entités hydratées donnant ce qui influe sur la quantité d'eau présente dans les zones du gel de la masse active positive de la batterie au plomb.

L'étude électrochimique par voltamétrie cyclique et décharge intensiostatique montre clairement l'effet positif de l'ajout du Bismuth, sur la capacité de décharge de l'accumulateur au plomb.

**Mots clés:** PbO<sub>2</sub>, Bismuth, stockage d'énergie.

[T1-OP 03]

**POLYSTYRENE FUNCTIONALIZED BY PENTADENTATE NICKEL  
(II)-SCHIFF BASE COMPLEX AS BIFUNCTIONAL MATERIAL FOR  
ELECTROCATALYTIC WATER SPLITTING AND METHANOL  
OXIDATION.**

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**Abstract:**

Hydrogen present a clean source of energy with gravimetric energy density of 120-142 MJ/kg, it is one of most important chemicals for chemical industry. Water presents the most abundant source of hydrogen, but producing hydrogen from water suffering from important overpotential at the cathode and anode. Several researches were carried out to solve this problem and important results were obtained [1,2].

In the present work we focus on the anode material development to reduce overpotential toward water oxidation reaction or by using methanol as sacrificial electron donor, Polystyrene functionalized by a Schiff base ligand as N,N'-bis(salicylidenepropylenetriamine)-N-para-aminomethylpolystyrene was synthesized from poly(4-chloromethylstyrene) and Schiff base ligand N, N'-bisalicylidenepropylenetriamine. Coordinated with nickel ion, this polystyrene Schiff base gives the expected nickel complex. The modified electrodes obtained from graphite paste and nickel complex covalently grafted onto the polystyrene were prepared in a ratio 70:30 wt%. Its voltammogram, recorded in alkaline solution shows bifunctional behavior toward oxygen evolution reaction and methanol oxidation reaction in alkaline medium. At low scan rate of 1 mVs<sup>-1</sup> and in absence of methanol, oxygen evolution reaction (OER) takes place around 1550mv Vs RHE. In presence of methanol a new catalytic peak appear before the OER and increase linearly with methanol concentration with a catalytic peak current of 450µA at concentration of 1M methanol.

**Keywords:**

Polystyrene-Ni(II)-Schiff base complex MGPE, Cyclic voltammetry. OER, MOR.

**References**

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[T1-OP 04]

Ni-Co HYDROXIDES AND CARBON NANOTUBES ELECTROCHEMICAL  
MEASUREMENTS FOR ENERGY STORAGE « SUPERCAPACITORS »

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**Abstract:** In this study, a facile and low cost free-template hydrothermal precipitation method was used to synthesize mesoporous Ni-Co based bimetallic carbonates (CO) and/or hydroxides (OH) micro/nanostructures with different morphologies (1D, 2D and 3D) based on variant stoichiometric compositions.

The effect of the growth temperature, synthesis time as well as the Ni/Co-precursors ratio on the physico-chemical properties and faradic electrochemical behavior of these products was investigated. The as obtained mono- and bi-phase nanohybrids were characterized extensively structurally and morphologically.

The textural analysis results confirmed the presence of mesoporous products with a  $\sim 50 \text{ m}^2 \cdot \text{g}^{-1}$  BET-SSA ( $0.52 \text{ cm}^3 \cdot \text{g}^{-1}$  pore volume) for the 3D urchin-like structure and a  $\sim 47.14 \text{ m}^2 \cdot \text{g}^{-1}$  BET-SSA ( $0.31 \text{ cm}^3 \cdot \text{g}^{-1}$  pore volume) was obtained for the 2D nanoflakes structure. The electrochemical measurements performed in a 6.0 M KOH aqueous electrolyte depicted excellent electrochemical performance ascribed to the optimized composition of NiCo LDH (or  $\alpha$ -Ni(OH) ) with Co (OH) Cl and their unique hierarchical mesoporous nanoflake and urchin-like architectures. In addition, an exceptionally notable specific capacitances (capacities) of  $1700 \text{ F} \cdot \text{g}^{-1}$  ( $161 \text{ mAh} \cdot \text{g}^{-1}$ ) and  $1379 \text{ F} \cdot \text{g}^{-1}$  ( $192 \text{ mAh} \cdot \text{g}^{-1}$ ) were obtained for both structures at  $5 \text{ mV} \cdot \text{s}^{-1}$  scan rate ( $0.5 \text{ A} \cdot \text{g}^{-1}$  gravimetric current density) respectively. These are much better than mono - hydroxides synthesized in same conditions with  $351 \text{ F} \cdot \text{g}^{-1}$  ( $90 \text{ mAh} \cdot \text{g}^{-1}$ ) for Ni and  $216 \text{ F} \cdot \text{g}^{-1}$  ( $21.5 \text{ mAh} \cdot \text{g}^{-1}$ ) for Co. A good cyclic stability of  $\sim 98 \%$  after 2000 charge-discharge cycles at  $30 \text{ A} \cdot \text{g}^{-1}$  was recorded depicting their potential as suitable materials for energy storage devices.

**Keywords:** Nanostructured materials, Specific surface area, Energy storage, Supercapacitor, Electrochemical.

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## Topic 2: Electrochemistry and Environment.

[T2-OP 05]

### OXYDATION PHOTOCATALYTIQUE D'UN COLORANT AZOÏQUE EN PRESENCE D'UN FILM DE MnO<sub>2</sub>

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#### Résumé :

Les colorants sont largement utilisés dans l'industrie textile, les tanneries, l'industrie de matière plastiques (pigments), l'industrie pharmaceutique (colorants), l'industrie agroalimentaire (colorants alimentaires), les industries de fabrication de pâtes et papiers l'industrie des cosmétiques et savonneries. Ainsi, l'industrie textile reste la plus grande consommatrice de colorants. Cependant, les colorants synthétiques sont appréciés principalement pour leur stabilité chimique et photolytique, pour la facilité de leur synthèse et pour leur variété de couleurs [1, 2].

Parmi les nombreuses familles de colorants synthétiques, les colorants azoïques sont les plus largement utilisés (de 60 à 70 %). Ces colorants sont très stables et relativement peu biodégradables. Des méthodes de dégradation des colorants azoïques sont développées au cours de ces dernières années [3].

Dans cette étude, nous avons synthétisé un colorant azoïque, le 1-phénylazo 2-naphtol (sudan 1), ensuite, nous avons suivi la photodégradation du colorant par photolyse et photocatalyse, comme catalyseur nous avons synthétisé par chronoampérométrie le dioxyde d'étain (MnO<sub>2</sub>). Le procédé de photodégradation sous une irradiation visible a été suivi par deux techniques différentes, la spectroscopie UV-Visible et la voltampéroétrie à onde carrée.

Les pourcentages des rendements de la photodégradation par les deux procédés ont montré que pour une concentration initiale en colorant de 10<sup>-4</sup> M, une décoloration de 35% avec le matériau photocatalytique MnO<sub>2</sub>, Contre seulement une décoloration de 9% dans le procédé de photolyse. Concernant la quantité adsorbé par le film de MnO<sub>2</sub> est dans l'ordre de 44 mg/g. Ce résultat a été obtenu après un temps d'équilibre de 120 min d'illumination visible.

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[T-2OP 06]

**INVESTIGATION D'ELIMINATION DE CETYLPYRIDINIUM  
BROMURE A PARTIR DES SOLUTIONS AQUEUSES SUR  
L'ALUMINE ET LE PHOSPHATE D'ALUMINIUM  
ELECTROGENEREES**

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**Résumé**

Le tensioactif, cétylpyridinium bromure a été étudié dans des cellules électrochimiques qui contiennent des solutions aqueuses de NaCl 0.1 M et dans d'autres solutions contenant des phosphates à pH neutre. Les électrolyses sont réalisées en utilisant des électrodes sacrificielles d'aluminium en mode potentiostatique 200 mA. Le pH, la turbidité et la conductivité de la solution ont été suivies durant l'électrolyse. La variation de la concentration du bromure de cétylpyridinium est contrôlée durant des périodes d'électrolyse par spectrophotométrie UV-Vis. Les solides électrogénérés tels que l'alumine, AlPO<sub>4</sub> en présence du cétylpyridinium ont été analysés par calcination, thermogravimétrie (TG), la désorption et la spectrophotométrie infrarouge (IR). L'utilisation des cathodes en aciers et en graphite ont montré l'élimination de cétylpyridinium (CP) dans un milieu de NaCl 0.1 M, cette constatation a été confirmée par une étude voltammétrique qui a montré une réduction de CP. Les résultats obtenus montrent que le bromure de cétylpyridinium est retenu par AlPO<sub>4</sub> pendant l'électrolyse et réduit sur les cathodes dans NaCl 0.1 M. Cette rétention peut être totale en cas de prolongement du temps d'électrolyse. Enfin, notons que ce processus de rétention a été seulement observé sur AlPO<sub>4</sub> et non pas sur l'alumine lorsque le bromure de cétylpyridinium est ajouté après électrolyse.

**Mots clés:** Cétylpyridinium, Electrocoagulation, Electrolyse, Aluminium





[T2-OP 07]

## DEGRADATION OF COLORING BASIC FUCHSIN BY THE ELECTRO-FENTON PROCESS

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### Abstract:

Basic fuchsine, a dye of triphenylmethane very useful as in the dyeing of textiles such as cotton, artificial fiber, leather, and paper ... etc. This pollutant exists in nature because of the fluid discharges of the industries, which can directly influence the living organisms and the environment.

Among the various methods to produce hydroxyl radicals, the electro-Fenton approach (which relies on the electrochemical production of  $H_2O_2$  via the reduction of dissolved  $O_2$  in the presence of  $Fe(II)$ ), stands out as one of the most promising approaches to develop advanced oxidation technologies

This work consists in eliminating basic fuchsine by the following advanced oxidation processes: electro-Fenton, and the effect of operating conditions such as  $Fe(2+)$  concentration, applied current, solution pH and temperature on the efficacy of the process was investigated.

The degradation of this substance was followed by spectrophotometry UV/Visible. The oxidation rate increased with increasing of current density and temperature and with decreasing of pH and the electro-Fenton process with 100% degradation rings in a very short time obtains the best result.

**Keywords:** Advanced oxidation processes, dye, electro-Fenton; Fenton, hydroxyl radicals, water treatment.

[T2-OP 08]

**DEVELOPMENT AND APPLICATION OF ELECTROCHEMICAL  
PROCESS IN WATER TREATMENT**

BOUTEIBA Ali

**Résumé :**

Ce travail concerne, l'élaboration des électrodes Pb/PbO<sub>2</sub>, et leur utilisation dans la dégradation de deux polluants à savoir le Cristal violet et le phénol par voie électrocatalytique. La caractérisation des électrodes obtenues par anodisation, indique que seul la phase PbO<sub>2</sub> est présente dans la matrice de l'électrode elle est de type β-PbO<sub>2</sub>. La caractérisation de cette même électrode par la voltamétrie cyclique à montre deux pics de réduction et un seul pic d'oxydation. Les deux pics (a) et (b) correspondants respectivement à la réaction de réduction de Pb<sup>4+</sup> en Pb<sup>2+</sup> et Pb<sup>2+</sup> en Pb, un seul pic (c) a été observé en oxydation correspondant à l'oxydation Pb<sup>2+</sup> en Pb<sup>4+</sup>.

Les résultats de la dégradation électrochimique de Cristal violet à l'aide de cette électrode montrent qu'est efficace pour l'élimination des grandes et de petites concentration (ppm jusqu'à 500 mg/l). En général, la dégradation s'arrête au stade des acides carboxyliques. Dans tous les cas, le rendement est supérieur à 95%, une dégradation totale a été obtenue après 180 min de réaction pour une concentration initiale de 100 mg/l, la même réaction a conduit à 82% de dégradation après 360 min pour une concentration initiale de 500 mg/l. L'étude de l'influence de la température a montré que les taux de dégradation augmentent avec l'augmentent de la température, (60% pour 25°C, 90% pour 85°C). Notons enfin que le pH n'a pas d'effet important sur la réaction (82% pour le pH = 2, et 90% pour le pH = 12).

La dégradation de phénol par la même électrode et dans les mêmes conditions dépasse 90% de rendement pour une concentration initiale de 1g/l.

L'analyse des produits réactionnels par HPLC à montrer que la dégradation conduit à des produits intermédiaires comme le catéchol, l'hydroquinone, l'acide malique et de l'acide fumarique. La voltamétrie cyclique réalisée dans le domaine [-0,5. 1,5] mV avec une vitesse de balayage de 30 mV/s montre un seul pic de réduction attribue au couple rédoxe Pb (2+)/PbO<sub>2</sub> (4+), ce couple rédox joue le rôle médiateur dans la réaction catalytique du phénol.

**Mots clés :** oxydation électrochimique, Pb/PbO<sub>2</sub>, Cristal violet, phénol, DRX, voltamétrie cyclique, HPLC.

## Topic 3: Corrosion and Electrochemical Engineering.

[T3-OP 09]

### EVALUATION OF THE ANTI-CORROSION ACTIVITY OF A NEW AMINOPHOSPHONIC DERIVATIVE: EXPERIMENTAL AND THEORETICAL STUDY.

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#### Abstract:

A new  $\alpha$ -aminophosphonic derivative has been synthesized and its molecular structure has been characterized by spectroscopic methods: UV-vis, IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>31</sup>P NMR and MS. The anti-corrosion activity of the synthesized compound in 0.5M H<sub>2</sub>SO<sub>4</sub> solution was studied by weight loss measurements, electrochemical and theoretical methods. The polarization study indicates that the synthesized compound acts as a mixed type of inhibitor. The adsorption of the studied molecule on the carbon steel surface obeys the Langmuir isotherm. Also, quantum chemical parameters of the optimized geometry of the synthesized molecule have been calculated using the density functional method (DFT). In addition, the interactions between the adsorbed molecules and the Fe (110) surface were calculated using molecular dynamic simulations (MDS). The theoretical results obtained by DFT and MDS are in good agreement with the experimental results.

**Keywords:**  $\alpha$ -aminophosphonate, Corrosion inhibition, Adsorption, quantum chemical calculations, MDS.

[T3-OP 10]

**EFFET DE L'ELECTROLYTE SUR LES  
PROPRIETES ELECTROCHIMIQUE ET STRUCTURALE DE LA  
COUCHE DE TiO<sub>2</sub> ELABOREE PAR ANODISATION**

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**Abstract:**

L'anodisation électrochimique du titane a reçu beaucoup d'attention ces dernières années, plus particulièrement pour son application à des fins biomédicales, offrant un oxyde de titane biocompatible avec une architecture ayant de grande surface énergétique, et des caractéristiques nano-échelle hautement contrôlables.

Dans ce travail, des couches de TiO<sub>2</sub> ont été synthétisées sur une surface en titane bien polie, par la technique d'anodisation oxydative, sous une tension de 20 V, pendant 15, 30 et 45 minutes respectivement, en utilisant comme électrolyte spécifique : 1M CH<sub>3</sub>COOH /1M Na<sub>2</sub>SO<sub>4</sub>/0,5 Wt% NaF.

L'intégrité interfaciale et la variation de la composition élémentaire le long de l'interface ont été étudiées par microscopie électronique à balayage (MEB), équipé d'un analyseur aux rayons X à énergie dispersive (EDX). Le comportement électrochimique a été étudié à la fois par la mesure de l'évolution du potentiel à circuit ouvert (OCP), par spectroscopie d'impédance électrochimique (EIS), et par polarisation linéaire (LP) dans la solution de Hank comme environnement physiologique. La nature de la phase cristalline formée pendant le traitement de recuit a été mise en évidence par la spectroscopie Raman et par la technique de diffraction aux rayons X (XRD). La caractérisation au MEB révèle des réseaux de nanotubes de TiO<sub>2</sub> uniformes et alignés verticalement sur des substrats de titane, sans un effet significatif de la durée d'anodisation sur le diamètre des nanotubes. D'autre part, les mesures EIS montrent une nette amélioration du comportement à la corrosion.

**Keywords:** Oxydation anodique, EIS, Ti6Al4V, Raman, DRX, Biocompatibilité.

[T3-OP 11]

**ARTEMISIA LEAVES EXTRACT AS GREEN INHIBITOR FOR ALUMINUM CORROSION IN HYDROCHLORIC ACID SOLUTION**

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**Abstract:**

The good corrosion resistance of aluminum and its alloys is improved by the spontaneous formation of a compact adherent passive oxide film. But when exposed to aggressive environment, such as the use of acid solutions in pickling, industrial cleaning, chemical and electrochemical etching, it corrodes severely and can sometimes in addition to the important economic lost lead to serious accidents. In order to prevent against this phenomenon, different corrosion inhibitors are used, especially organic compounds, which contain in their structure some heteroatoms such as sulphur, phosphor, nitrogen and oxygen, in addition, they also contain double bonds, triple bonds and aromatic rings. Synthetic compounds have a good anticorrosion action, however, most of them are highly toxic to humans and to the environment and most of them are banned.

As a result of environment requirements, researches and development works carried out on oils and plant extracts as a new sources of green inhibitors; biodegradable compounds, ecological guarantee and high efficiency at cheaper price. The current study occurred in this context. Artemisia plant was chosen for this purpose. It was picked in the east of Algeria, dried in the darkness and directly extracted in HCl 1 M. The inhibition efficiency of AA2017 in HCl 1 M was evaluated by electrochemical measurements (potentiodynamic polarization, electrochemical impedance spectroscopy and gravimetric measurements). The results indicate an increase of the protection efficiency with the inhibitor concentration to attain 83% for an optimum concentration of 1,5 g/L of inhibitor and that the Artemisia extract acts as mixed-type inhibitor.

**Keywords:** corrosion, green inhibitor, aluminum alloy, A2017, HCl, polarization, EIS.

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[T3-OP 12]

**ELABORATION ET CARACTERISATION D'UN MATERIAU  
COMPOSITE A BASE DE DIOXYDE DE TITANE ET DE  
POLYPYRROLE : APPLICATION A LA PROTECTION D'ACIER  
INOXYDABLE TYPE L304 CONTRE LA CORROSION.**

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**Résumé :**

Nous présentons une stratégie simple et facile pour la dispersion des nanoparticules de dioxyde de titane dans une matrice de polymère par électropolymérisation in situ en utilisant de dodécyl sulfate de sodium (SDS). Les revêtements préparés à partir de polypyrrole et de particules de TiO<sub>2</sub> synthétisé par polymérisation in situ se sont révélés présenter une excellente résistance à la corrosion bien supérieure au polypyrrole (PPy) seul dans la solution de NaCl 3,5 %.

Les matériaux résultants ont été testés comme revêtements pour la protection de l'acier inoxydable L304 contre la corrosion. L'analyse de ces matériaux par la spectroscopie d'impédance électrochimique et par les courbes de polarisation montre que la vitesse de corrosion diminue et les potentiels de corrosion se trouvent décalés vers des potentiels nobles avec l'ajout de dodécyl sulfate de sodium (SDS). Cependant les composites PPy-TiO<sub>2</sub> et PPy-TiO<sub>2</sub>-SDS peuvent être des bons candidats pour la protection des aciers contre la corrosion.

**Mots clés :** Dioxyde de titane, Résistance à la corrosion, Polypyrrole, Matériau composite.

[T3-OP 13]

**INHIBITORY EFFECTIVENESS OF SOME BENZIMIDAZOLES TO  
WARDS MILD STEEL CORROSION IN HCL SOLUTION**

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**Abstract:**

In recent years, non-toxic benzimidazole and its derivatives compounds have received notably attention due to their inhibition characteristics for metallic corrosion and have been demonstrated that they are excellent inhibitors for metals in acidic solutions. The nitrogen atoms and the aromatic ring in their molecular structure facilitate the adsorption of these compounds on the metallic surface. According to the literature quoted above, it is noted that 2-aryl-1-arylmethyl-1H-benzimidazole have not been studied as corrosion inhibitors in acidic medium with respect to the XC52 steel. In this order, we report the investigation on the corrosion inhibition performance of the eco-friendly compounds derived from benzimidazole against corrosion of the XC52 steel in 1M hydrochloric acid solution. The inhibiting action of the **1-Benzyl-2-phényl-1H-benzimidazole (BI)** and **1-(4-Nitrobenzyl)-2-(4-nitrophényl)-1H-benzimidazole (NNBI)** has been studied by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization. Results showed that the inhibition efficiency increased with the elevating of the concentration of the benzimidazole derivatives. Tafel polarization showed that both benzimidazole derivatives are mixed corrosion inhibitors. The adsorption of the benzimidazole derivatives decreases the double layer capacitance and increases the polarization resistance. The inhibitor performance depends strongly on the type of functional groups on benzene ring. It was found that the presence of the nitro group in the benzimidazole derivative (NNBI) reduces the adsorption of molecules on the surface than it is the case with BI. The adsorption process is spontaneous one and follows Langmuir adsorption isotherm model. Calculation of thermodynamic parameters such as  $DG_{ads}^{\circ}$ ,  $DH_a^{\circ}$  and  $DS_a^{\circ}$  shows that the inhibitive action of the inhibitors is in the following order BI > NNBI.

**Keywords:** Corrosion, organic inhibitors, benzimidazole, XC52 steel.



[T3-OP 14]

**SYNTHESIS OF ETHYLENE BIS [(2-HYDROXY-5, 1, 3-PHENYLENE) BIS METHYLENE]} TETRAPHOSPHONIC ACID AND THEIR ANTICORROSIVE EFFECT ON CARBON STEEL IN 3% NaCl SOLUTION**

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**Abstract:**

A new phosphonic acid derivative namely {ethylene bis [(2-hydroxy-5, 1, 3-phenylene) bis methylene]} tetraphosphonic acid (ETPA) was synthesized and characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR; <sup>31</sup>P NMR and MS spectroscopic methods. Its inhibitive action on the corrosion of carbon steel in 3%NaCl solution at 298 K has been studied. Weight loss measurements, potentiodynamic polarisation and impedance spectroscopy (EIS) methods have been used. The inhibition efficiency increases with the concentration of ETPA to attain 95% at 10<sup>-3</sup>M. We have noted a good agreement between gravimetric and electrochemical methods (potentiodynamic and impedance spectroscopy (EIS)). Polarisation curves showed their behaviors as mixed-type inhibitor. EIS spectra exhibit one capacitive loop and confirm the inhibitive ability. Surface analysis was carried out to establish the mechanism of corrosion inhibition of carbon steel in 3%NaCl solution.

**Keywords:** Synthesize, Phosphonate, Corrosion, Inhibition, Carbon steel.





[T3-OP 15]

**EFFECT OF IODIDE IONS ON CORROSION INHIBITION OF  
COPPER IN 0.5 M H<sub>2</sub>SO<sub>4</sub>**

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**Abstract:**

The effect of iodide ions on the corrosion inhibition of copper in 0.5 M sulfuric acid in the presence schiff base was studied by different electrochemical techniques such as electrochemical impedance spectroscopy, potentiodynamic polarization and polarization resistance measurements. The obtained results showed that schiff base effectively reduces the corrosion rate of copper. The addition of potassium iodide (KI) enhanced the inhibition efficiency (P%). The adsorption of schiff base alone and in combination with KI followed Langmuir adsorption isotherm. AFM microscopy images revealed that the damage of copper surface has diminished in the presence of inhibitor.

[T3-OP 16]

**$\alpha$ -HYDROXYPHOSPHONATES DERIVATIVES AS A NOVEL  
CORROSION INHIBITOR FOR  $\alpha$  CARBON STEEL XC48:  
EXPERIMENTAL, DENSITY FUNCTIONAL THEORY AND  
MOLECULAR DYNAMIC SIMULATION**

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**Abstract:**

New [hydroxy (phenyl) methyl] phosphonate acid (HPMPA) and the diethyl [hydroxy (phenyl) methyl] phosphonate (DHPMP) [1] have been synthesized and their structures were confirmed by IR, UV-Vis,  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{31}\text{P}$  NMR spectroscopy. Their inhibitive capacities on the XC48 steel corrosion in 1M HCl has been determined by chemical and electrochemical measurement. The weight loss proves that the adsorption of AHPMP and DHPMP on the surface of XC48 steel obeys Langmuir isotherm, while the polarization measurements indicate that the inhibition process is mixed, finally, Electrochemical Impedance Spectroscopy (EIS) results show that the inhibition efficiency increased with increasing the inhibitors concentrations. The Atomic Force Microscopy (AFM) analyses the attacked surfaces. The Density Functional Theory (DFT) using **(B3LYP) method with 6-31G (p, d) basis sets** [2] was performed to calculate some thermodynamics parameters influencing the corrosion inhibition effect. In addition, the Molecular Dynamics Simulations (MDS) [3] was performed to simulate the interface configuration between the inhibitors molecules and Fe (110)/H<sub>2</sub>O or Fe<sub>2</sub>O<sub>3</sub> (110)/H<sub>2</sub>O.



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[T3-OP 17]

**EFFECT OF IODATE SALTS ON CORROSION COPPER IN  
CHLORHYDRIC ACID.**

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**Abstract:**

Corrosion inhibition effect of copper by iodates in neutral and basic media is well known, but in acid medium, no systematic studies are carried out. The present work showed the inhibitory properties of iodates in 0.5 M HCl medium; the effect of the cations (Na<sup>+</sup>, K<sup>+</sup>, and Cs<sup>+</sup>) of the iodate salts is highlighted. Mass loss and electrochemical technics were used. The results shown that iodate acts effectively against corrosion, and this efficiency depends on the type and size of the cations. Smaller the cation size was, better is the inhibitory efficiency. The thermodynamic study allows determining the type of interaction of the inhibitor with the metal surface. The free adsorption enthalpies values determined to confirm the chemical character of the inhibitor/metal interactions.

**Keywords:** iodates; copper; corrosion; inhibition; Mass loss.



## Topic 4: Organic and Bioelectrochemistry.

[T4-OP 18]

### SYNTHESIS, CHARACTERIZATION, ELECTROCHEMICAL AND BIOCHEMICAL EVALUATIONS OF ANTIOXIDANT ACTIVITY OF NEW HYDRAZONES

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#### Abstract:

The hydrazones Hyd1, Hyd2 were synthesized via the condensation of the substitute benzaldehyde, indolbenzaldehyde and pyridinehydrazine, characterized by panel spectroscopic methods such as UV, IR, elemental analysis, NMR ( $^1\text{H}$ ,  $^{13}\text{C}$ ), and mass spectroscopy. Molecular structure proprieties were evaluated using density functional theory (DFT) via B3LYP/6-31G (d,p) method. The antioxidant effect was evaluated by electrochemical and biochemical assays using Superoxide anion scavenging method. In electrochemical assay, cyclic voltammetry (CV) study was carried out employing a Voltalab 40 model PGZ301 potentiostat/galvanostat controlled by a personal computer through the voltmaster4 software. For biochemical evaluation, the assay was carried out using alkaline DMSO method for generate Superoxide anion.

The obtained results indicate high antioxidant effect of the both hydrazones with inhibition percentage of 77.3 % and 83.51% respectively in electrochemical assay and 87 %, 75.88 % in biochemical assay. We conclude that the both methods confirm the higher antioxidant activity of the synthesized compounds.



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[T4-OP 19]

**A BIOELECTROCHEMICAL STUDY OF A SUSPENSION OF  
*Escherichia coli* CELLS METABOLIZING GLUCOSE AND LACTOSE**

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**Abstract:**

The metabolism of glucose and lactose in *Escherichia coli* K-12 cells has been studied using a bioelectrochemical (BEC) approach. The magnitude and the duration of the response of a BEC anode were found to be functions of the composition of nutrient media and the concentration of bacterial cells. The amount of electricity that is generated enzymatically during the metabolism of a particular substrate depends on the activity of the relevant enzymes. This suggests that the BEC approach can be used for evaluating the activity of particular enzyme systems.

**Keywords:** Metabolism, cells, mediator, bioelectrochemical, approach.

[T4-OP 20]

**APPLICATION OF ION IMPRINTED 2-POLYPYRROLE-COOH  
FILMS FOR THE DETECTION OF LEAD IN WATER—A HIGHLY  
SELECTIVE APPROACH**

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**Abstract:**

In this work lead-imprinted polypyrrole films were electrochemically prepared on glassy carbon electrodes in aqueous solutions of 2-pyrrole carboxylic acid, lead (Pb) and LiClO<sub>4</sub> as supporting electrolyte. Cyclic voltammetry and chronoamperometry were used as electropolymerization methods. The lead template ion was successfully trapped in the polypyrrole-COOH (PPy-COOH) film where it created artificial recognition sites. After extraction of the template, the PPy-COOH film acted as an ionic imprinted polymer (IIP) for the specific and selective recognition of lead whereas the non imprinted polymer (NIP) film did not exhibit any oxidation peak which demonstrates that the imprinted PPy-COOH films are specific towards Pb. The lead-imprinted PPy-COOH films were found to selectively detect Pb against the ions interferents as Copper, Cadmium, Nickel and Zinc. The plot of the variation of the current intensity as a function of the concentration of Pb is a straight line which varies linearly in the potential range studied and the detection limit was estimated at nanomole with signal to noise ratio (S/N =3).

This work highlights the possibility to design, via a simple and rapid electrochemical fabrication procedure, ions imprinted polymer films for specific, selective and sensitive electroanalysis of ions.

**Keywords:** Polypyrrole, ionic imprinted polymers, cyclic voltammetry, square wave voltammetry, electrodedetection, environment.

[T4-OP 21]

**ELECTROCATALYTIC OXIDATION OF FLUMEQUINE BY  
ELECTROGENERATED PPy-Ag MODIFIED ELECTRODE.**

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**Abstract :**

Aquaculture practice and production has increased because of the improved conditions in the aquaculture facilities. Such improvements include better water quality, infection control etc. However, this rising development has a negative effects on the aqua-mediums i.e., it generates undesirable effects e.g., contamination with chemicals and antimicrobial. Besides, residues of antibiotics are a real danger to human health since their abusive use leads to the development of antibiotic-resistant bacteria and allergic reactions [1]

Flumequine (Flu) is considered one of the commonly antibiotics that are used for fish treatment in aquaculture ponds. The present study aims to synthesize an electrogenerated PPy-Ag modified electrode for the electrocatalytical oxidation of Flumequine (Flu). A highly sensitive electrocatalytic polymer nanocomposite material of polypyrrole-Ag was synthesized by means of cyclic voltammetry. In addition, the composite films electrocatalytical effect were investigated by cyclic voltammetry. Square-wave voltammetry [2] was chosen as detection technique for the detection and monitoring of (Flu) amounts. A linear relationship between (Flu) concentration and its corresponding oxidation pics was found. A detection limit of  $10^{-8}$  M was found by linear regression.

**Keywords:**Flumequine, composite material, catalysis, environment.

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[T4-OP 22]

**LES LIGANDS «CALIXARENES», ET LEURS COMPLEXES  
METALLIQUES: SYNTHÈSE, CARACTÉRISATION,  
COMPORTEMENT ELECTROCHIMIQUE ET EFFET  
THERAPEUTIQUE (POUVOIR OXYDANT ET ANTI-  
BACTERIEN)**

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**Résumé :**

L'étude des complexes fut depuis des années l'objectif de plusieurs chercheurs et scientifiques. La compréhension de leurs structures, compositions, conformations, et même leurs réactivités vis-à-vis des réactions chimiques industrielles pharmaceutiques ou biologiques a fait l'objet de plusieurs investigations.

Ils peuvent aussi avoir une vaste application dans l'industrie « lutte contre la corrosion » ou dans l'environnement en général comme dans le « traitement des eaux » à cause de leur grande capacité de complexation des métaux de transition et notamment dans la récupération des métaux lourds toxiques provenant des rejets industriels ainsi que des éléments radioactifs induits dans les effluents nucléaires.

Les ligands type « calixarènes » et leurs complexes représentent une classe importante de chélatants dans la chimie de coordination. Ces composés ont toujours joué un rôle important dans différents domaines de la chimie. En effet, des efforts conséquents ont été déployés pour innover et généraliser leur obtention par les méthodes synthétiques.

La thiourée est d'un grand intérêt, d'où son utilisation en chimie analytique, en biologie, en environnement et surtout en médecine, en raison des activités biologiques qui sont attribuées à ses complexes métalliques. Ces derniers possèdent un effet antibactérien, antiviral, antioxydant, anti tumoral, anti-cancérigène et anti-inflammatoire.

Ils sont largement utilisés en pharmacie pour la conception de certains médicaments ou en médecine dans le traitement de plusieurs maladies.



## Topic 5: Physical and Analytical Electrochemistry.

[T5-OP 23]

### SYNTHESIS, CHARACTERIZATION, ELECTROCHEMICAL AND THERMAL BEHAVIOR OF A NOVEL COMPLEX OF MANGANESE (III) CONTAINING AN ANILINIC MOIETY WHICH USED AS MODIFIED ELECTRODES.

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#### Abstract:

The tetradentate Schiff base manganese complex was synthesized through the reaction of N,N-Bis[5-(N,N-methylphenylaminomethyl)methylsalicylidine]-1,2-diaminoethane and stoichiometric amount of tetrahydrated manganese acetate in absolute ethanol at 50 °C under nitrogen atmosphere given the expected tetradentate Mn(II)-Schiff base complex. The synthesized compound was characterized by different spectroscopic methods such as FT-IR, UV-Vis, mass spectrometry, and thermal analyses (TGA, DTG). Cyclic voltammetry was also employed to investigate the redox behavior of the manganese complex in dimethyl formamide (DMF). The corresponding manganese complex was then electropolymerized onto glassy carbon (GC) and indium tin oxide (ITO) electrode surfaces. These films were obtained by the successive cycling at the appropriate potentials. The polymer, which was electrodeposited onto ITO substrates, was characterized by scanning electron microscopy (SEM) and dispersive energy X-ray spectroscopy.

**Key words:** Manganese(II)-Schiff base complex, Cyclic voltammetry, Scanning electron microscopy (SEM), Thermal analysis.

[T5-OP 24]

**CONCEPTION D'UNE ELECTRODE A BASE DE COMPOSITE  
GRAPHITE-MAGHNITE EN VUE DE LA DETECTION DES METAUX  
LOURDS A L'ETAT DE TRACES (PLOMB ET CADMIUM)**

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**Résumé :**

Les métaux lourds [1] sont rejetés dans l'environnement principalement par les activités industrielles et sont connus pour être un poison métabolique général et un inhibiteur d'enzymes. Beaucoup de ces métaux (par exemple, Hg (II), Cu (II), Cd (II), Pb (II), Cr (VI)) sont cancérigènes et sont impliqués dans de nombreuses maladies dont la maladie de Parkinson, la maladie d'Alzheimer, la sclérose, les troubles du développement et l'échec de nombreux organes, même lorsqu'ils sont trouvés à l'état de traces [2].

Ce travail décrit l'élaboration réussie d'une électrode à pâte de carbone modifiée par une bentonite algérienne (Maghnite [3]) comme capteur électrochimique non polluant et sensible en vue de la détection du cadmium et du plomb à l'état de traces en solution aqueuse grâce à la voltampérométrie par redissolution anodique en mode onde carrée (SWASV).

Les valeurs optimales des paramètres de l'étape de preconcentration (accumulation) sous potentiel à circuit ouvert ont été : pH de preconcentration = 3,4 ; teneur de la pâte de l'électrode en Maghnite =14% en masse et temps de preconcentration (tp) = 4mn. Les valeurs optimales des paramètres de l'étape de détection (par redissolution anodique en mode onde carrée et après échange de milieu) ont été: potentiel de déposition ( $E_d$ ) = -0,8V, temps de déposition ( $t_d$ ) = 15s, hauteur de l'impulsion ( $P_H$ ) = 50mV, durée de l'impulsion ( $P_W$ ) = 20ms, hauteur du pas ( $S_H$ ) = 8mV.

Sous ces conditions optimales déterminées, la réponse de l'électrode développée était linéaire ( $R^2 = 0.9828$ ) avec la concentration de plomb dans la gamme étudiée de 10.3– 414.3  $\mu\text{g} / \text{L}$  (51 nM – 2,0  $\mu\text{M}$ ) avec une limite de détection (LOD) égale à 8,5  $\mu\text{gL}^{-1}$  (41 nM).

**Mots clés :** Bentonite, Maghnite, Electrode à pate de carbone, Voltampérométrie par redissolution anodique, Voltampérométrie à onde carrée, Capteur électrochimique, Cadmium et Plomb.

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[T5-OP 25]

**STUDY OF THE ELECTRICAL PROPERTIES OF ORGANO-MINERAL HYBRID MATERIALS (THIOLS-SMECTITE) ELABORATED BY ELECTRODEPOSITION ON A METAL ALLOY**

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**Abstract:**

The development of organo-mineral hybrid materials based on the functionalization of surfaces is a topic of current research. These composite materials possessing physicochemical, mechanical and electrical controllable properties find applications in several fields such as corrosion, storage, and bio-detection. We investigated the electrical properties of a hybrid deposit consisting of a smectite clay and organic alkyl thiol molecules ( $\text{CH}_3[\text{CH}_2]_n\text{SH}$ ) electro-deposited from an electrolyte solution on a metallic electrode alloy constituted mainly from gold. Smectites are phyllo-silicate clay minerals formed from nano-layers that crystallize in a TOT (Tetra, Octa, Tetra) -hydric structure, they carry a permanent negative charge, which provides them easy interactions in electrolytic solutions. Self-assembled thiol monolayers (SAMs) are very thin organic coatings used to functionalize solid surfaces and modify their electrochemical properties. Thiols have a high affinity for gold, they self-assemble to form a 2D network of type  $(\sqrt{3} \times \sqrt{3}) R30$ . Electrodeposition provides in-situ electrochemical cleaning of the substrate and rapid formation of SAM with reproducible electrochemical properties.

In this work we have imposed on a working electrode (substrate deposition) of metal alloy a continuous potential, varied cyclically in the redox region between -1 and 0.5 volts versus the reference electrode (Ag/AgCl). The evolution of faradic current is measured from cyclic voltammograms. The amplitude and position of the current peaks are directly linked to the quality and quantity of the deposit. Two types of deposits are studied; one is formed of smectite deposits on the gold alloy, the other is a thiol-smectite hybrid. The electrical properties and the reversibility of the resulting deposits are studied as a function of the scanning rate and the thickness of the deposit.

**Key words:** electrodeposition, hybrid materials, SAMs

[T5-OP 26]

**INVESTIGATION OF BIMETALLIC ELECTRODEPOSITION ON  
PENCIL GRAPHITE ELECTRODE FOR GLUCOSE SENSING**

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**Abstract:**

This work aims to investigate the electrodeposition parameters for the preparation of an efficient glucose sensor. Bimetallic electrodeposition of nickel and copper on a disposable pencil graphite electrode (PGE) was carried out using cyclic voltammetry. The morphology and composition of the modified PGE electrode were characterized by field gun-emission scanning electron microscopy (FEG-SEM) and energy-dispersive X-ray spectroscopy (EDX). Different amounts of both metals were electrodeposited onto pencil graphite electrodes depending on the electrodeposition cycles. The as-prepared electrodes were used to determine glucose concentration for the direct monitoring and control of glucose in human blood application. Sensitivity and reliability of the different electrodes against glucose determination were studied by varying the amperometric potential.

It was found that the as-prepared electrodes showed a very good sensitivity for glucose. The synergetic effect of both metals is likely to be the key parameter in glucose determination.

Both metals could catalyze glucose electrooxidation at a certain level. The presence of copper and nickel at the surface of the electrode shows an enhancement in the catalytic effect if compared with single metal modified electrode.

This investigation shows that the electrochemical modification of pencil graphite is a fast and a simple method for accurate detection of glucose. The applicability of the bimetallic modified pencil graphite electrode for glucose determination in human blood serum was successfully achieved. These findings confirm once again the applicability of the new amperometric sensor for glucose determination.

**Keywords:** Glucose, Amperometric Sensor, Bimetallic, Electrodeposition

## Topic 7: Nanomaterial for Electrochemical and Photovoltaic Applications.

[T7-OP 27]

### EFFECT OF AL PROPORTION ON NANOSRUCTURED AZO FILMS PROPERTIES

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#### Abstract:

In this work, we present the conditions of development of ZnO thin films pure and doped with Aluminium ions noticed AZO films by electrochemical method on conducting transparent substrates (FTO) from aqueous mixed bath of zinc nitrate and potassium chloride with 2%,4%,6%,8% of Al(NO<sub>3</sub>)<sub>3</sub> as doping via cyclic voltamperometry technique.

Thin and adherent films were obtained after same deposited time of 60min. The structural properties have been performed using X ray diffraction which showed that all obtained AZO thin films present wurtzite phase with highly c-axis preferred orientation.They have a great transparency which increases with the increasing of doping percentage of aluminum ions. The SEM analysis revealed that AZO deposits present nanostructures with smaller size than the ZnO which exhibits greater compactness and well tightly arranged nanorods with important increasing generated photocurrents under illumination. It led also to the reduction of the optical gap layers of both ZnO and AZO.

**Keywords:** ZnO , AZO, Electrodeposition, Al doping.

[T7-OP 28]

## PLASMONIC EFFECT OF SILVER NANOPARTICLES ON THE PERFORMANCE OF ORGANIC PHOTOVOLTAIC SOLAR CELLS

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### Abstract:

Over the past decade, we have witnessed rapid advances in the development of organic photovoltaic devices (OPVs). At present, the highest level of efficiency has surpassed 10%, suggesting that OPVs have great potential to become competitive with other thin-film solar technologies. Light trapping, due to the embedding of metallic nanoparticles, shows the beneficial for a better photoabsorption in organic solar cells. The different enhancement mechanisms that have been proposed and indicates future trends in the development of NPs incorporated in OPVs. Plasmon based light trapping technologies functioned efficiently as a prominent way to improve light harvesting of photoactive materials, thus to improve device power conversion efficiency (PCE). The positive results that have been obtained pave the way for practical applications of high-efficiency plasmonic OPVs devices. Broadband enhanced light absorption and the relationship between resonance and photoactive material absorption should be further investigated to improve the light harvesting properties of plasmonic solar cells. An OPVs, in which long wavelength absorbing was doped and Ag NPs were embedded into the PEDOT: PSS, was successfully fabricated. The absorption spectra of P3HT: PCBM reveals significant enhancement of the photocurrent Ag NPs-embedded PEDOT:PSS the strong local field enhancement around the metal nanoparticles to increase absorption efficiency in the active layer of OPVs. Subsequently, the power conversion efficiency (PCE) of the OPV with Ag NPs improves compared with the device without Ag NPs. The embedded Ag NPs into OPVs offers an effective strategy to improve the performance of OPVs.

**Key worlds:** organic solar cell, plasmonic effect, silver Nanoparticles



[T7-OP 29]

## ELECTROCHEMICAL DEPOSITION OF METAL OXIDES FOR SOLAR CELL APPLICATION

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### Abstract:

In this work, we attempted to build on previous research in the field of electrochemical deposition for the electrodeposition of metal oxides nanostructures to improve the photovoltaic properties of metal oxide-based film solar cell devices [1,2,3]. The development of n-type ( $\text{Cu}_2\text{O}$  or  $\text{ZnO}$ ) to obtain high efficiency of solar cells based on metal oxides hetero or homo-junctions remain a challenge. Different methods were used for producing zinc and cuprous oxides as two of the most promising metallic oxides semiconductors materials for nanotechnology. Electrochemical deposition (ECD) presents several advantages over the other techniques, such as low temperature processing, potentially low cost owing to the nonrequirement of either high vacuum or high temperature, precisely control the thickness and morphology of films by adjusting electrodeposition parameters, and higher deposition rates. In effect, electrodeposition of zinc oxide ( $\text{ZnO}$ ) and cuprous oxide ( $\text{Cu}_2\text{O}$ ) are a versatile growth method and many various nanostructures can be easily designed by this technique [1,4].

In this work, both  $\text{ZnO}$  and  $\text{Cu}_2\text{O}$  nanostructures were prepared by electrochemical deposition (ECD) method from aqueous metallic solution onto FTO) substrates. A systematic study of different parameters of deposition on the electrochemical, surface morphology, structural and optical properties of these nanostructures were examined. Characterization of obtained nanostructures was realized using conventional electrochemical techniques (cyclic voltammetry, Mott-Schottky), atomic force microscopy (AFM), scanning electron microscopy (SEM), X-ray diffraction (XRD), UV-vis and photoluminescence techniques.



[T7-OP 30]

**SYNTHESIS OF A CONDUCTING NANOCOMPOSITE BY  
INTERCALATIVE COPOLYMERISATION OF THIOPHENE AND  
ANILINE IN MONTMORILLONITE**

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**Abstract:**

In this study, nanocomposites polyaniline/ $M-H^+$ , polythiophene / $M-H^+$  and poly(ANI-co-Th)/ $M-H^+$  were successfully synthesised by chemical oxidative polymerisation using thiophene and/or aniline monomers in the presence of Maghnite- $H^+$  ( $M-H^+$ ). All organic monomers used were first intercalated into the interlayer regions of clay hosts followed by a one-step in-situ polymerisation.

The unique properties of the as synthesised nanocomposites materials are analysed by X-ray diffraction ,UV–Vis spectroscopy, Fourier-transform infrared(FT-IR)spectroscopy and the electrochemical behaviour of the polymers extracted from the nanocomposites has been studied by cyclic voltammetry which indicates the electro-active effect of nanocomposite.

**Keywords:** polythiophene , nanocomposites , polyaniline, conducting polymer.



[T7-OP 31]

## MATERIAUX D'ELECTRODES : SUPPORT/POLYMERE CONDUCTEURS-MICROPARTICULES METALLIQUES

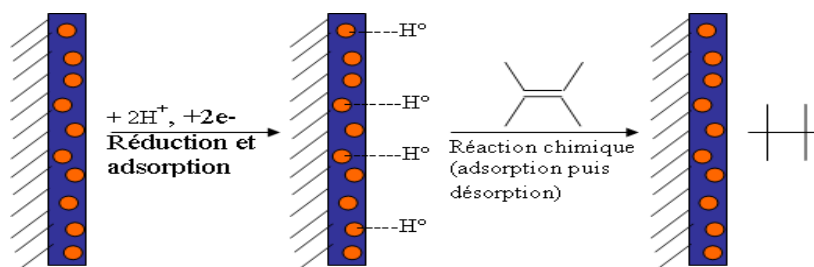
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### Résumé:

La modification de la surface d'électrodes par des architectures moléculaires possédant des propriétés catalytiques et analytiques, connaît un intérêt grandissant pour les systèmes de l'analyse et de la catalyse. Plus généralement, le rôle économique croissant des micro- et nano-technologies nécessite la mise au point de nouvelles méthodes de fonctionnalisation de surfaces par des films minces de matériaux nanostructurés à propriétés spécifiques.

Ce travail concerne des études fondamentales dans le domaine de l'élaboration et de la caractérisation de matériaux composites, plus précisément des matériaux polymères contenant une dispersion de nanoparticules de métaux à propriétés catalytiques (schéma 1), analytiques et magnétiques (métaux de transition). Les applications développées sont l'hydrogénation électrocatalytique de composés organiques, les cellules photovoltaïques et les générateurs électrochimiques.



**Schéma 1:** Electrohydrogénation d'un alcène sur une électrode modifiée par un film de polymère contenant des microparticules de platine.

**Keywords:** Matériaux d'électrodes, polymères conducteurs, catalyse, cellules photovoltaïques



# **Poster presentations**

## Topic 1: Batteries and Energy storage.

[T1-PP 01]

### AMÉLIORATION DES PROPRIÉTÉS CAPACITIVES DE POLYANILINE PAR INCORPORATION DE GRAPHÈNE

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#### Résumé:

Dans ce travail, des films composites polyaniline-graphène (PANI-GR) ont été élaborés par chronoampérométrie sur une électrode en oxyde d'étain dopé au fluor (FTO), le graphène a été synthétisé par une méthode d'exfoliation électrochimique. Au cours de l'électropolymérisation, l'incorporation du graphène synthétisé dans la matrice de polyaniline a été assurée par agitation de l'électrolyte ( $10^{-2}$  M Aniline / 1M H<sub>2</sub>SO<sub>4</sub>) contenant différentes masses de graphène (1, 2 et 3 mg). Grâce à la haute conductivité de GR et de la pseudocapacité de PANI, le film composite FTO / PANI-GR a été pris comme exemple pour l'application aux matériaux d'électrode de supercondensateur. La morphologie et la structure de FTO/PANI et de FTO/PANI-GR ont été caractérisées par différentes techniques MEB, DRX, FTIR, spectroscopie Raman et spectroscopie UV-visible. La performance électrochimique a été évaluée par voltamétrie cyclique, tests de charge-décharge galvanostatique et spectroscopie d'impédance électrochimique (SIE). La capacité spécifique obtenue pour le matériau PANI seul est d'environ 176,29 Fg<sup>-1</sup> cette valeur a été augmentée jusqu'à 305,57 Fg<sup>-1</sup> pour le film composite PANI-GR 3mg à 5 mVs<sup>-1</sup>.

**MOTS-CLES:** électropolymérisation, Polyaniline, Graphène, films composites, supercondensateurs.



[T1-PP 02]

## CHEMICAL AND ELECTROCHEMICAL SYNTHESIS OF NANOSTRUCTURED $\gamma$ -MnO<sub>2</sub>

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### Abstract:

The manganese dioxide with all its varieties has a large utilization in the chemical industry as in zinc batteries applications, especially  $\gamma$ -MnO<sub>2</sub> due to its high electrochemical properties.

$\gamma$ -MnO<sub>2</sub> as powder was prepared with chemical method by mixing MnSO<sub>4</sub>.H<sub>2</sub>O with (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as oxidizing agent in high temperature for 24h, while it was prepared electrochemically by electrodeposition on titanium plate substrate from hot MnSO<sub>4</sub>.H<sub>2</sub>O aqueous solution with pH value adjusted to 2.

The two  $\gamma$ -MnO<sub>2</sub> powders were characterized by using SEM and XRD techniques.

**Keywords:** Nanostructured,  $\gamma$ -MnO<sub>2</sub>, Electrodeposition.



[T1-PP 03]

**SYNTHESE ELECTROCHIMIQUE DE MATERIAUX COMPOSITES  
POLYANILINE /MNO<sub>2</sub> POUR DES SUPERCAPACITEURS  
ELECTROCHIMIQUES.**

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**Résumé :**

L'objectif recherché dans cette étude est électrodéposition de PANI/MnO<sub>2</sub> sur des plaques d'ITO a été préparé Par électropolymérisation de polyaniline ou le MnO<sub>2</sub> est considéré comme agent additif. Les films ont été préparées par chronoampérométrie à partir d'une solution aqueuse d'aniline (0.1M) + H<sub>2</sub>SO<sub>4</sub> (1M) comme électrolyte support à un potentiel imposé de 800 mV/ECS à la présence et en absence de MnO<sub>2</sub>.

La caractérisation des films déposés a été réalisée par des méthodes électrochimiques (voltammétrie cyclique, spectroscopie d'impédance électrochimique (SIE), charge-décharge galvanostatique). Les résultats obtenus montrent que les particules de MnO<sub>2</sub> sont incorporées dans la matrice polymère (PANI). De plus, le matériau composite (PANI / MnO<sub>2</sub>) présente une conductivité électrique modérée par rapport au film PANI pur. Il est à noter que le matériau composite présente une amélioration significative des propriétés électrochimiques qui nous renseignent sur les applications dans les supercondensateurs.

**Mots-cles :** électropolymérisation, Polyaniline, dioxyde de manganèse, supercondensateurs.

[T1-PP 04]

**INFLUENCE OF POLYANILINE COATING ON THE CYCLE LIFE OF  
PbO<sub>2</sub> AND Pb ELECTRODE IN LEAD ACID-BATTERIES**

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**Abstract:**

Polyaniline deposits on the Pb or PbO<sub>2</sub> surface were synthesized using cyclic voltammetry and pulsed in sulfuric acid media in the presence of aniline. Polyaniline coating of various controlled thicknesses are applied to Pb substrate, simultaneously without and with the PbO<sub>2</sub> formation and evaluated for their capacity in a H<sub>2</sub>SO<sub>4</sub> 1M environment using cyclic voltammetry. The characterization of coatings and their chemical functions were evaluated by FTIR, UV-Visible and DRX. The voltammetry data clearly indicate that a pure PANI layer is not formed by electrochemical oxidation of aniline on lead. Instead a mixed layer consisting of lead oxide, lead sulfate, polyaniline and its degradation products is produced. The performance of this mixed layer as anticorrosion layer was determined from polarization curves recorded in aqueous 1.5M H<sub>2</sub>SO<sub>4</sub>. Corrosion tests showed that the best coatings decreased the corrosion current density of the electrode material by up to 99 % and allowed maintaining lead in its passive state. Long-term cycling was performed by cyclic voltammetry in 1.5M H<sub>2</sub>SO<sub>4</sub> media. It was found that the deposition of polyaniline yielded a coating with good capacity. Electrochemical studies have demonstrated the efficiency of polyaniline coating for use as current collector of the positive PbO<sub>2</sub> and Pb electrode in lead-acid battery.

**Keywords:** capacity, lead acid battery, polyaniline coating, electrochemical methods (CV, EIS, Tafel).

[T1-PP 05]

**ELABORATION ET CARACTERISATION D'UN MATERIAU  
COMPOSITE P3HT/NIO ; APPLICATION EN  
SUPERCONDENSATEUR ELECTROCHIMIQUE.**

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**Résumé :**

Dans ce travail, des films P3HT modifiés par incorporation d'oxyde de nickel (NiO) avec différentes quantités ont été élaborer parla méthode de spin coating sur des substrats d'oxyde d'indium-étain (ITO). La poudre de NiOa été synthétisée par la méthode chronoampérométrie. Les effets de la teneur en NiO sur la structure morphologique et sur les propriétés optiques des films P3HT ont été étudiés par DRX, MEB, AFM et UV-vis, les performances électrochimiques ont été évaluées par voltammétrie cyclique (VC), spectroscopie d'impédance électrochimique (EIS) et par charge-décharge galvanostatiques.

Les résultats ont montré que le NiO se dispersait uniformément dans le P3HT et modifiait la rugosité de la surface et l'absorption des films de P3HT. Nous avons observé une amélioration remarquable de la capacité électrochimique de ces surfaces modifiées pour une quantité de NiO comprise entre 1 et 10%. La capacité spécifique obtenue pour le matériau P3HT seul est d'environ 20,8 Fg<sup>-1</sup>, cette valeur a été augmentée jusqu'à 81,4 Fg<sup>-1</sup> pour le film composite P3HT-NiO10% à 0,1 A / g.

**Mots-clés:** NiO, P3HT, film composite, super condensateur électrochimique.

[T1-PP 06]

**REACTIVITE ELECTROCHIMIQUE DE  $PbO_2$  PREPARE PAR VOIE  
CHIMIQUE ET ELECTROCHIMIQUE**

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**Résumé :**

Le dioxyde de plomb est préparé par voies chimique et électrochimique. L'échantillon préparé par voie chimique est plus amorphe et présente des particules de taille de cristallites plus petite par rapport à la variété électrochimique. L'analyse thermique et les techniques électrochimiques sont utilisées pour expliquer la différence de l'activité des deux variétés.

La mesure de la capacité que ce soit en mode galvanostatique ou en cyclage a montré que la variété électrochimique est plus active car l'eau de structure est répartie au sein des grains de  $PbO_2$  d'une manière homogène et ordonnée ce qui facilite la diffusion du proton. En revanche, la répartition hétérogène et aléatoire de cette eau dans la structure de  $PbO_2$  chimique limite la diffusion du proton et la capacité de la masse positive. C'est le point clé qui pourrait expliquer la différence dans l'activité électrochimique qui existe entre les deux variétés.

**Mots clés :** *Accumulateur au plomb, dioxyde de plomb, capacité, Taille des grains. Structure d'eau.*



[T1-PP 07]

**SYNTHESE DE NANOPARTICULES DE PbO<sub>2</sub> : CORRELATION  
ENTRE LA TAILLE DES GRAINS ET L'ACTIVITE  
ELECTROCHIMIQUE**

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**Abstract:**

The aim the objective of this subject and to find methods of synthesis of PbO<sub>2</sub> on a nanometric scale and to measure its electrochemical activity. The results of X-rays powder showed the presence of the  $\alpha$  phase and  $\beta$  PbO<sub>2</sub> and the PbO<sub>2</sub> particles in the sample Mte-PbO<sub>2</sub> are smaller than those of chemical PbO<sub>2</sub>. Analysis by SEM and EDAX showed that PbO<sub>2</sub> particles are arranged in parallel planes. By impedance spectroscopy, it was found that the chemical PbO<sub>2</sub> has a conductivity value significantly higher than that of the composite material. Comparative electrochemical study in H<sub>2</sub>SO<sub>4</sub> electrolyte of this composite material and chemical PbO<sub>2</sub> showed that the composite material has good electrochemical reactivity and give larger discharge capacities. In order to promote our material, we tested the electrocatalytic activity of our electrodes, the results of the electrochemical study showed that the composite material clay-PbO<sub>2</sub> has a good electric conductivity which confers to him a good efficacy for the degradation of phenol.

**Keywords:** Nanostructures, X-ray diffraction, Electrocatalytic activity. Degradation of organic Effluents.

**[T1-PP 08]**

**MÉCANISME DE LA RÉDUCTION DE L'OXYGÈNE MOLÉCULAIRE SUR  
L'OXYDE  $\text{La}_{0.45}\text{Sr}_{0.55}\text{MnO}_3$  SUPPORTÉ SUR LE VULCAN XC-72 À  
TEMPERATURE AMBIANTE.**

Mabrouk Cheriti

Université F. Abass Sétif-1

**Résumé:**

La réduction de l'oxygène a été largement étudiée en raison de son importance majeure dans plusieurs domaines, tels que la conversion d'énergie (piles à combustibles alcalins, piles métal-air), l'électro-synthèse du peroxyde d'hydrogène et les capteurs galvaniques à oxygène [1].

Cette réaction a été examinée sur différents matériaux d'électrode, en milieu acide et basique [2]. Parmi ces matériaux, les manganites de lanthanes dopés au strontium ont montré une bonne activité électrochimique vis-à-vis de la réduction de  $\text{O}_2$ .

Le présent travail s'intéresse à l'étude du comportement électrochimique de la simple pérovskite  $\text{La}_{0.45}\text{Sr}_{0.55}\text{MnO}_3$ , supporté sur du carbone (Vulcan XC-72), vis-à-vis de réduction de l'oxygène moléculaire en milieu basique ( $\text{NaOH}$  0.5M) à température ambiante.

A cet effet, diverses techniques électrochimiques ont été employées: Voltammétrie cycliques, voltmmétrie linéaire et spectroscopie d'impédance. En particulier, la voltammétrie linéaire sur une électrode à disque tournant montre que l'électrode  $\text{La}_{0.45}\text{Sr}_{0.55}\text{MnO}_3/\text{C}$  présente une bonne électroactivité et que la réduction de  $\text{O}_2$  se fait par un mécanisme direct à 4 électrons. L'étude détaillée de ce mécanisme par la technique d'impédance est en cours d'examen.

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[T1-PP 09]

**CARACTERISATION ELECTROCHIMIQUE D'UN ALLIAGE A BASE  
DE PLOMB DE RECUPERATION**

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**Abstract:**

Les alliages de plomb issus du système Pb-Sn-Sb, généralement utilisés comme matériaux d'électrodes dans les batteries acides, sont obtenus par des procédés couplant des étapes de coulée gravitaire et de mise en forme par laminage [1]. Leurs propriétés physico-chimiques, en particulier leur résistance au fluage et leur tenue en corrosion en milieu acide sulfurique concentré [2] sont loin d'être satisfaisantes. En effet, les transformations microstructurales adoucissantes telles que recristallisation et vieillissement sont activées dès la température ambiante [3].

Le but de notre travail c'est d'étudier le comportement électrochimique d'un alliage à base de Plomb de deuxième génération dont la composition chimique déterminée par EDS et de 1.84% Sb, 0.6-0.8% Sn, 0.039 % P, 0.12 % Cu, 0.072% Mn, 0.081 % Ni, 0.102 % W . Cet alliage est caractérisé par des essais mécaniques tels que (dureté, traction, Métallographie ...). Nombreux sont des essais réalisés par des méthodes électrochimiques, ces techniques ont pour but de déterminer expérimentalement les paramètres de corrosion de l'échantillon N°1 (grilles),  $I_{corr}$ ,  $V_{corr}$ ,  $R_p$ ,  $E_{corr}$ , dans deux milieux agressifs à différentes concentrations : a) acide sulfurique , b) acide nitrique et le mélange entre les deux, en simulons à chaque fois les conditions de travail d'une batterie de voiture.

**Mots clé:** Alliage de Plomb, Corrosion, Acide sulfurique, Les Batteries

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[T1-PP 10]

**PROCESS OF FORMATION OF CORROSION FILM ON Pb AND Pb-Sn ALLOYS**

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**Abstract:**

Lead-acid batteries are still the most popular secondary batteries used due to their high performance, reasonable cost, wide usable temperature range, safety and reliability. Low-antimony grid and the lead-calcium grid are widely used in lead-acid batteries. Batteries with lead-antimony alloys require regular maintenance due to the decomposition of water because of the lower over-potential of hydrogen evolution on the antimony surface during the charge/discharge process[1,2]. The lead-calcium grids exhibit a better mechanical properties and maintenance-free performance but cause a short deep-discharge cycle life. This is due to the formation of Pb(II) film with high resistivity on the positive grid alloys, seriously shortening the cycle life.

This phenomenon was called premature capacity loose (PCL)[3]. To resolve this problem, many elements are tested (such as Sn, Bi, Sm, Pr, Gd, La, Y, Sr, Ce, In, Cd, Ag and others)[4-12]. They all have improved some properties of the alloys. However, some of them such cadmium can pollute the environment heavily and be harmful to the health of the workers; others are too expensive compared to the tin. In these works, no mechanism of action of these elements on the process of formation of corrosion film is given. In this paper, we propose a mechanism of action of tin taken as a model element to clarify the performance of lead acid battery using different techniques such CV, LSV, EIS, Mott-Schotky, AC voltammetry, DRX, MEB and EDX.

**Keywords:** Lead-acid battery, Pb-Sn alloy, AC voltammetry, EIS, semiconductor.

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[T1-PP 11]

**IONIC CONDUCTIVITY OF PVA-BASED SOLID ELECTROLYTE MATERIAL**

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**Abstract:**

Proton-conducting solid electrolyte composed of polyvinyl alcohol (PVA), phosphotungstic acid (PWA) and orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>) has been prepared and characterized using various experimental techniques such as X-ray diffraction (XRD) and electrochemical impedance spectrometry (EIS). The ionic conductivity of this polymer electrolyte, which is a function of the surrounding relative humidity, increases with increasing the acid content in the polymer matrix. A maximum protonic conductivity of  $1.8 \cdot 10^{-2} \text{ S cm}^{-1}$  has been reached at RH = 100% for a sample containing 40 wt% PWA and 40 wt% H<sub>3</sub>PO<sub>4</sub>. The temperature dependence of ionic conductivity revealed the conduction mechanism to be Arrheniusthermally activated process. The minimum activation energy of the ternary PVA-PWA(40 wt.%)<sup>-</sup>H<sub>3</sub>PO<sub>4</sub>(40 wt.%) complex is found to be 1.9 kJ mol<sup>-1</sup> for optimum conductivity condition. Using this ternary PVA-EG(45 wt.%)<sup>-</sup>H<sub>3</sub>PO<sub>4</sub>(5 wt.%) complex film as electrolyte, an "all solid state" Zn/MnO<sub>2</sub> cell was fabricated and its discharge characteristics profile has been studied.

**Keywords:** Proton-conducting, PVA, PWA, protonic conductivity, impedance spectroscopy.



[T2-PP 12]

**EFFECT OF MINERAL ADDITIVE ON THE PERFORMANCE OF  
LEAD ACID BATTERY PLATE**

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**Abstract:**

The objective of this work is to improve the performance of the positive electrode of lead-acid battery. The use of the additive in the positive past is to increase the capacity and cycle life of the positive active materiel (PAM). Mineral porous additives, dispersed uniformly in the PAM, may act as acid reservoirs and favor the ionic diffusion. The results showed that the addition of mineral additive in the paste before oxidation influences the composition and the crystal size of the PAM after oxidation. We observed a remarkable improvement of the discharge capacity of the PAM for amount of additive ranging between 1 -5%, an increase of the capacity is due that water is well ordered in the structure of lead dioxide in this simples, when facilitates the diffusion of the protons in the gel parts of PbO<sub>2</sub> and gives more activity to it.

**Keywords:** Positive plate, Additive of porosity, Discharge capacity, Lead acid battery.

## Topic 2: Electrochemistry and Environment.

### [T2-PP 13]

#### STRUCTURAL AND ELECTRICAL PROPERTIES OF Cu-DOPING OF NiAl<sub>2</sub>O<sub>4</sub> AND THEIR PHOTOCATALYTIC ACTIVITY TOWARD CONGO RED UNDER SOLAR LIGHT IRRADIATION

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#### Abstract:

The environment is a capital to preserve from the high technologies and industries that produce some pollutant as heavy metals, organic compounds and dyes which are seriously toxic to microorganisms, aquatic life and also for human beings [1,2]. The present work was focused on the effect of Cu substitution on the crystal structure and photocatalytic activity of nano-spinel oxides Ni<sub>(1-x)</sub>Cu<sub>x</sub>Al<sub>2</sub>O<sub>4</sub> (x = 0.0-1.0). The as-prepared compounds were synthesized by the co-precipitation route and characterized by X-ray diffraction, scanning electron microscopy, FTR, X-Ray photoelectron spectroscopy and UV-Vis diffuse reflectance spectra. The catalytic activities were followed by UV-Vis spectroscopy and electrochemical impedance spectroscopy. The photocatalytic efficiency of the synthesized catalysts was investigated for the degradation of Congo red dye under solar light irradiation. The efficient catalyst was Ni<sub>0.2</sub>Cu<sub>0.8</sub>Al<sub>2</sub>O<sub>4</sub> with 90.55% of the removal dye after 180 min. Other parameters influencing the dye degradation like initial concentration and the catalyst were also studied for the optimum degradation and the results have been discussed. In this study, we showed that the adsorption kinetic of the Congo red followed well the Langmuir isotherm model. The high photocatalytic activity of Ni<sub>0.2</sub>Cu<sub>0.8</sub>Al<sub>2</sub>O<sub>4</sub> come from its valence band which increase the mobility of photo excited charge carriers.

**Key Words:** Spinel, XRD, Photocatalysis, Congo Red dye, Kinetic, isotherm.

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[T2-PP 14]

## HOMOGENEOUS CATALYTIC REDUCTION OF BROMOCYCLOPENTANE WITH ELECTROGENERATED NICKEL SCHIFF BASE

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### Abstract:

Catalytic reduction of alkyl halides by electrogenerated nickel(I) species has long been a focus of research conducted in our laboratory and elsewhere [1-3]. Pletcher and coworkers launched this field in 1978 with two publications dealing, respectively, with mechanistic aspects of the catalytic reduction of several alkyl halides [4] and with the addition of catalytically formed alkyl radicals to olefins [5].

In the present work, we have employed cyclic voltammetry to investigate the electrochemical behavior of the nickel Schiff base complex at glassy carbon cathodes in DMF containing 0.1 M Et<sub>4</sub>NBF<sub>4</sub> as the supporting electrolyte. The electrocatalytic performances of this complex was examined in the electroreduction reaction of bromocyclopentane.

Finally, we present a set of mechanistic steps that can account for the effects of proton donors on the catalytic reduction of an alkyl monohalide (bromocyclopentane), that take into consideration the possibility of both metal- and ligand-centered one-electron reduction of nickel(II) Schiff base, that provide pathways for alkylation of the imino bonds of nickel(II) Schiff base during these processes, and that show how the hydrocarbon products are formed.

**Keywords:** Catalytic reduction, Bromocyclopentane, Nickel(I) Schiff base, cyclic voltammetry.

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[T2-PP 15]

## ELECTROCHEMICAL AND ELECTROCHROMIC PROPERTIES OF V<sub>2</sub>O<sub>5</sub> THIN FILMS DOPED BY MO SYNTHESIZED BY SPRAY PYROLYSIS TECHNIQUE

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### Abstract:

Thin films of Mo doped V<sub>2</sub>O<sub>5</sub> were deposited by spray pyrolysis technique on preheated glass substrates at 500 °C with 2, 4, 6 at.% of Mo concentration. The deposited films were characterized by XRD, Raman, MEB, and electrochemical analysis. All the films exhibited orthorhombic structure with (001) predominant orientation. The Raman spectra confirmed the orthorhombic structure. The effects of concentration of the Mo on the optical properties of these films were studied in their colored and discolored or bleached states. The transmittance value decreased from 65% to 11% at 690 nm when the films were colored; however, when V<sub>2</sub>O<sub>5</sub> films were discolored to 1.5V, they showed a strong absorption in the UV range (350-400 nm). The electrochemical studies were performed in LiClO<sub>4</sub> 0.5 mol /L C<sub>4</sub>H<sub>6</sub>O<sub>3</sub> electrolyte.

**Keywords:** Spray pyrolysis, V<sub>2</sub>O<sub>5</sub>, (V<sub>2</sub>O<sub>5</sub>)<sub>1-y</sub>(Mo<sup>6+</sup>) thin films, Electrochromism.

[T2-PP 16]

**ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY STUDY OF  
ORANGE II DEGRADATION**

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**Abstract:**

After optimization of the operational parameters of the electro-Fenton process (nature and concentration) we identified and carried out the quantitative follow-up of the reaction intermediates formed during current controlled electrolysis. Electrochemical impedance spectroscopy (EIS) technique is performed to study electronic and ionic processes to evaluate the Orange II degradation mechanism. It is observed that the short circuit current decreases significantly whereas a noticeable increase in open circuit voltage. It is found that the decrease in current is mainly due to dissociation of Orange II molecules with respect to current and potential increase. Electrochemical impedance spectroscopy (SIE) on the Pt electrode shows a semicircle over the entire frequency range. According to the shape of the Nyquist diagram, the corresponding equivalent circuit is the one used to represent an electrochemical interface controlled by a pure charge transfer where the species transport is infinitely fast in front of the speed of electronic transfer. Moreover, the diameter of the loop is reduced, which reflects the increase of charge transfer on the active sites of Pt. The electrochemical impedance measurements carried out in acid medium made it possible to highlight the active character of the surface. Pt / electrolyte thin film thus promoting significant ion exchange at the electrode / electrolyte interface. The study of the interaction of the Pt / electrolyte films also made it possible to confirm the importance of the pH of the electrolyte.

**Key words:** Orange II, Electro-Fenton, Hydroxyl radicals, Impedance spectroscopy.

[T2-PP 17]

**ETHANOL / OXYGENE MICROFLUIDIC BIOFUEL CELLS**

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**Abstract:**

This work presents the construction of an ethanol microfluidic biofuel cell (MBFC) based on bioelectrodes and operating in a Y-shaped microfluidic channel. At the cathode, the oxygen is reduced by laccase, whereas at the anode, ethanol is oxidized by alcohol dehydrogenase. The enzymes were immobilized in the presence of reactive species at gold electrode surfaces. Oxidant and Fuel streams move in parallel laminar flow without turbulent mixing into a microchannel. The benefit of the carbon nanoparticles with higher surface porosity was explained by the high porous structure that offered a closer proximity to the reactive species and improved diffusion of ethanol and oxygen within the enzyme films. The higher current and power densities were achieved for shorter and wider electrodes that allow for thinner boundary layer depletion at the electrodes surface resulting in efficient catalytic consumption of fuel and oxidant. This miniaturized device generated maximum power density of  $90 \mu\text{W cm}^{-2}$  at 0.6 V for a flow rate  $16 \mu\text{L min}^{-1}$ .

**Keywords:** microfluidics ; carbon nanoparticles ; bioelectrodes ; enzymatic biofuel cell



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[T2-PP 18]

**OXYDATION DES COLORANTS TEXTILES SUR DES ELECTRODES  
A BASE DE POLYANILINE**

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**Résumé :**

L'intérêt croissant de la recherche pour les nanocomposites rend ce domaine d'étude dynamique, ceci repose sur leurs propriétés ce qui les confère plusieurs applications dans différents secteurs industriels.

Dans ce travail on a synthétisé la polyaniline et des composites de polyaniline/métaux de transition et polyaniline/oxyde de métaux (Pani/Fe, Pani/Cu, Pani/SnO<sub>2</sub> et Pani/ PbO<sub>2</sub>) par voie chimique. Puis on a caractérisé les matériaux obtenus par les méthodes physico-chimiques (DRX, Meb, IR et UV-vis).

A travers ces analyses nous avons pu montrer que les nanocomposites élaborés sont bien formés avec la formation de forte interaction entre les métaux et la polyaniline.

On a utilisé ces matériaux comme anode et cathode dans les procédés d'oxydation électrochimique avancés pour dégrader le rejet de l'usine de textile de Bougaa.

**Mots clés :** Polymérisation chimique, polyaniline, matériaux composite, oxydation électrochimique avancée.

[T2-PP 19]

**THE EFFICIENCY OF ADVANCED ELECTROCHEMICAL  
OXIDATION PROCESSES ON THE DEGRADATION OF DIRECT  
SOLOPHENYL RED DYE.**

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**Abstract:**

This work deals with the efficiency of the advanced oxidation electrochemical processes (PEOA) for the decoloration of the direct solophenyl red 3BL dye (RS). The electrochemical processes used are: 1) Anodic oxidation (OA) on lead dioxide deposited on a plate of partinium (Pt / PbO<sub>2</sub>), 2) Electro Fenton (EF) or oxygen peroxide is generated by the reduction of oxygen on a graphite rod and 3) by the combination of AO-EF. These treatment methods are carried out in an electrochemical cell with two electrodes immersed in an electrolyte compartment.

**Keywords:** Solophenyl red 3BL dye, advanced oxidation electrochemical processes, Anodic oxidation, Pt / PbO<sub>2</sub>, Electro Fenton.

[T2-PP 20]

**ELIMINATION DU PLOMB DES SOLS ARTIFICIEL PAR  
ELECTROCINETIQUE**

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**Résumé :**

Le traitement électrocinétique est une technique in situ utilisée pour éliminer les contaminants organiques ou inorganiques tels que les métaux lourds ou les molécules aromatiques dans les sols sous l'application d'un champ électrique.

Dans ce travail, le phénomène de remédiation électrocinétique a été testé pour éliminer les ions de plomb ( $[Pb^{2+}] = 200$  mg/kg) à partir d'un sol artificiel. L'étude expérimentale a été réalisée dans une cellule en plexiglas (volume  $\sim 2$  L), constituée de trois compartiments : anodique, cathodique, et celui du milieu, séparés par un tissu. L'acide sulfurique  $H_2SO_4$  (0.1N) a été utilisé comme milieu d'étude. Le courant imposé est de 100 mA, pendant une durée de 240 heures (10 jours).

Les résultats obtenus ont montré une substantielle diminution de la concentration en ions  $Pb^{2+}$  dans le sol, avec un tau d'élimination de  $\sim 40$  %.

**Mots clés:** Electroremédiation, décontamination, sol pollué, plomb.

[T2-PP 21]

**SYNTHESE ET CARACTERISATION DE L'OXYDE MIXTE  
La<sub>0.5</sub>Sr<sub>0.5</sub>MnO<sub>3</sub> : ETUDE DE L'EFFET DE TEMPERATURE DE  
CALCINATION SUR LES PROPRIETES CATALYTIQUES VIS-A-VIS  
DE LA REDUCTION DE L'OXYGENE**

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**Abstract :**

Les perovskites sont utilisées comme matériau d'électrode à oxygène [1]. Les méthodes classiques telles que la voie hydrothermale [2], la méthode de co-précipitation [3] et la méthode de la décomposition thermique [4] utilisées dans la littérature ont permis d'obtenir des grains de grandes tailles et des surfaces spécifiques faibles, ce qui engendre une baisse de l'activité catalytique vis-à-vis de la réaction de réduction de l'oxygène. La voie sol gel, par contre, présente l'avantage d'obtenir des composés de haute pureté, des tailles de grain faibles et homogènes à des températures de calcination relativement basses [5,6].

Le présent travail consiste à l'élaboration par voie sol-gel, en utilisant la méthode Pechini [7], d'un matériau nanométrique La<sub>0,5</sub>Sr<sub>0,5</sub>MnO<sub>3</sub>. La caractérisation de ce matériaux a été effectuée par thermogravimétrie, diffraction des rayons X et microscopie électronique à transmission. Les résultats obtenus ont permis les conclusions suivantes:

i-Leur utilisation en tant que matériau d'électrode à oxygène a montré une activité catalytique élevée vis-à-vis de la réduction de l'oxygène.

ii-L'activité de l'oxyde La<sub>0,5</sub>Sr<sub>0,5</sub>MnO<sub>3</sub> ne dépend pas uniquement de la surface spécifique mais aussi de la cristallinité.

**Mots clés:** Perovskite, Pechini, Réduction, Oxygène, Sol-gel.

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[T2-PP 22]

## STRUCTURAL AND ELECTRICAL PROPERTIES OF ELECTRODEPOSITED NI-FE THIN FILM

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### Abstract:

In this Work electrochemical from aqueous solution (0.5 M NiSO<sub>4</sub>.6H<sub>2</sub>O + 0.02MFeSO<sub>4</sub>.7H<sub>2</sub>) was performed to synthesis binary nickel-iron (Ni-Fe) on transparent conducting glass (ITO). The conditions of electrodeposition for Ni-Fe thin film growth by electrochemical deposition process were optimized. The crystalline structure, electrical and morphology of the thin films were characterized by X-ray diffraction (XRD), and electrochemical impedance spectroscopy (EIS). XRD results show that FeNi<sub>3</sub> thin film with face-centered cubic (FCC) crystalline structure was formed.

**Keywords:** electrodeposition, Ni-Fe, thin film, X-ray, EIS





[T2-PP 23]

**ANTIBACTERIAL ACTIVITY OF POLYPYRROLE AND  
POLYPYRROLE COPPER AGAINST THE GRAM-NEGATIVE  
BACTERIA *PSEUDOMONAS* AND GRAM-POSITIVE  
BACTERIA *STAPHYLOCOCCUS***

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**Abstract:**

Electroactive polypyrrole (PPy) particles obtained by oxidative polymerization of pyrrole in acetonitrile were used to remove copper ion from aqueous solutions. FTIR, cyclic voltammetry (CV), X-ray diffraction (XRD) and conductivity measurements were applied to analyze copper interaction with the synthesized polymer. Moreover, The activity of PPy and PPy-Cu was evaluated by agar well cut diffusion method against the Gram-negative bacteria *Pseudomonas* and Gram-positive bacteria *Staphylococcus*.

PPy and PPy-Cu had antibacterial activity against *Pseudomonas*, it causes inhibition zone diameter of 11 and 17 mm for PPy and PPy-Cu, respectively, but no inhibition effect against *Staphylococcus*.

**Keywords:** Polypyrrole, Synthesis, complexation, Metallic ions, Antibacterial effec. *Pseudomonas*, *Staphylococcus*.



[T2-PP 24]

**ETUDE ELECTROCHIMIQUE D'UN NOUVEAU LIGAND BASE DE  
SCHIFF ET SON COMPLEXE METALLIQUE**

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**Résumé**

La chimie des complexes métalliques fait l'objet de plusieurs recherches dans le monde entier, pour cela les ligands à base de Schiff hétérocycliques et de leurs complexes avec des métaux de transition ont été largement étudiés.

Les complexes des bases de Schiff présentent des intérêts fondamentaux dans le domaine pratique.

Dans ce travail, nous avons étudié le comportement électrochimique d'un ligand base de Schiff hétérocyclique et de son complexe de Cu(II) par voltampérométrie cyclique en milieu organique (DMF-  $C_{16}H_{36}BF_4N$ , 0.1 M) sur une électrode de platine de 2 mm de diamètre.

**Mots clés** : voltampérométrie cyclique, base de Schiff, complexes, métal, électrode.

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[T2-PP 25]

**ELECTROCHEMICAL REDUCTION OF ALKYL HALIDES ON A  
GLASSY CARBON ELECTRODE MODIFIED BY  
ELECTRODEPOSITION OF A POLY- [CO (III) SALEN] FILM.**

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**Abstract:**

The tetradentate Schiff base cobalt complex was synthesized through the reaction of N,N-Bis[5-(N,N-methylphenylaminomethyl)methylsalicylidyne]-1,2-diaminoethane and a stoichiometric amount of tetrahydrated cobalt acetate in absolute ethanol at 50 °C under nitrogen atmosphere given the expected tetradentate Co(II)-Schiff base complex. The synthesized compound was characterized by different spectroscopic methods such as FT-IR, UV-Vis, mass spectrometry.

Cyclic voltammetry was also employed to investigate the redox behavior of the cobalt complex in dimethyl formamide (DMF) solution. The corresponding cobalt complex was then electropolymerized onto glassy carbon (GC) and indium tin oxide (ITO) electrode surfaces in the solution containing  $n\text{-Bu}_4\text{N}^+\text{ClO}_4^-$  (TBAP 0.1 M). These films were obtained by the successive cycling at the appropriate potentials.

The polymer, which was electrodeposited onto ITO substrates, was characterized by scanning electron microscopy (SEM) and dispersive energy X-ray spectroscopy. The electrocatalytic properties towards reduction of some alkyl halides such as bromocyclopentane, ethyl 2,3-dibromo-3-phenyl propanoate, and ethyl bromoethanoate have been examined.

**Key words:** Electrocatalytic reduction, Cobalt(II)-Schiff base complex, Cyclic voltammetry, Scanning electron microscopy (SEM),.



[T2-PP 26]

**ELECTROOXIDATION OF GLUCOSE AT A COPPER-POLYANILINE  
MODIFIED INDIUM TIN OXIDE**

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**Abstract:**

Polyaniline (PAni) is prepared electrochemically by anodic oxidation of aniline in aqueous sulfuric medium on indium tin oxide (ITO) substrate, which was followed by the incorporation of copper (Cu) microparticles within the polymer matrix. The morphology analysis of the Cu-PAni composite films shows that copper particles were uniformly dispersed over the polymer surface. The electrochemical reactivity of the prepared modified electrode was tested towards the oxidation of glucose in 0.1 M NaOH solution using cyclic voltammetry. It has been demonstrate that the proposed modified electrode can be successfully used for the glucose oxidation.

**Keywords:** polyaniline, anodic oxidation, copper, modified electrode, glucose.

[T2-PP 27]

**USE COPPER SUBSTITUTED HETEROPOLYANION AS CATALYST  
IN ELECTROCHEMICAL OXIDATION OF ORGANIC POLLUTANTS,  
APPLICATION IN THE REMOVAL OF NAPHTOL BLUE BLACK DYE  
FROM WATER**

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**Abstract:**

Dye removal from industrial effluents Textile effluent are superabundant source of colored pollutant that present an enhancing the environmental danger. Textile industry generates highly polluting waste water which contains dyes and their decomposition is creating very serious problem to waste water treatment plant.

In this study, we report a detailed discussion on the electrochemical oxidation of Naphthol blue black (NBB) in aqueous solution using a Dawson-type heteropolyanion copper substituted  $[H_{1,2}Cu_{2,4}P_2W_{12}Mo_6O_{61}, 21H_2O]$  as catalyst. This reaction is part of the treatment of polluted waters and in particular to the treatment of discharged water by the textile industry, soiled by the organic dyes. The choice of the naphthol blue black is dictated by the fact that it is an azo dye which presents a high toxicity to the environment because of the presence of the groups, phenolic, anilino, naphthalene and sulfonated. It is an industrially important acidic diazo dye, which has a high photo- and thermal- stability. The kinetics of the reaction was followed by UV-Vis spectrometry at wavelength of 618 nm. The mineralization of the dye was followed by the total organic carbon (TOC) removal. The maximum mineralization current efficiency values were obtained at 300mA in the presence of 0.02mM of catalyst.

**Keywords:** heteropolyanions, Electrooxidation, toxic dyes, water treatment,



[T2-PP 28]

## ELECTROCHEMICAL ELABORATION AND CHARACTERIZATION OF Cu<sub>2</sub>O, WHICH IS USED FOR RHB ELCTROCHEMICAL DETECTION

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### Abstract:

An electrochemical technique is employed for Cu<sub>2</sub>O deposition on copper substrate. The Cu<sub>2</sub>O films are formed by chronoamperometric. The deposition potential is chosen from the cyclic voltammetry, Cu<sub>2</sub>O films are homogeneous, uniform and well adhered. The characterization was done by X-ray diffraction (XRD), UV–Vis spectroscopy, electrochemical impedance spectroscopy (EIS) and The Mott–Schottky characteristic shows straight line with a negative slope, due to *p*-type conductivity. A flat band potential of 0.42 V<sub>SCE</sub> are obtained. The electrochemical impedance spectroscopy (EIS) spectra measured in the range (10<sup>-2</sup> –10<sup>5</sup> Hz). As application, electrochemical detection of rhodamine B (RhB), a recalcitrant dye.

**Keywords:** Cu<sub>2</sub>O Thin film; Electrochemical elaboration Electrochemical detection; Rhodamine B.

[T2-PP 29]

**SYNTHESIS OF NEW Cu<sup>(II)</sup> COMPLEXE OF CHALCONE  
DERIVATIVES: X-RAY CRYSTAL STRUCTURE,  
ELECTROCHEMICAL PROPERTIES AND HIRSCHFELD SURFACE**

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**Abstract:**

The reaction of copper (II) with 4-hydroxy-3-[(2E)-3-(1H-indol-3-yl)prop-2-enoyl]-6-methyl-2H-pyran-2-one (HL) lead to a new complexe: **Cu(L)<sub>2</sub>(DMF)<sub>2</sub>**. The crystal structure of the Cu(L)<sub>2</sub>(DMF)<sub>2</sub> complex have been determined by X-ray diffraction methods. The Cu(II) lying on an inversion centre is coordinated to six oxygen atoms forming an octahedral elongated. Additionally, the electrochemical behavior of the metal complexe was investigated by cyclic voltammetry at a glassy carbon electrode (GC) in CH<sub>3</sub>CN solution, showing the quasi-reversible redox process ascribed to the reduction of the M<sup>II</sup>/M<sup>I</sup> couple. The X-ray single crystal structure data of the complex was matched excellently with the optimized monomer structure of the desired compound; Hirschfeld surface analysis supported the packed crystal lattice 3D network intermolecular forces.

**Keywords:** cyclic voltametry, M<sup>II</sup>/M<sup>I</sup>, X-ray, Hirschfeld surface.



[T2-PP 30]

## PHOTOCATALYTIQUE DEGRADATION OF RHODAMINE B (RhB) DYE UNDER SOLAR LIGHT USING IRON DOPED ZnO ( $Zn_{1-x}Fe_xO$ ) NANOPARTICLES FABRICATED BY SOL-CEL METHOD

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### Abstract:

Fe doped ZnO with different percentages of Fe content ( $Zn_{1-x}Fe_xO$ ) thin films have been fabricated on ultrasonically cleaned quartz substrates by the sol-gel technique. The as-formed  $Zn_{1-x}Fe_xO$  nanopowder was characterized by Powder X-ray diffraction (PXRD), Scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), Raman, and UV- VIS. The as-formed nanopowder was used as photocatalyst for the degradation of the dye Rhodamine B (RhB), under solar irradiation, in an aqueous suspension at room temperature. The different characterization methods (DRX, FTIR, Raman) confirmed the formation of the ZnO compound of hexagonal structure (würtzite type). The visible light activity of  $Zn_{1-x}Fe_xO$  (1, 3, 5, 7) was identified through studies of phenol degradation under visible light photocatalysis, which highlight that  $Zn_{0.97}Fe_{0.03}O$  (2) shows the best activity. Typical degradation profiles indicated that the simultaneous degradation of pollutants is more effective than the removal of individual pollutants.

**Keywords:** photo-catalysis, pollutants,  $Zn_{1-x}Fe_xO$ , Rhodamine B, under solar irradiation



[T2-PP 31]

**MONONUCLEAR OXOVANADIUM (IV) COMPLEX: SYNTHESIS, SPECTROSCOPY, ELECTROCHEMISTRY AND ANTIBACTERIAL ACTIVITY.**

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**Abstract:**

The extensive variety of chemical, biological and catalytic properties of oxovanadium has created constant interest towards its coordination chemistry [1]. Among the transition metals, oxovanadium is incredibly precise due to its broad range of coordination numbers and geometries, available redox states, low toxicity, and involved in many enzymatic and metabolic processes [2]. Diimino ligands and the corresponding oxovanadium complexes have also played a considerable role in the development of chemistry. Thus, a large number of these compounds have been studied in the past because of their wide applications owing to their interesting properties as in electrocatalysis [3], inhibitors of corrosion [4], and biosensors [5]. Moreover the oxovanadium complexes are found to be of importance as analytical and antimicrobial agents [6]. Considerable efforts have been made to study the behavior and functions of these complexes in biological system [7] as well as in catalytic and pharmaceutical applications [8]. In this study, we have described synthesis of Schiff base ligand and its corresponding oxovanadium(IV) complex, which have been characterized by IR, UV-Vis spectroscopy, <sup>1</sup>HMRN and MS. The electrochemical properties of this complex have been investigated by cyclic voltammetry (CV) on a glassy carbon (GC) electrode in DMF solvent. The diffusion coefficient has been determined using the Levich plot on a GC rotating disk electrode. Furthermore, the bio-efficacy was tested *in vitro* against various strains of bacteria at different concentration levels to evaluate their antimicrobial activity.

**Keywords:** oxovanadium(IV), Schiff base, cyclic voltammetry, bio-efficacy

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[T2-PP 32]

**THIOSULPHATE ELECTROANALYTICAL DETERMINATION USING  
NICKEL SCHIFF BASE MODIFIED ELECTRODE.**

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**Abstract:**

Thiosulphate is a prime pollutant resulting from photographic, paper and textile industries. It is, therefore, necessary to develop a sensitive, fast low cost and user-friendly analytical method for its determination [1]. Different analytical methods such as iodometry are useful but take more time and not reliable.

We report there the use of carbon paste nickel complex modified electrode as reliable and sensitive electrode material for the determination of thiosulfate. Our electrode material was elaborated by mixing a water insoluble nickel Schiff base complex with graphite paste, the (N,N'-bisalicylidenepropylenetriamine)Ni (II) complex was synthesized and characterized by X-ray diffraction, the modified graphite paste electrode was characterized by scanning electron microscopy (SEM), energy dispersive X-Ray spectroscopy (EDXS), cyclic voltammetry and chronoamperometry and used in alkaline medium. A well defined Ni<sup>III</sup>/Ni<sup>II</sup> redox couple were obtained and a linear dependency of catalytic current was observed after addition of thiosulphate to the reaction medium. In conclusion the electrode has excellent sensitivity, good selectivity and remarkable stability.

**Keywords:**

Ni(II)-Schiff base complex modified graphite paste electrode, Cyclic voltammetry. Electrooxidation of thiosulphate, water cleaning

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[T2-PP 33]

ELECTROCHEMICAL CHARACTERIZATION OF THE MECHANISM OF  
OXIDATION OF A PHENYL UREA HERBICIDE BY CYCLIC VOLTAMMETRY

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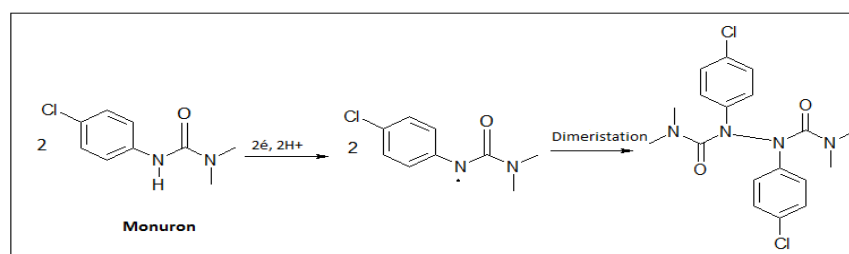
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**Abstract:**

Cyclic voltammetry was used to determine the electrochemical behavior and the mechanism of oxidation of a phenyl urea herbicide; 1-(4-chlorophenyl) -3,3-dimethylurea. The experiments were carried out in a three-electrode cell using carbon paste electrode as working electrode in a Phosphate buffer solution. At the potential range 0.4V to 1.2V the phenyl urea herbicide exhibits one irreversible anodic peak at potential  $E_p = +0.85V$ . The effect of pH on the potential of the oxidation peak was investigated, the peak potentials were found to shift linearly towards more negative values with the increase in pH, indicating the intervention of protons in the electrochemical process, the slope value of the curve almost matches the theoretical Nernst equation, suggesting that the same numbers of protons and electrons are involved in the electrochemical oxidation ( $n_e = n_H$ ). The effect of scan rate ( $v$ ) on the peak ( $I_p$ ) and potential ( $E_p$ ) of the oxidation peak was also studied, the linear relation between the scan rate and the peak current indicates that the oxidation of monuron is an adsorption-controlled process.  $\alpha$  the anodic transfer coefficient and  $n$  the number of electrons involved in the oxidation were calculated experimentally using the slope of the peak potential versus the logarithm of the scan rate, they were equal to 1 and 0.488, respectively. According to the previous results the proposed mechanism for the oxidation of monuron, it thought to involve one electron and one proton removal pathway to produce a free cationic radical intermediate which in turn dimerises.



Scheme: Mechanism of the oxidation of monuron

**Keywords:** carbone paste electrode, herbicide, phenylurea, electro-oxidation

## Topic 3: Corrosion and Electrochemical Engineering.

### [T3-PP 34]

#### ETUDE DE L'EFFET INHIBITEUR DES EXTRAITS AQUEUX DES PLANTES ORTIE ET AVOINE SUR LA CORROSION DE L'ACIER DOUX EN MILIEU NaCl A 3 %

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#### Résumé:

L'acier, métal couramment utilisé au vu de son coût peu élevé et de ces propriétés mécaniques intéressantes, se trouve sérieusement affecté par le phénomène de corrosion qu'il soit au contact d'atmosphères humides, immergé en eau douce ou saline, implanté dans les sols ou en présence de solutions plus ou moins agressives. L'adoption de mesures préventives contre la corrosion est donc nécessaire et l'utilisation des inhibiteurs est une méthode adaptée et pratique pour protéger les métaux. Ces inhibiteurs peuvent interférer avec la réaction anodique ou cathodique et forme une barrière protectrice sur la surface du métal contre les agents corrosifs. L'utilisation d'inhibiteurs verts a été largement plébiscitée au cours de cette dernière décennie ; leur utilisation est actuellement préférée à celles d'inhibiteurs inorganiques pour des raisons d'écotoxicité essentiellement.

L'objectif de ce travail est de tester une nouvelle formulation inhibitrice qui soit à la fois efficace, économiquement rentable et en accord avec les réglementations concernant l'environnement. Les inhibiteurs testés sont des extraits bruts de deux plantes, à savoir : l'ortie et l'avoine. Après la cueillette des plantes et leurs mises en condition (séchage, broyage, tamisage, etc.), les extraits sont obtenus par la méthode à reflux dans l'eau. Ces extraits sont ensuite utilisés comme inhibiteurs de corrosion de l'acier doux dans un milieu aqueux connu pour son agressivité vis-à-vis de ce métal, à savoir le chlorure de sodium (NaCl à 3%). Cette étude a été réalisée par des techniques électrochimiques et gravimétriques.

Les résultats obtenus ont montré que ces extraits inhibent de manière satisfaisante la vitesse de dissolution du fer. L'efficacité d'inhibition augmente avec la concentration des inhibiteurs et atteint 84 % à 0,3 g.L<sup>-1</sup> d'extrait d'ortie et 72 % à 5 g.L<sup>-1</sup> d'extrait d'avoine. Les données de polarisation indiquent que l'extrait d'ortie est de type anodique, tandis que l'extrait d'avoine est de type cathodique. L'adsorption des inhibiteurs sur la surface de l'acier doux dans NaCl à 3% suit l'isotherme d'adsorption de Langmuir. L'étude de l'effet de la température (20–80 °C) sur le comportement de l'acier doux dans le milieu NaCl à 3% en absence et en présence des extraits montre que l'efficacité inhibitrice reste stable dans cet intervalle de température. L'analyse MEB de l'état de surface des électrodes confirme l'adsorption des inhibiteurs en recouvrant de manière efficace la surface des électrodes. Les énergies d'activation correspondantes en absence et en présence des inhibiteurs ont été également déterminées.

**Mots clés:** Corrosion, Acier, Ortie, Avoine, Inhibiteur.

[T3-PP 35]

**CORROSION INHIBITION OF CARBONE STEEL XC52 IN  
HYDROCHLORIC ACIDE BY LAUNAEA RESEDIFOLIA EXTRACT**

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**Abstract:**

Green corrosion inhibitors are biodegradable and free of heavy metals and other toxic compounds. Some research groups have reported the successful use of naturally occurring substances to inhibit the corrosion of metals in acidic and alkaline environments. Among the various eco-friendly inhibitors, the plant extract are becoming more and more popular. In this study, the inhibitive and adsorption properties of the aerial parties extract of *Launaea resedifolia* as corrosion inhibitors of carbon steel XC52 in 1M HCl were investigated using weight loss method of monitoring corrosion.

The corrosion rate was found to decrease with increasing inhibitor concentration. The adsorption mechanism between the extract and the mild steel substrate is found to obey Langmuir and Temkin isotherms. There was also a positive correlation between temperature and corrosion rate.

**Keywords:** *Launaea resedifolia*; Corrosion inhibition; weight loss method; carbon steel; adsorption.

[T3-PP 36]

LA PROTECTION DE L'ACIER CONTRE LA CORROSION

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**Abstract:**

Nous nous sommes intéressés à la synthèse des polymères conducteurs intrinsèques, et plus particulièrement des produits à base de polyaniline et de poly (N-méthylpyrrole) ainsi qu'à leur utilisation pour la protection de l'acier contre la corrosion en milieu NaCl 3.5%.

Dans un premier temps, nous avons effectué une synthèse électrochimique par voltamétrie cyclique, dans un milieu acide, de plusieurs films en faisant varier plusieurs paramètres opératoires à savoir la nature du monomère, sa concentration et le nombre de cycles de synthèse. L'étude électrochimique basée sur polarisation a montré que ces polymères fonctionnalisés ont l'avantage de déplacer le potentiel de corrosion soit anodiquement soit cathodiquement, et cela en fonction de la nature de chaque polymère et le nombre de cycles utilisés pour sa synthèse.

Les revêtements obtenus dans les différentes conditions présentent une protection de l'acier et l'augmentation du nombre de cycles améliore considérablement la protection contre la corrosion de l'acier, notamment le PNMPy et le copolymère. Cette amélioration pourrait être le résultat d'une auto-cicatrisation de la couche passive sous jacente. En effet, lorsque la surface de l'acier est attaquée par des agents corrosifs, le polymère conducteur se réduit et il en résulte l'expulsion des contre anions. Ces derniers une fois libérés peuvent auto-réparer le film par formation «*in-situ*» d'une couche passive à l'endroit des lésions.

**Keywords:** Polypyrrole, aniline, Corrosion, Protection, Acier.

[T3-PP 37]

**CORROSION DES CONDUITES DE TRANSPORT DU PETROLE PAR  
LES BACTERIES**

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**Résumé:**

Nous avons étudié le mode d'action des biocides et les conditions de leurs actions sur les bactéries sulfato-réductrices. Ces derniers sont les principales responsables des phénomènes de corrosion en absence d'oxygène. Plusieurs types de corrosion sont constatés au niveau des circuits de transport du pétrole au champ de Hassi Messaoud: corrosion localisée due à la présence d'oxygène dans les circuits fermés, corrosion sous dépôt ou aération différentielle rencontrée souvent au niveau des dessaleurs, des échangeurs et à l'intérieur des conduites et corrosion en crevasse due à la présence du fer. Les BSR peuvent agir soit par contact direct avec le métal en utilisant l'hydrogène formé à la surface soit à distance en consommant l'hydrogène gazeux. Lorsqu'on ajoute un aldéhyde, un biocide ou un mélange à un milieu de culture contenant une population bactérienne, il est important de comprendre à quel niveau agit-il sur les microorganismes car après un temps de contact d'adaptation, les substances sont consommées et le processus n'est pas enrayé. L'étude constitue une contribution à la compréhension de l'action corrosive des bactéries et leurs activités hydrogénase dans les eaux d'injection et les eaux contenues en émulsion. Des tests d'évaluation de l'efficacité des biocides, pour remédier à la corrosion des conduites transportant du pétrole et l'acheminement de ce dernier, ont été effectués ainsi que leur mode d'action. Des mesures de pH du milieu de culture en fonction du temps et de la forme des pièces métalliques ont été étudiées. Des courbes de cinétique de croissance des BSR dans différents milieux en présence de biocides ont été aussi réalisées. La seconde partie de ce travail s'intéresse au phénomène de corrosion en étudiant l'élimination des gouttelettes d'eau contenant les BSR. En effet l'injection de biocides est favorisée en présence d'émulsion stable à base d'ammonium quaternaire qui permet d'augmenter la surface de contact et abaisser la tension superficielle ce qui facilite l'entraînement des gouttelettes d'eau.

**Mots clés:** Biocorrosion; bactéries sulphato-réductrices; Biocides; Energie d'activation; Emulsion.

[T3-PP 38]

## EFFECT OF OXALATE IONS ON THE PITTING CORROSION BEHAVIOUR OF STEEL REBAR IN SIMULATED CONCRETE PORE SOLUTION

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### Abstract:

Corrosion of reinforcement steel, induced by chlorides ions penetration, is the main cause of concrete structures damage. Because of its serious economic and social consequences, the protection against this phenomenon remains a major challenge for many countries. Thus, various methods to combat this problem have been proposed, among them the use of corrosion inhibitors has become a more attractive alternative due to its effectiveness, ease of use and low cost price.

The aim of this work is to study the inhibitive effect of sodium oxalate on the pitting corrosion behavior of steel rebar in a simulated concrete pores solution, using different electrochemical techniques: corrosion potential measurement, potentiodynamic and electrochemical impedance spectroscopy tests.

The obtained results indicate that the addition of oxalate ions to the chlorinated solution decreases significantly the steel corrosion rate due to their role of reducing agent. So, in the presence of these ions, the measured electrochemical parameters ( $E_{\text{corr}}$ ,  $E_{\text{pit}}$ ,  $I_{\text{corr}}$ ,  $R_p$ ) evolve in the direction of a clear improvement of the corrosion resistance of steel rebar. Surface observations have confirmed this trend. The optimal inhibition rate is obtained for an oxalate ion concentration corresponding to  $[\text{C}_2\text{O}_4^{2-} / \text{Cl}^-]$  equal to 0.25.

**Keywords:** steel, concrete, corrosion, chlorides, inhibitors, sodium oxalate.



[T3-PP 39]

**COMPUTATIONAL AND EXPERIMENTAL STUDIES ON THE  
INHIBITION EFFICIENCIES OF TYROSINE AND PHENYLALANINE  
AS GREEN INHIBITORS FOR MILD STEEL IN ACIDIC MEDIUM**

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**Abstract:**

Inhibition efficiencies of Tyrosine(Tyr) and Phenylalanine(Phe), two amino acids products, have been studied as green corrosion inhibitors on corrosion of mild steel in acidic medium(solution of 0,1M of H<sub>2</sub>SO<sub>4</sub>). Experimental results showed that these products suppressed both anodic and cathodic reactions and have an interesting inhibitory property despite the hostile environment. In this case, for all concentrations used, the inhibition efficiencies followed the order : Tyr > Phe.

In order to further study the corrosion mechanism, quantum chemical parameters such as E<sub>HOMO</sub> (highest occupied molecular orbital energy), E<sub>LUMO</sub> (lowest unoccupied molecular orbital energy), the energy gap ( $\Delta$ ), Softness (s) and others parameters have been calculated at the **B3LYP** level of theory with 6-31++G(d,p) basis set, using density functional theory (**DFT**) method in protonated forms and aqueous phase.

The results obtained indicate a good agreement between some quantum chemical parameters and the experimental inhibition efficiencies. Also, the results showed that these two inhibitors can adsorb on mild steel surface by donor acceptor interactions between lone-pair electrons of heteroatom and vacant d-orbital of iron.

**Keywords:** Simulation of Corrosion inhibition, Amino acids, Inhibitory efficiency, Mild Steel, acidic medium, DFT.

[T3-PP 40]

**ELECTROCHEMICAL MEASUREMENTS FOR THE CORROSION  
INHIBITION OF COPPER IN 1M HYDROCHLORIC ACID BY USING  
THE POLLEN**

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**Abstract:**

The influence of Pollen as a green inhibitor as a corrosion inhibitor on copper in 1M Hydrochloric acid was studied by tafel polarization, linear polarization resistance (LRP) and electrochemical impedance spectroscopy (EIS) techniques. The potential of zero charge (PZC) of the copper samples in inhibited solutions were determined with the help of EIS. Surface of copper before and after exposing to test solution were examined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The results showed that the inhibition efficiency (IE%) of Pollen increased with increasing inhibitor concentrations, and the study inhibitor has a mixed character. The negative value of  $E_r$  calculated by the PZC method shows that the surface of copper is negatively charged in the inhibited solution, and the Pollen molecules can adsorbed through electrostatic interactions between the negatively charged metal surface and the positively charged inhibitor molecules, which forms a protective film and reduces the metal dissolution. The formation of protective film was further confirmed by SEM and AFM.

**Keywords:** corrosion, copper, Pollen, SEM, AFM



[T3-PP 41]

**LAURUS NOBILIS AS ECO-FRIENDLY CORROSION INHIBITORS  
FOR MILD STEEL IN ACIDIC SOLUTION.**

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**Abstract:**

Corrosion prevention plays an important role in the field of scientific research. In this context, researchers and industrials work together to develop alternative and innovative solutions to prevent metals from corrosion. The use of inorganic and organic inhibitors is one of the best methods due to their cost effectiveness and ease of application in industry, but unfortunately many of these inhibitors are highly toxic. That's why current research has focused on "Green chemistry" with the research of environmentally friendly materials especially those from natural resources as green corrosion inhibitors due to their natural origins and non-toxic nature. Plant extracts are ideal green candidatures to replace traditional corrosion inhibitors and to reduce the environmental risk. The plant extracts are the suitable candidates to replace the expensive and toxic traditional synthetic corrosion inhibitors.

The main objective of the present work is to promote a plant "laurus nobilis" of Jijel department by using these leaves as green corrosion inhibitor of mild steel in acid medium (HCl 1M). The anti-corrosion action of the extracts and essential oil are evaluated by electrochemical impedance spectroscopy and polarization measurements. The study allowed us to highlight the existence of a good inhibitory efficiency in acid medium (1M HCl). The efficiency of each extract phase increases as the concentration increases. The surface morphology of mild steel with and without extract was analyzed by scanning electron microscopy (SEM) and confirmed the presence of a protective layer formed with the addition of the extracts, thus confirming the good electrochemical results obtained.

Finally, the quantum calculations using Density Functional Theory (DFT) method is performed to define the relationship between inhibition performance of investigated compound and their molecular structure. This study shows an obvious correlation between the molecular surface of the molecules and the inhibition efficiency.



[T3-PP 42]

## THEORETICAL AND EXPERIMENTAL STUDY OF COPPER CORROSION BY THIOCYANATE INHIBITOR

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### Abstract:

This study is conducted around the corrosion inhibition of a copper in a mixture acidic solution of HCl and HNO<sub>3</sub> by an inhibiting formulation thiocyanates-based that was composed, synthesized, and characterized from DRX and IR at the laboratory. On the other hand, a comparative study was adopted to check the efficiency of the compounds Zn(SCN)<sub>2</sub>(Tu)<sub>2</sub>, Zn(SCN)<sub>2</sub>(An)<sub>2</sub>, Zn(SCN) and bromide of tetra-pentyl-ammonium. Steady-state current-voltage curves were combined with, electrochemical impedance spectroscopy to characterize the inhibiting properties of each compound and to optimize the concentration of the compounds. The results found proved that Zn(SCN)<sub>2</sub>(An)<sub>2</sub> was the most effective inhibitor. The later is effective of at 77, 19% with a concentration of 200 ppm. Nonetheless, the effectiveness of inhibiting and the mechanism of inhibition of the four examined inhibitors were discussed from a theoretical study by Monte-Carlo simulation. The results obtained, however, starting from simulation; polarization and impedance are in accordance.

**[T3-PP 43]**

**INHIBITION EFFECT OF METHYLENE BIS [(2-HYDROXY-5, 1-PHENYLENE) METHYLENE]} BIS PHOSPHORIC ACID ON THE CORROSION OF COPPER IN 0,5M HCL MEDIUM**

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**Abstract:**

Inhibition of copper corrosion was studied in 0,5M HCl medium by a new molecule derived from phosphonate ({methylene bis [(2-hydroxy-5, 1-phenylene) methylene]} bis phosphoric acid (ADP)).

Gravimetric and electrochemical studies revealed significant inhibition of corrosion in the presence of ADP. The inhibition efficiency increases with the concentration of ADP to attain 95% at  $10^{-6}$  M. Potentiometric polarization curves show that ADP acts essentially as a mixed-type inhibitor.

To better approximate the inhibition mechanism, impedance spectra were plotted at different concentrations of the inhibitor. The impedance spectra plotted after 30 minutes of immersion are characterized by the appearance of a one loop at the high frequencies attributed to the film, which shows slow film formation kinetics. XRD analysis indicates peaks attributed to phosphorus and carbon sign of the presence of the molecule.

The adsorption of the inhibitor was spontaneous ( $\Delta G < 0$ ), supported the physical/chemical adsorption mechanism and obeyed to the Langmuir adsorption isotherm. The thermodynamic functions of adsorption and dissolution processes were calculated.

**Keywords:** Copper, Corrosion inhibitor, Phosphonate, Electrochemical characterization, Impedance spectroscopy.

[T3-PP 44]

**ELECTROCHEMICAL BEHAVIOR OF Ti-6Al7Nb ALLOY FOR  
TOTAL HIP PROSTHESIS APPLICATIONS**

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**Abstract:**

The aim of the present work is to evaluate the corrosion behaviour of Ti-6Al7Nb alloy in different simulated body fluid (NaCl, PBS and hank's solution), using potentiodynamic polarization and electrochemical impedance spectroscopy (EIS), studies to ascertain its suitability for biomedical applications (total hip prosthesis). The study reveals that there is a strong dependence of the corrosion resistance of Ti-6Al7Nb alloy on the chemical composition of the simulated body fluid. Increase in Ca and Pa ion concentration from Hank's to PBS's solution shifts the corrosion potential ( $E_{corr}$ ) from  $-245,3$  to  $-362,1$  mV vs. SCE. Shifts the corrosion current density ( $i_{corr}$ ) from  $1.05$  to  $0.49$   $\mu\text{A}/\text{cm}^2$ . Increases the double-layer capacitance ( $C_{dl}$ ) from  $42.16$  to  $54,17$  ( $\mu\text{F}.\text{cm}^{-2}$ ). and reduces the charge transfer resistance ( $R_{ct}$ ) from  $18,64$  to  $16,69$  ( $\Omega$ ). In spite of the active dissolution, the Ti-6Al7Nb alloy exhibit passivity at anodic potentials at all studied simulated body fluids.

**Keywords:** Ti-6Al7Nb; electrochemical behavior; simulated body fluid; Total hip prosthesis.

[T3-PP 45]

**ELECTROCHEMICAL BEHAVIOR OF FLAME SPRAYED NICKEL-BASED COMPOSITE COATINGS IN NaCl AND Na<sub>2</sub>SO<sub>4</sub> AQUEOUS SOLUTIONS**

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**Abstract:**

Owing to the heterogeneous nature of thermally sprayed coatings, the assessment of corrosion behavior of these materials remains an important assignment in order to avoid sudden breakdowns in ion-containing environments. The present investigation is aiming at determining the effect of ion concentration on the electrochemical response of flame sprayed NiCrBSi-WC cermet in NaCl and Na<sub>2</sub>SO<sub>4</sub> aqueous solutions with concentrations ranging from 1 g/l to 35 g/l. The experimental study has involved cyclic potentiodynamic polarizations, open circuit measurements, electrochemical impedance spectroscopy analysis and various metallographic observations, conducted on coated X18steel substrates. The electrochemical tests allowed the estimation of parameters that govern the corrosion process. Additionally, the metallographic observations permitted the determination of the coating microstructure and the evaluation of the degradation threshold of the coatings after polarization tests. The results revealed that the chloride containing mediums engendered drastic pitting corrosion after two cycles of potentiodynamic polarization. The pits have the trend to be initiated at various borides and carbides containing sites. They may severally propagate in-depth and along a limited number of grain boundaries. The results confirmed that the nickel phases in both eutectic and dendritic areas are all very prone to general corrosion when compared with Ni<sub>3</sub>B eutectic phase. Moreover, the EIS curves proved that the increase in ion concentration augments the charge transfer at the coating/electrolyte interface and accelerates corrosion. In the sulfur solutions, the coatings form a thin and compact passive film layer that makes the charge transfer permanently constant at high concentrations.

**Keywords:** Composite, Cyclic Potentiodynamic Polarization, OCP, EIS, Microstructure.



[T3-PP 46]

**CORROSION INHIBITION OF MILD STEEL IN HCl SOLUTION BY  
THE EXTRATS OF JUNIPERUS PHOENICEA.**

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**Abstract:**

The possibility to use green corrosion inhibitors became currently an interesting alternative compared to other protective methods. This work consists in extracting the Juniperus phoenicea in order to use it like steel corrosion inhibitor in hydrogen chloride solution at 0,5M. The extracting the Juniperus phoenicea reduces the corrosion rate and the inhibition efficiency increases with its concentration and attains 71, 54% à 1,25 g/l.

The temperature effect on the corrosion, behavior of mild steel without and with inhibitor, and the associated activation energy have been determined and proved that the inhibitor interacts on the steel surface by electrostatic effect. The inhibitor was adsorbed on the steel surface according to the modified Freundlich adsorption isotherm model.

The SEM allowed the visualization of an adherent and stable layer on the surface of the steel.

**Key words:** Corrosion, extracting the Juniperus phoenicea, adherent and stable layer, Freundlich.



[T3-PP 47]

**STUDY OF THE INHIBITOR EFFECT OF A TRIAZOLE DERIVATIVE  
WITH RESPECT TO THE CORROSION OF MILD STEEL IN 0.5M  
H<sub>2</sub>SO<sub>4</sub> MEDIUM**

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**Abstract:**

Acidic solutions are widely used in industry, the main fields of application being acid stripping or cleaning, stimulation of oil wells, elimination of localized deposits (non uniformly distributed scale, rust, deposits bacterial, etc.), as well as in many industrial synthesis processes. Due to the aggressiveness of these acidic solutions, the use of corrosion inhibitors has become essential to limit the attack of metallic materials.

Triazole derivatives are generally excellent organic inhibitors of corrosion in an acid medium. In this work, we are interested in the anti-corrosive study of mild steel by an organic compound of the triazole-thione type in 0.5M H<sub>2</sub>SO<sub>4</sub> medium by using the method of weight loss and electrochemical methods (curves of Polarization and Impedance Spectroscopy (EIS)). We studied the effect of concentration and temperature on the inhibition rate. The work conducted on the triazole derivative revealed that this inhibitor is effective against the corrosion of mild steel in 0.5M H<sub>2</sub>SO<sub>4</sub> medium. We noted that the inhibitory efficacy increases with the inhibitor concentration to reach optimal values equal to 91% for a concentration of  $5 \times 10^{-4}$  M.

**Keywords:** Triazole, Mild Steel, Inhibitor, Corrosion, Sulfuric Acid



[T3-PP 48]

**ETUDE DE LA CORROSION DE L'ACIER DE PIPELINE API 5L X52  
DANS LA SOLUTION NS4**

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**Abstract:**

De nos jours, la détérioration des conduites souterraines par le phénomène de corrosion est devenue une préoccupation croissante des secteurs industriels et environnementaux à cause des agents agressifs présents dans le sol environnant du pipe, des conditions climatiques locales, et les teneurs en oxygène...etc. En effet afin de garantir un fonctionnement sûr des conduites, il est important d'étudier leurs comportements à la corrosion électrochimique dans un environnement de sol simulé. Pour cela la corrosion électrochimique de l'acier API 5L X52 a été étudiée dans une solution de sol simulé (NS4) par spectroscopie d'impédance électrochimique (SIE) et par polarisation potentiodynamique, en variant le temps d'immersion et le pH. Il a été montré que lorsque le temps d'immersion augmente la densité de courant de corrosion augmente et inversement la résistance de polarisation diminue. Pour l'effet du pH les résultats ont indiqué une accélération de la corrosion pour des valeurs de pH acides et alcalines.

**Mots clés:** Acier X52 ; Corrosion ; Solution NS4; Spectroscopie d'impédance électrochimique; Polarisation potentiodynamique.

[T3-PP 49]

**NEW SYNTHESIZED SCHIFF BASE AS INHIBITOR OF MILD STEEL  
CORROSION IN ACID MEDIUM: EXPERIMENTAL  
AND COMPUTATIONAL STUDY**

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**Abstract:**

In order to prevent corrosion, the primary strategy adopted is to isolate the metal from corrosive media. It is a well-known fact that acids play crucial roles in our daily lives due to their excellent properties. For this reason it has recently been reported that a new potential class of corrosion inhibitors has recently emerged. A new corrosion inhibitor namely, 4,4'-bis(4-diethylaminosalicylaldehyde) diphenylethanediiimine (L2) has been synthesized and its corrosion inhibition on mild steel in 1.0 M HCl was studied through experimental and theoretical measurements. Weight loss, Electrochemical impedance spectroscopy and Potentiodynamic polarization results indicate that L2 is an excellent inhibitor. It gives a maximum inhibition efficiency of 95.33 % at  $5 \times 10^{-4}$  M. Polarization study suggested that L2 acts as mixed type inhibitor with some cathodic predominance. Meanwhile, the adsorption process obeys the Langmuir adsorption isotherm model, and thermodynamic parameters were calculated and discussed. Scanning electron microscopy studies revealed the formation of a protective inhibitor layer on the mild steel surface. Quantum chemical parameters obtained using the density functional theory (DFT) supported the experimental results. Local reactive sites of the present molecule have been analyzed through Fukui indices.

**Keywords:** Corrosion Inhibitors, Weight Loss, Electrochemical Measurements, Quantum Chemical parameters

[T3-PP 50]

## CORROSION INHIBITION OF STEEL XC48 IN CHLORYDRIC ACIDIC SOLUTION BY A IMINIC COMPOUND

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### Abstract:

Steel is an important material widely used in various industries. Hydrochloric acid and sulphuric acid are the medium generally being used for pickling of steel. Generally, acids cause damage to the substrate, because of their corrosive nature. Thus, protecting steel from corrosion is the most important problem. Several methods were used to decrease the corrosion of metals in acidic medium, but the use of inhibitors is most commonly used. Hence, the corrosion process may be censored by the protective film of inhibitor on the metal surface. Majority of the organic inhibitors for the corrosion of steel in different aggressive media are the organic compounds containing N, S, O and P atoms or N-hetero cyclic compounds with polar groups. These groups of atoms or bonds facilitate electronic interactions between organic corrosion inhibitors and metal surface thereby aid adsorption of the inhibitors onto metal surface.

In the present study we report the effect of the addition of *a iminic compound* on the behavior of carbon steel corrosion in 1M chlorydric acid solution. The inhibition effect of the compound on XC48 was studied using potentiodynamic polarization and electrochemical impedance spectroscopy. Results show that inhibition efficiency increases when the inhibitor concentration increases. The inhibition efficiencies obtained from all methods employed are in good agreement with each other. The adsorption of this compound on a steel surface from 1M HCl acid obeyed the Langmuir adsorption isotherm.

**Keywords:** Corrosion, Inhibitor, Schiff base, potentiodynamic polarization, electrochemical impedance spectroscopy.

[T3-PP 51]

**ÉLECTRODEPOSITION D'ALLIAGES DE ZN-SN DANS UN BAIN DE SULFATE.**

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**Résumé :**

La corrosion pose de graves problèmes et grands dégâts que ce soit sur le plan matériel ou économique. Les conséquences sont importantes dans divers domaines et en particulier dans l'industrie : arrêt de production, remplacement des pièces corrodées, accidents et risque de pollutions, risque à la santé humaine....etc.

Le zinc est un métal de plus en plus utilisé vu sa faible densité et ses bonnes propriétés mécaniques. Il est l'élément le plus électropositif parmi les métaux industriels (magnésium, aluminium, cadmium, fer, plomb, étain, nickel, cuivre, argent or et platine). Lorsqu'il est utilisé comme revêtement, il se comporte comme une anode sacrificielle et protège le substrat contre la corrosion.

Ces dernières années, l'électrodéposition des métaux sur différents substrats a été largement étudiée [1,2]. Les dépôts obtenus sont couteux, plus faciles à mettre en œuvre (température et pression ambiantes).

L'objectif de ce travail est d'obtenir par électrodéposition, des couches protectrices d'alliages zinc-étain à différentes teneurs en étain sur l'acier. L'étude consiste à tester différents paramètres d'électrodéposition, à savoir l'effet de la concentration d'étain, du potentiel cathodique afin d'obtenir un meilleur rendement des dépôts et d'évaluer ensuite, par étude électrochimique, la résistance des dépôts de la corrosion.

**Mots clés :** Zinc, corrosion, anode sacrificielle, électrodéposition et acier.

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[T3-PP 52]

## SCHIFF BASE AS CORROSION INHIBITOR FOR XC48 IN HYDROCHLORIC ACID SOLUTION

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### Abstract:

Acid solutions are widely used in industry, such as acid pickling of iron and steel, chemical cleaning and processing, ore production and oil well acidification. Use of inhibitors is one of the most practical methods for protection against corrosion especially in acid solutions to prevent unexpected metal dissolution and acid consumption. Different organic and nonorganic compounds have been studied as inhibitors to protect metals from corrosion attack. Usually, organic compounds that exert a significant influence on the extent of adsorption on the metal surface and therefore can be used as effective corrosion inhibitors. The efficiency of these organic corrosion inhibitors is related to the presence of polar functions with S, O or N atoms in the molecule, heterocyclic compounds and p electrons. Inhibitors molecules adsorbed on surface of mild steel, forming a barrier and consequently preventing reactions (cathodic or anodic) from processing at the surface of mild steel. These inhibitors could react with the iron atom at the mild steel surface to form in-organic complexes, blocking the surface of mild steel. The corrosion inhibition and adsorption behavior of a novel Schiff base on XC48 steel in hydrochloric acid was investigated using the potentiodynamic polarization and electrochemical impedance spectroscopy. Results show that the inhibition efficiency increases when the inhibitor concentration increases. Potentiodynamic polarization curves reveal that the used Schiff base is mixed-type inhibitor. Experimental data indicate that these Schiff base inhibitor adsorb at the XC48 steel/solution interface according the Langmuir adsorption isotherm.



**[T3-PP 53]**

**THE INHIBITION ACTIVITY OF 1,10 - BIS(2-FORMYLPHENYL)-  
1,4,7,10- TETRAOXADECANE (ALD )**

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**Abstract:**

The use of corrosion inhibitor is one of the most effective measures for protecting metal surfaces against corrosion in acid environments. Some organic compounds are found to be effective corrosion inhibitors for many metals and alloys. Generally, inhibitor molecules may physically or chemically adsorb on a metal surface forming an adsorption layer that functions as a barrier protecting the metal from the corrosion.

Acid solutions are widely used to remove unwanted sediments and rust in many industrial processes. Adsorption depends on the charge of the metal but on the chemical structure of the inhibitor. Organic compounds containing heteroatoms such as N, O and S have been reported as being efficient corrosion inhibitors for metals.

The aim of the present work is to investigate the inhibitory action of aldehyde, named 1,10 - bis(2-formylphenyl)-1,4,7,10- tetraoxadecane (**Ald**).

The research was done on carbon steel XC48 in HCl 1M aggressive medium by electrochemical methods and weight loss. The compound is an efficient corrosion inhibitor and their inhibition efficiency are increased with increasing inhibitor concentration



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**[T3-PP 54]**

**ETUDE DE LA POLARISATION ANODIQUE DU ZINC EN MILIEU  
NaCl**

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**Résumé :**

L'objectif de ce travail est d'une part manipuler avec la méthode de polarisation du zinc pur pour obtenir des couches passives qui peuvent avoir des compositions chimiques dotées de composés très intéressants comme le ZnO. D'autre part déterminer les conditions (pH, vitesse de balayage, concentration de l'électrolyte ...) optimales pour réaliser ces couches. On s'intéresse plus particulièrement à la composante ZnO dans le film passif vue son intérêt et son application dans divers domaines.

Les caractérisations des films passifs obtenues sont faites par la diffraction des rayons X, la microscopie à force atomique (AFM), et la photoluminescence.





[T3-PP 55]

**QUANTUM CALCULATIONS AND SPECTROSCOPIC  
INVESTIGATION ON {[ (4-HYDROXY-PHENYL)-  
PHOSPHONOMETHYL-AMINO]-METHYL}-PHOSPHONIC ACID BY  
DENSITY FUNCTIONAL METHOD**

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**Abstract:**

It is important to note that modeling and simulations are becoming increasingly popular in the field of corrosion inhibition of metal surfaces. In this work, we investigated the corrosion inhibition efficiencies of {[ (4-Hydroxy-phenyl)-phosphonomethyl-amino]-methyl}-phosphonic acid against the corrosion of iron metal using DFT approach. Observable correlation was found between corrosion inhibition efficiency and quantum chemical parameters, using DFT method.

**Keywords:** Aminophosphonic acid, DFT; HOMO, LUMO, Fukui indices; Metal Protection; Corrosion Inhibitors.

[T3-PP 56]

**ETUDE COMPARATIVE DE DEUX INHIBITEURS DE CORROSION  
SUR ACIER AU CARBONE EN MILIEU ACIDE**

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**Résumé :**

La corrosion est un phénomène de dégradation des matériaux métalliques sous l'influence des milieux très acides et très oxydants [1-4]. L'utilisation des inhibiteurs est une méthode adaptée et pratique pour protéger les métaux. En matière de protection, les inhibiteurs bases de Schiff constituent un moyen original pour lutter contre la corrosion. Les composés organiques contenant des liaisons insaturées et /ou des atomes polaires comme O, N, S et P, sont généralement considérés comme étant des inhibiteurs de corrosion efficaces [5-7]. Les bases de Schiff ont ainsi des propriétés inhibitrices importantes en raison de la présence du groupement ( $-C=N-$ ) [8-11]. Les calculs chimiques quantiques et l'utilisation des simulations dynamique moléculaire sont des outils utiles et modernes pour étudier l'interaction entre les inhibiteurs et la surface métallique [12-14]. Notre travail a pour objectif, la synthèse, la caractérisation spectroscopique de nouvelles molécules bases de Schiff et leurs applications comme inhibiteurs de corrosion de l'acier au carbone en milieu acide chlorhydrique 1M. Des mesures électrochimiques et théoriques ont été effectuées pour réaliser cette étude.

L'application de ces bases de Schiff comme inhibiteurs de corrosion pour la protection de l'acier en milieu acide chlorhydrique 1M a montré que:

Les efficacités inhibitrices, obtenues à partir des mesures d'impédance électrochimique et des mesures de polarisation potentiodynamique sont en bon accord. Les calculs théoriques ont mis en évidence l'existence d'une bonne corrélation entre la structure moléculaire des inhibiteurs étudiés et leur pouvoir inhibiteur.

**Mots-clés :** Electrochimie organique, base de Schiff, inhibiteur de corrosion, DFT.

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[T3-PP 57]

**FREE-CYANIDE SYNTHESIS AND CHARACTERIZATION OF Cu–Zn  
ALLOY BY AN ELECTRODEPOSITION-ANNEALING ROUTE USING  
ZINC CHLORIDE BATH**

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**Abstract:**

Substitution of cyanide in electroplating is a current challenge. We present an alternative method aiming to reduce the toxicity and the cost of electroplating of Cu-Zn alloy (usually prepared from cyanide baths) while maintaining the decorative qualities and anticorrosive properties of the coating.

For this purpose, Cu-Zn alloys were obtained in two steps from non-cyanide electrolytes. First, a copper layer electrodeposited onto a nickel under-layer, followed by a thin layer of zinc from Zinc chloride bath. The Zn/Cu/Ni sandwich system was then subjected to heat treatment, to ensure the diffusion of zinc into the copper layer to give the desired Cu-Zn alloy structure.

The synthesized films were characterized by using X-ray diffraction XRD, scanning electron microscopy and energy dispersive X-ray spectroscopy (EDS). XRD demonstrated that the electrodeposited film are crystalline and present the  $\text{Cu}_{0.7}\text{Zn}_{0.3}$  phase with preferential (111) orientation. An analysis of XRD patterns revealed that after heat treatment, the Cu-Zn alloys were composed of a predominating  $\alpha$ -phase structure. After annealing, well defined pseudo-spherical Cu-Zn grains were formed covering the entire substrate surface. The EDS analysis indicated the formation of  $\text{Cu}_{0.7}\text{Zn}_{0.3}$  brass alloys. The results showed the feasibility of this low-cost new route for the preparation of good quality Cu–Zn alloys from cyanide-free electrolytes.

**Keywords:** electrodeposition; Cu–Zn alloy; brass; non-cyanide bath; heat treatment.

[T3-PP 58]

**ETUDE GRAVIMETRIQUE ET ELECTROCHIMIQUE DE  
L'INHIBITION DE LA CORROSION DE L'ACIER DOUX PAR UNE BASE  
DE SCHIFF DERIVEE DE QUINOLEINE « SB2 (I-Br) » DANS LE MILIEU  
HCl 1M.**

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**Résumé :**

La corrosion détruit un quart de la production annuelle mondiale d'acier, ce qui représente environ 150 millions de tonnes par an ou encore 5 tonnes par secondes.

La protection contre ce fléau, souci majeur pour les pays industrialisés ou les coûts de la corrosion représentent trois à quatre pourcent du produit brut constitue l'une des plus importantes recherches scientifiques et techniques actuelles.

Dans ce travail, nous nous intéressons à l'utilisation des inhibiteurs de corrosion qui est devenue indispensable pour limiter l'attaque des matériaux métalliques. Cependant, les méthodes utilisées pour inhiber la corrosion doivent être évaluées en fonction des paramètres particuliers du système, car les mesures préventives utilisées avec succès dans un environnement donné peuvent être néfastes dans d'autres conditions.

L'étude du pouvoir inhibiteur du composé base de Schiff « SB2 (I-Br) » sur l'acier doux en milieu HCl 1M a été déterminé par spectroscopie d'impédance électrochimique (SIE), les courbes de polarisation et par la méthode de perte de masse (la gravimétrie).

Les valeurs obtenues montrent que l'augmentation de la concentration en inhibiteur est suivie d'une augmentation de la résistance de transfert de charge, d'une diminution de la densité de courant et d'une diminution de perte de poids, et atteint une valeur maximale de 90% à  $2,5 \cdot 10^{-3}$  M. Les résultats obtenus mettent en évidence le caractère mixte de notre composés base de Schiff. Les trois méthodes utilisées pour cette étude sont en bonne corrélation.

**Mots clés :** Corrosion, acier doux, base de Schiff, efficacité inhibitrice.

[T3-PP 59]

**SYNTHESIS AND INHIBITING ACTIVITY OF A NEW BENZIDINE  
DERIVATIVE ON THE CORROSION OF X48 CARBON STEEL IN 1 M  
HCl**

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**Abstract:**

The use of organic compounds to inhibit the corrosion of mild steel and iron has become of great importance because of their application to prevent corrosion under different corrosive environments [1,2]. Mild steel is widely used as structural material in automobiles, pipes and chemical industries [3, 4]. Mild steel undergoes severe corrosion in pickling processes. Hydrochloric and sulfuric acids are widely used for stripping and descaling mild steel. Different organic compounds have been reported to be effective as corrosion inhibitors during acidification in industrial cleaning processes.

In this work, we synthesized a new Schiff base (L) containing heteroaromatic compound of nitrogen and sulfur as a substitute in its structure, was used for the first time for a study of the corrosion of mild steel X48 in the hydrochloric acid medium using weight loss measurements, potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS).

**Keywords:** Benzidine, Corrosion, X48, EIS, Polarization.

**References :**

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[T3-PP 60]

**SYNTHESIS, CHARACTERIZATION AND CORROSION  
PROTECTION PROPERTIES OF POLY (2-METHYLFURAN -CO-  
BITHIOPHENE) COATINGS ON STAINLESS STEEL**

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**Abstract:**

Poly(2-methylfuran-co- bithiophene) copolymer films were synthesized by electrochemical deposition on 304-stainless steel, from an acetonitrile (ACN) solution containing  $10^{-2}$  M bithiophene,  $10^{-2}$  M 2-methylfuran and  $10^{-1}$  M lithium perchlorate ( $\text{LiClO}_4$ ), by cyclic voltammetry (CV) between 0 V and 2 V vs. SCE, with a scan rate of  $50 \text{ mV} \cdot \text{s}^{-1}$ . The copolymers coated were studied in a corrosive sulfuric acid medium ( $\text{H}_2\text{SO}_4$  1 N) using the potentiodynamic polarization method and the electrochemical impedance spectroscopy (EIS). Copolymers coated characterization was performed using scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. The polarization curves show that the copolymer film formed on A304, shifts the corrosion potential towards more negative potentials. The presence of the poly(2-methylfuran-co- bithiophene) improves the corrosion resistance of the metal in a corrosive medium  $\text{H}_2\text{SO}_4$ . This protection against corrosion is caused by the barrier effect of the layer of copolymer, which covers the surface of the A304 stainless steel against the aggressive ions of the corrosive medium.

**Keywords:** 2-methylfuran; bithiophene; copolymers; protection against corrosion; stainless steel.

[T3-PP 61]

**CORROSION PROTECTION OF MILD STEEL BY A NEW  
PHOSPHONATE INHIBITOR IN HCl**

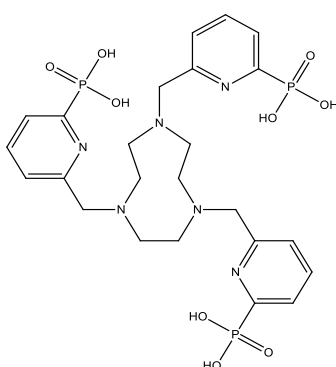
Lilia Tabti<sup>a</sup>, Embarek Bentouhami<sup>a</sup> and Loïc Charbonnière<sup>b</sup>

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**Abstract:**

The adsorption behavior and inhibition mechanism of (1, 4, 7-Tris [hydrogen (6-methylpyridin-2-yl) phosphonate] -1, 4, 7-triazacyclononane) (TPP) on the corrosion of carbon steel in 1 M HCl were investigated by weight loss technique, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) methods for different concentrations at 25°C. Results obtained show the inhibition efficiency values depend on the amount of immersion times and the concentration. The inhibition efficiency was at 90% at the highest concentration of the compounds according to weight loss measurements. The adsorption of corrosion inhibition was well supported by AFM study.



**Fig.1.** Chemical structure of the inhibitors TPP.

[T3-PP 62]

**INHIBITION DE LA CORROSION D'UN ACIER A316 PAR UN  
COPOLYMERE POLY(PYRROLE+METHYL-2 FURANNE) EN  
MILIEU CORROSIF**

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**Résumé :**

Les hétérocycliques sont des composés organiques considérés comme inhibiteurs efficaces pour lutter contre la corrosion des métaux exposés aux milieux agressifs. Ces derniers, lorsqu'ils sont ajoutés en faibles quantités; ils peuvent réduire ou même stopper la corrosion du métal dans le milieu considéré, donc leurs principe de fonctionnement se base généralement sur la protection du métal de son environnement.

L'objectif de ce travail est d'étudier la tenue à la corrosion d'un acier A316 modifié par un copolymère de poly(pyrrole+methyl-2 furanne) dans un milieu corrosif NaCl 3%. L'électrodéposition des films de copolymère a été effectuée par la voltampérométrie cyclique.

Les courbes voltampérométriques montrent au cours du cyclage des pics anodiques et cathodiques caractéristiques de l'oxydation et de la réduction du copolymère formé. Les diagrammes d'impédance montrent un arc de cercle aux hautes fréquences suivi par une droite aux basses fréquences qui sont, respectivement, caractéristiques d'un processus de transfert de charge et d'un autre purement diffusionnel. La tenue à la corrosion d'un acier A316 protégé par un film du copolymère a été aussi testée dans un milieu corrosif NaCl 3%. L'efficacité inhibitrice de revêtement a été aussi justifiée lors du tracé des courbes de Tafel, par un déplacement des potentiels de corrosion vers des valeurs négatives. Le MEB a permis la visualisation d'une couche adhérente et stable à la surface de l'acier. Les résultats obtenus montrent que ces copolymères organiques peuvent servir d'inhibiteurs de corrosion.

**Mots clés:** *pyrrole, methyl-2 furanne, copolymère, voltampérométrie cyclique, spectroscopie d'impédance, corrosion.*



[T3-PP 63]

**ELECTRODEPOSITION OF NICKEL-ZINC ALLOY COATING ON  
LOW CARBON STEEL FROM A SULPHATE BATH TITLE**

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**Abstract**

The present work investigates the electrodeposition of Zn, Ni and Zn–Ni alloy from a sulphate bath on stainless steel substrate using cyclic voltammetry and transient current methods. The effect of the experimental parameters (deposition potential, bath temperature, zinc concentration, presence of additives) on the coating composition, morphology and structure was studied. The kinetic study allowed optimization of electrodeposition conditions of Zn–Ni alloy layers [1]. Analysis of the transient currents using Scharifker Hills model [2] indicates that the nucleation mechanism of Zn–Ni alloy follows instantaneous nucleation, and the three-dimensional growth (Volmer-Weber) limited by diffusion [3].

**Keywords:** Electrodeposition, corrosion, cyclic voltammetry, nickel-zinc alloy,

**References**

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[T3-PP 64]

**ELECTROCHEMICAL INVESTIGATION OF NEW AZO-  
CONTAINING OXOVANADIUM(IV) COMPLEXES**

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**Abstract:**

Azo chemistry has been studied extensively and they have advanced applications in high technology areas such as laser, liquid crystalline displays and inkjet printers [1-3]. Oxovanadium complexes with azo- ligands were recognized as important elements with regard to their widespread application in optical, catalysis and medicinal properties [4-6].

Numerous electrochemical studies have been made for versatile oxovanadium(IV) complexes derived from azo-compound [7]. The investigation reveals that these complexes show, in strong coordinating solvents such as DMF and (CH<sub>3</sub>)<sub>2</sub>SO, a quasi-reversible diffusion-controlled one-electron transfer process [7].

Herein, we report the direct synthesis, spectral analysis and crystal structure of new monomeric diazomethine oxovanadium (IV) complexes, VOL<sup>X</sup>. The crystallographic studies of VOL<sup>X</sup> were solved by X-ray diffraction, which revealed that these complexes adopt a square pyramidal N2O3 geometry. In the packing, weak intermolecular H-bonding and  $\pi \dots \pi$  stacking are observed.

The electrochemical properties of VOL<sup>X</sup> complexes were followed in DMF solvent by cyclic and linear voltammeteries, on a glassy carbon GC electrode. A reversible response, in the potential range 100–900 mV vs SCE, is shown, involving a single electron V(V)/V(IV) redox couples. The diffusion coefficients are calculated, from Levich plot.

**Keywords:** Azo-complexes, Oxovanadium, Crystal structure, Cyclic voltammetry, Diffusion coefficient.

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[T3-PP 65]

**ELABORATION D'UN REVETEMENT A BASE DE POLYANILINE  
DOPEE PAR LES IONS MOLYBDATES. APPLICATION A LA  
PROTECTION CONTRE LA CORROSION DE L'ACIER EN MILIEU  
ACIDE.**

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**Résumé :**

Les revêtements organiques à base de polymères conducteurs sont largement utilisés dans la protection des métaux contre la corrosion. Cependant, la variété du milieu corrosif rend nécessaire de développer des revêtements de meilleures qualités. De ce fait, l'utilisation des dopants appropriés peut rendre ces revêtements plus résistants et plus performants pour la protection anti-corrosion.

C'est dans cette optique que s'inscrit notre étude qui est basée sur l'élaboration d'un revêtement à base de Polyaniline capable d'apporter une meilleure protection contre la corrosion de l'acier en milieu acide et ce grâce au dopage du film de polymère par les ions molybdate.

La synthèse des films de polymères a été réalisée par voie électrochimique en milieu acide oxalique. Plusieurs méthodes d'analyses expérimentales ont été utilisées afin de caractériser la morphologie et la composition des films obtenus. Les tests de corrosion menés montrent une nette amélioration du temps de protection du métal et l'existence d'un optimum de protection pour une composition du mélange de contre-ions. Ces derniers une fois libérés auto-réparent le film par formation « *in-situ* » d'une couche passive à l'endroit des lésions.

**Mots clés :** Protection contre la corrosion, Polyaniline, Dopage, Molybdate de sodium.

[T3-PP 66]

**SYNTHESE, CARACTERISATION PHYSICO-CHIMIQUE ET  
CRISTALLOGRAPHIQUE DE NOUVELLE BASE DE SCHIFF  
DERIVES DES PYRAZOLONES ET ETUDE DE SON ACTIVITE  
INHIBITRICE VIS A VIS DE LA CORROSION D'ACIER DE  
CONSTRUCTION**

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**Résumé:**

La corrosion des armatures est une des causes majeures de dégradation des structures et ouvrages en béton armé. La durée de vie de ces derniers est conditionnée par les altérations physiques et chimiques de l'environnement, ainsi que par la résistance mécanique et chimique des matériaux constitutifs. La corrosion se développe principalement suite à deux processus: la carbonatation du béton et l'attaque par les chlorures qui, lorsqu'ils se trouvent en quantité suffisante à la surface de l'armature, génèrent des piqûres de corrosion.

En matière de protection, les inhibiteurs constituent un moyen original et efficace pour lutter contre la corrosion dans tous les domaines et principalement dans le béton. Parmi les inhibiteurs utilisés, les composés organiques notamment les bases de Schiff, semblent être très promoteurs.

C'est dans ce cadre que s'inscrit l'objectif de ce travail qui consiste à étudier la protection de l'acier de construction F10, dans un milieu chloruré simulant l'interface (armature/béton) par une nouvelle base de Schiff dérivée de pyrazolone le 4-[(3-hydroxy phenyl amino) phenyl methylene]-5methyl-2phenyl-pyrazol-3-one. Cette base de Schiff est obtenue par la condensation de l'acide benzoylpyrazolone HPMBP préalablement synthétisé avec le 3-amino phénol, La caractérisation de ce produit a été faite par IR, UV-Vis, SM et RX. Ce composé est un monocristal. L'étude de l'inhibition de la corrosion est examinée par voltamétrie cyclique et par impedancemétrie. La théorie de la densité fonctionnelle (DFT) a été utilisée pour définir les sites d'action de cet inhibiteur et confirmer ou infirmer les résultats électrochimiques. Il ressort une bonne concordance.

**Mots clés :** Pyrazolone, Base de Schiff, Corrosion, Inhibiteur, Béton armé, DRX, DFT.

[T3-PP 67]

**ETUDE DU POUVOIR INHIBITEUR DE LA CORROSION DE L'ACIER  
EN MILIEU HCl 1M**

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**Résumé:**

La corrosion est un phénomène qui affecte le comportement mécanique et physique des matériaux. Elle résulte d'une action chimique ou électrochimique d'un environnement sur les métaux et les alliages. Ses conséquences engendrent des coûts importants dans différents domaines et en particulier dans l'industrie. Dans ce travail, nous étudions le pouvoir anticorrosif de nouveau composé base de Schiff: 4,4'-Soufre bis{N-[ ( E ) -phénol-3-ylmethylidene]aniline}(BS) sur l'acier XC48 en milieu HCl 1M à température ambiante. Le pouvoir inhibiteur de ce composé a été déterminé par la méthode de perte de masse « gravimétrie, spectroscopie d'impédance électrochimique (SIE), les courbes de polarisation ». L'effet de concentration montre que la base de Schiff (BS) agit comme un bon inhibiteur de corrosion de l'acier XC48 dans la solution HCl 1M. L'efficacité inhibitrice augmente avec la concentration en inhibiteur pour atteindre une valeur maximale 91,2% à  $2,5 \cdot 10^{-3}$  M pour gravimétrie. L'inhibition de la corrosion du métal par la base de Schiff (BS) étudiée est expliquée par son adsorption sur la surface métallique. Cette dernière suit l'isotherme de Langmuir.

**Mots-clés :** base de Schiff, corrosion, inhibiteur, gravimétrie, HCl, acier.



[T3-PP 68]

## CORROSION INHIBITION PERFORMANCE OF NEW SCHIFF BASE AND ITS COMPLEX ON CARBON STEEL IN 1 M HCl

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### **Abstract:**

Corrosion inhibitors are compounds which control, reduce or prevent reactions between metals and corrosive media. Many efficient organic inhibitors have  $\pi$ -bonds in their structures as well as heteroatoms such as nitrogen, oxygen and sulphur. Among those are Schiff bases and their metal complexes.

In acidic environment, many factors exhibit high inhibiting properties by providing electrons to interact with metal surface including the size of the molecule, the environment and nature of the metal, experimental parameters such as inhibitor concentration, molecular structure and nature of the substituents in the molecule itself should also be considered.

In this work, the corrosion inhibition performance of new Schiff base and its complex of Cu (II), on carbon steel substrate in 1 M HCl was studied using weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy methods. It was seen that this compounds decreased the corrosion rate of mild steel in acidic medium due to the adsorption on the metal surface, and it was found that the inhibition efficiency increased with increasing the inhibitor concentration. The adsorption of inhibitors on the carbon steel surface obeys the Langmuir isotherm equation.

[T3-PP 69]

**ETUDE DE L'EFFICACITE INHIBITRICE DE DEUX BASES DE SCHIFF VIS-A-VIS DE LA CORROSION DES ARMATURES DU BETON ARME EN MILIEU NaCl 0.5M**

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**Resumé:**

La corrosion des métaux est un phénomène néfaste qui conduit à des dégâts et déficits importants dans l'économie mondiale. A cet effet, il est noté qu'un problème très ennuyeux est rencontré dans le domaine du génie civil à savoir la corrosion des armatures du béton. La lutte contre ce phénomène néfaste se fait par différents moyens dont l'utilisation des inhibiteurs de corrosion. Les bases de Schiff sont une famille de composés hétérocycliques récemment très utilisés présentant des pouvoirs inhibiteurs remarquables vu leurs forte habilité d'adsorption sur les métaux.

Notre travail consiste en premier lieu à la synthèse et la caractérisation de deux inhibiteurs organiques bases de Schiff L<sub>1</sub> et L<sub>2</sub> dérivés du même produit de départ (3-amino-2-naphtol). En second lieu l'étude électrochimique par la polarisation potentiodynamique et spectroscopie d'impédance du pouvoir inhibiteur de ces produits vis-à-vis de la corrosion d'un acier doux de construction dans le béton immergé dans un milieu agressif 0.5M NaCl en fonction de différentes concentrations de L<sub>1</sub> et L<sub>2</sub> (10<sup>-4</sup>- 10<sup>-6</sup>M), du temps d'immersion (2- 28 jours) et de température (30- 60°C). En fin une simulation par les calculs quantiques en utilisant la méthode de densité électronique théorique (DFT) des structures électroniques de L<sub>1</sub> et L<sub>2</sub> est réalisée pour confirmer ou infirmer les résultats électrochimiques. Les résultats électrochimiques et thermodynamiques montrent que le L<sub>1</sub> et L<sub>2</sub> sont de bons inhibiteurs de corrosion de l'acier doux dans le béton exposé à un environnement de NaCl 0.5M. Ces bases de Schiff agissent par adsorption selon un procédé de chimisorption obéissant à l'isotherme de Langmuir confirmé par l'analyse MEB.

**Mots-clés :** Corrosion, Acier doux, Béton, Inhibiteur, Bases de Schiff, MEB, DFT.

[T3-PP 70]

## SYNTHESIS AND A COMPARATIVE STUDY OF THE CORROSION INHIBITIVE EFFICIENCY OF $\alpha$ -AMINOPHOSPHONATE AND SCHIFF BASE DERIVATIVES

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### Abstract:

Corrosion of steel is a major problem in our daily lives and in various industrial plants. In this context, many techniques and methods have been developed and used to protect steel against corrosion.  $\alpha$ -aminophosphonate and Schiff bases derivatives have been attracting an outstanding attention during the last decade as a promising class of viable compounds in the corrosion inhibition field.

New  $\alpha$ -aminophosphonate ( **$\alpha$ -APD**) and Schiff base (**E-NDPIMA**) derivatives have been prepared and their structures were proved by IR, UV-Vis, <sup>1</sup>H, <sup>13</sup>C and <sup>31</sup>P NMR spectroscopy. Their inhibitive capacities on the XC48 carbon steel corrosion in 0.5 mol L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> solution were explored by weight loss, Tafel polarization, electrochemical impedance spectroscopy (EIS) and atomic force microscope (AFM).

Experimental results illustrate that the synthesized compounds are an effective inhibitors and the adsorption of inhibitors molecules on the carbon steel surface obeys Langmuir adsorption isotherm. In addition, quantum chemical calculations performed with density function theory (DFT) method have been used to correlate the inhibition efficiency established experimentally. Also, the molecular dynamics simulations (MDS) have been utilized to simulate the interactions between the inhibitors molecules and Fe (100) surface in aqueous solution.

**Keywords:**  $\alpha$ -aminophosphonate, Schiff base, Corrosion inhibition, DFT, MDS.



[T3-PP 71]

**STUDY OF IODATE BEHAVIOR ON COPPER CORROSION IN NITRIC  
ACID SOLUTION**

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**Abstract:**

Le cuivre est l'un des métaux les plus utilisés dans différents domaines et pratiquement dans toutes les industries, grâce à ses propriétés; électrique, mécanique, chimiques et physique.

Dans le terrain industriel, il y a pas mal des pièces de cuivre en contact tout le temps avec des milieux agressifs comme les acides, qui provoquent la corrosion.

Le but de ce travail est d'appliquer les techniques électrochimiques stationnaires pour étudier l'effet de la concentration de l'acide nitrique sur les paramètres électrochimiques du cuivre, puis l'effet protecteur de l'iodate de potassium sur la surface de métal, et enfin l'effet de la température.

Les résultats montrent que la concentration de 1 M d' $\text{HNO}_3$  est la plus agressive pour le cuivre; et que le  $\text{KIO}_3$  est un bon inhibiteur de corrosion du cuivre, une efficacité optimum de 86.74% est atteinte. L'étude de l'effet de température a permis de déterminer l'isotherme d'adsorption de cette molécule, elle obéit à l'isotherme de Langmuir; et l'adsorption s'effectue par une interaction de type chimique entre la molécule et la surface métallique.

**Keywords:** Protection, Iodate de potassium, Cuivre, Corrosion, acide nitrique. DFT

## Topic 4: Organic and Bioelectrochemistry.

### [T4-PP 72]

#### SYNTHESIS, CHARACTERIZATION AND VOLTAMMETRIC DETERMINATION OF ANTIOXIDANT CHARACTER OF THREE $\alpha$ -AMINOPHOSPHONIC ACIDS.

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#### Abstract:

$\alpha$ -aminophosphonic acid derivatives constitute an important class of organophosphorus compounds on account of their versatile biological activity. They are considered to be structural analogues of amino acids [1].  $\alpha$ -aminophosphonic acids are found to compete effectively with their amino acid counterparts for binding to enzyme active centers or other cellular targets. This, together with their low mammalian toxicity makes the  $\alpha$ -aminophosphonic acids an important class of antimetabolites and a potential source of medicinal compounds [2].

In the present work, three  $\alpha$ -aminophosphonic acids were synthesized using various primary amines as the starting materials. The obtained compounds are stable solids characterized by high melting points and their structures were confirmed by UV-Visible, FT-IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and  $^{31}\text{P}$  NMR spectra.

These synthesized compounds were screened for their antioxidant activity by electrochemical method. Cyclic voltammetry method developed in this study measures the scavenging effect toward superoxide anion radical and indicate the strong interaction between the  $\text{O}_2^{\bullet-}$  and synthesized compounds, this is quantified in terms of the high obtained values of  $K_b$ . In the other hand, the negative sign of  $\Delta G^\circ$  suggests the spontaneity of the antiradical reaction. According to this method, we can also be proposed that the antiradical reaction mechanism is the hydrogen atom transfer.

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[T4-PP 73]

**NEW QUATERNIZED POLY(4-VINYLPYRIDINE-CO-DIVINYLBENZENE) MATERIAL CONTAINING NICKEL(II)-SCHIFF BASE COMPLEX : SYNTHESIS, THERMOGRAVIMETRY AND APPLICATION TO THE HETEROGENEOUS ELECTRO-OXIDATION REACTION OF ETHANOL**

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**Abstract**

Poly(4-vinylpyridine) crosslinked by 2% of divinylbenzene was partially quaternized (20% monomer equivalents) by a bidentate Schiff base copper complex containing C-Br function in its molecular structure using dioxane as solvent. The resulting poly(4-vinylpyridine-co divinylbenzene)-Ni(II) Schiff base complex (PVP-co-DVB-Ni(II)-L2) was characterized using various techniques such as elemental analysis and Fourier transform infrared spectroscopy (FT-IR). The thermogravimetric analysis (TG/DTG) of the non-modified copolymer, free complex and the copolymer modified with the nickel complex was carried out to elucidate the thermal behavior. The synthesized copolymer covalently grafted with the nickel(II) complex was mixed with carbon black paste to prepare new modified electrode. This electrode was characterized by wide-angle X-ray diffraction (WAXS), scanning electron microscopy/energy dispersive X-ray (SEM/EDX) and cyclic voltammetry. Electrochemical properties of the resulting modified electrode, recorded in alkaline solution, have been investigated by cyclic voltammetry, showing well-defined redox process corresponding to Ni<sup>III</sup>/Ni<sup>II</sup> redox couple. The effect of some parameters such as scan rate and concentration of ethanol were explored towards the electro-oxidation of ethanol for this modified electrode. The proposed mechanism of ethanol electro-oxidation based on the cyclic voltammetry study was suggested.

**Keywords:** Poly(4-vinylpyridine), Divinyl benzene, Ni(II) -Schiff base complex, Thermogravimetry, Cyclic voltammetry, Electro-oxidation.



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[T4-PP 74]

## NANOMATERIALS AS CATALYTIC ELEMENTS FOR ELECTROCHEMICAL DETECTION OF PHENOLS

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### Abstract:

Modified electrodes are new tools for the monitoring of phenolic compounds especially when it combined with nanomaterials. The unique properties of nanomaterials such as carbons, metal oxides, conducting polymers and nanoparticles facilitates the kinetic reactions and enhances the current response of sensors by increasing the charge electron transfer at the electrode surface, and consequently minimize the response time of sensor. All of these qualities nominated them to be a suitable choice in the fabrication of the electrochemical sensors. This study explores the potential and the role of nanomaterials as catalytic elements to enhance reactions mechanisms at the surface of modified electrodes. In this work, we study the principle of some catalytic reactions of the most performed chemically modified electrodes using nanomaterials as catalytic reagents.

**Keywords:** Nanomaterials, modified electrodes, electrochemical detection, phenolic compounds



[T4-PP 75]

## SUPEROXIDE ANION SCAVENGING EFFECT OF DICLOFENAC USING ELECTROCHEMICAL METHODS

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### Abstract:

Diclofenac is one of the most anti-inflammatory drug, was selected for their antioxidant effect against free radicals in this study, Molecular structure proprieties were evaluated using density functional theory (DFT) via B3LYP/6-31G (d,p) method. Superoxide anion scavenging was evaluated by electrochemical method using cyclic voltammetry (CV) study which was carried out employing aVoltalab 40 model PGZ301potentiostat/galvanostat controlled by a personal computer through the voltamaster4 software.

Our results show high antioxidant effect of the Diclofenac with inhibition percentage of 73.15 % and with IC<sub>50</sub> of 77.32 μM.

**Key word:** Electrochemical, voltammetry, antioxidant, Diclofenac.

[T4-PP 76]

**A NOVEL OXOVANADIUM (IV) COMPLEX WITH AN  
UNSYMMETRICAL TETRADENTATE – SCHIFF BASE: SYNTHESIS,  
ELECTROCHEMICAL AND CATALYTIC OXIDATION OF  
CYCLOHEXENE.**

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**Abstract:**

This work describes the synthesis of a new unsymmetrical tetradentate oxovanadium(IV) Schiff base complex VO(IV)L<sub>Cl</sub> containing N<sub>2</sub>O<sub>2</sub> donor atoms. The tetradentate ligand (H<sub>2</sub>L<sub>Cl</sub>) has been prepared by condensation of 5-bromosalicylaldehyd on 4-Chloro 1,2-diaminobenzene in methanol. The reaction of the ligand with an appropriate amount of oxovanadium(IV) (1 : 1 ratio) in the same solvent. The tetradentate ligand (H<sub>2</sub>L<sub>Cl</sub>) as mixed ligands where two nitrogen and two enolic oxygen atoms were chelated to the oxovanadium centre. This complex has been fully characterized by FT-IR, UV-Vis spectrophotometry, <sup>1</sup>H and <sup>13</sup>C NMR, Dept 135 and microanalysis and cyclic voltammetry.

Electrochemical properties of the complex were investigated, in DMF, by cyclic voltammetry (CV) using glassy carbon as working electrode under nitrogen. The diffusion coefficients of the complexes were investigated on GC rotating disk electrode (RDE) voltammetry using the Levich plot,  $I_{lim} = f(\omega^{1/2})$ . The complexes are exploited in the catalytic oxidation of cyclohexene in the presence of oxygen.

**Key words:** Schiff bases, complexes, Spectroscopy, Cyclic voltammetry, Catalytic oxydation, Cyclohexene.

[T4-PP 77]

## ETUDE ELECTROCHIMIQUE DE L'ACTIVITE ANTIOXYDANTE D'UNE SERIE DE BASES DE SCHIFF PAR VOLTAMETRIE CYCLIQUE

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### Résumé :

Les antioxydants sont des molécules capables d'interagir sans danger avec les radicaux libres et de mettre fin à la réaction en chaîne avant que les molécules vitales ne soient endommagées. Chaque molécule antioxydant ne peut réagir qu'avec un seul radical libre et par conséquent, il faut constamment refaire le plein de ressources antioxydants.

Dans ce travail, nous avons étudié l'activité antioxydante de deux molécules bases de Schiff 3,3', 4,4'-tétra [dihydroxy benzaldehyde imino] diphenyle (TDBID) et 3'3, 4'4-tétra [salicylaldehyde imino] diphenyle (TSAID) par voltamétrie cyclique.

L'évaluation du pouvoir antioxydant a été effectuée par méthode DPPH.

Les résultats de la voltamétrie cyclique permettent de constater que la vitamine C se caractérise par un pic d'oxydation irréversible qui se situe à 1 210 mV. Le même comportement électrochimique été observé avec le ligand L1 et L2 mais à des potentiels moins positives que le standards ; qui sont 110 et 80 mV respectivement. Ces résultats montrent que l'acide ascorbique s'oxyde à des potentiels plus élevé que les composés étudiés responsables de la réponse électrochimique.

Les résultats du test DPPH montrent que la IC<sub>50</sub> du composé TDBID est de l'ordre de 33,66 µM tandis que celle du composé TSAID est de l'ordre de 245,24µM, ceci indique que le composé plus riche en OH est plus efficace.

L'efficacité du composé TDBID est comparable avec celle de la vitamine C (référence).

**Mots clés :** activité antioxydante, base de Schiff, voltamétrie cyclique, DPPH

[T4-PP 78]

USE OF ELECTROCHEMICAL AND SPECTROSCOPICAL METHOD  
FOR THE QUANTIFICATION OF ANTIOXIDANT ACTIVITY FROM  
*Astragalus numidicus* METHANOLIC EXTRACT

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**Abstract:**

This work describes a use of electrochemical and spectroscopical method for the evaluation of the antioxidant activity from *Astragalus numidicus* methanolic extract.

The antioxidants properties of the root extract of *Astragalus numidicus* obtained by ultrasonically method was investigated using superoxide anion radical and diphenyl-1-picrylhydrazyl (DPPH) scavenging assay.

The cyclic voltammetry CV technique was used to generate the superoxide radical  $O_2^{\cdot-}$  by the one electron reduction of oxygen molecular and the UV- vis spectroscopy at used to the controlled of DPPH radical inhibition.

The result of antioxidant activity from methanolic extract obtained by (DPPH) test has 76% percentage inhibition with a concentration about 1 mg / ml. The value of IC<sub>50</sub> in mg/ml expresses the effective concentration of the antioxidant extract necessary for trapping 50% of DPPH radicals dissolved in methanol, according to the results obtained; the methanolic extract has a good antioxidant activity value of 0.9 mg/ml.

The standard antioxidant used in the  $O_2^{\cdot-}$  radical scavenging is Ascorbic acid. It showed powerful anti-radical activity of the order 93% and the *Astragalus numidicus* methanolic extract showed average activity in this test.

**Keywords:** antioxidant activity, cyclic voltammetry, *Astragalus numidicus*





[T4-PP 79]

**BIOCAPTEUR ELECTROCHIMIQUE POUR LE DIAGNOSTIC  
PRECOCE DE L'INFLAMMATION**

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**Résumé**

Ce travail concerne le développement d'un nouveau biocapteur électrochimique pour la détection rapide de l'inflammation aux stades précoces via la détection directe de l'interleukine-10 (IL-10), une cytokine anti-inflammatoire, à partir d'échantillons biologiques. Ce biocapteur est basé sur un substrat semi-conducteur au nitrure de silicium ( $\text{Si}_3\text{N}_4$ ) modifié successivement par le pyrrole silane (SPy) puis par une couche conductrice de polypyrrole (PPy) formant ainsi le composite  $\text{Si}_3\text{N}_4$ -(SPy-PPy). Ensuite, la couche de PPy a été modifiée électrochimiquement avec le sel de diazonium (acide 4-aminophénylacétique, CMA) afin d'être utilisée pour l'immobilisation directe de l'anticorps monoclonal humain interleukine-10 (mAcIL-10) responsable de la reconnaissance et de la détection d'antigène recombinant IL-10 (rh IL-10). La morphologie de surface du composite  $\text{Si}_3\text{N}_4$ -(SPy-PPy) a été étudiée par microscopie électronique à balayage (MEB) et spectroscopie infrarouge (FTIR). Le comportement électrochimique du film PPy avant et après la fonctionnalisation avec CMA a été caractérisé par la voltammétrie cyclique (VC) et la spectroscopie d'impédance électrochimique (SIE). La formation de la biocouche d'immunocapteur et la reconnaissance d'un antigène spécifique d'IL-10 par l'anticorps, immobilisé sur le substrat  $\text{Si}_3\text{N}_4$ -(SPy-PPy)-CMA a été surveillée par SIE. Les résultats ont montré que l'IL-10 a été détectée avec succès par le nouvel immunocapteur développé sur une plage de concentrations de 1-50 pg/ml. De plus, l'immunocapteur à base de PPy présentait une sensibilité et une sélectivité élevées de rh IL-10 par rapport à d'autres cytokines interférentes (p. ex. IL-8 et TNF- $\alpha$ ). Ce nouveau composite montre une sensibilité élevée et constitue une nouvelle approche pour le développement de nouvelles stratégies d'obtention de biocapteurs et de détection précoce et rapide des maladies.

**Mots clés :** Polypyrrole, Nitrure du silicium, Interleukine-10, Spectroscopie d'impédance électrochimique.



[T4-PP 80]

**ELECTROCHEMICAL BEHAVIOR OF SUPEROXIDE ANION  
RADICAL TOWARDS N'-METHYLFERROCENYL-N'-  
PHENYLACETOHYDRAZIDE: ANTIOXIDANT ACTIVITY  
EVALUATION**

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**Abstract:**

In the present study, we show the electrochemical generation of superoxide anion radical followed by its interaction with N'-methylferrocenyl-N'-phenylacetohydrazide. The fate of electrochemically generated  $O_2^{\cdot-}$  was probed in the presence of N'-methylferrocenyl-N'-phenylacetohydrazide while monitoring the changes in the shape of its voltammogram. The nature of the interaction between  $O_2^{\cdot-}$  and N'-methylferrocenyl-N'-phenylacetohydrazide was quantified in terms of binding constant and binding free energy values and compared with standards of ascorbic acid. The antioxidant activity of N'-methylferrocenyl-N'-phenylacetohydrazide was determined by measuring the ability of the test sample to quench radicals (% Inhibition of) and expressed as  $IC_{50}$ .

**Keywords:** Cyclic Voltammetry, ferrocene derivatives, antioxidant activity, superoxide anion radical, binding parameters.

[T4-PP 81]

**SYNTHESIS AND CORROSION INHIBITION POTENTIAL  
OF 2-AMINO-N-(2-AMINOPHENYL)-3-(1H-IMIDAZOL-5YL)  
PROPANAMIDE**

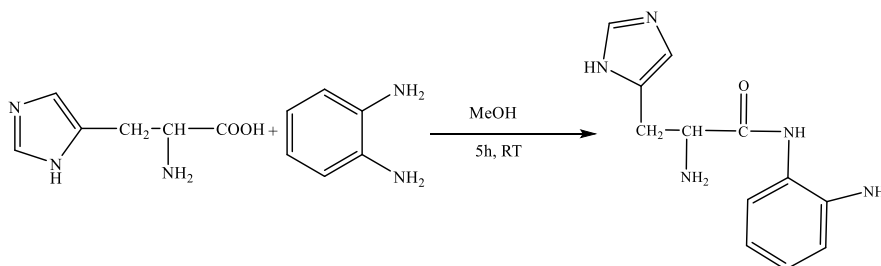
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**Abstract:**

2-amino-N-(2-aminophenyl)-3-(1H-imidazol-5yl) propanamid was synthesized from condensation reaction between o-Phenylenediamine and histidine :



The inhibitory effect of the synthesized product on the corrosion of carbon steel in saline medium NaCl 3% was studied using gravimetric and electrochemical methods.

Gravimetric measurements indicate that the synthesized product has interesting inhibiting properties, despite the hostility of the environment (NaCl%) against steel. In this direction, an inhibition efficiency exceeding 90% is obtained after immersion for one day and concentration of 2 ppm.

These results are in good agreement with those obtained by potentiodynamic technique.

**Keywords** — Corrosion, Carbon Steel, Histidine, electrochemical.



[T4-PP 82]

## ELECTROCHEMICAL SYNTHESIS OF ALUMINOPHOSPHATE AND THEIR ADSORPTION PROPERTIES

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### Abstract:

The aluminophosphate present as the first example of inorganic molecular sieves composed of a material other than silica.  $AlPO_4$  exist both in amorphous and crystalline forms, are micro porous solids with welldefined structures. This material possess ion-exchange properties, acidity and catalytic activity. The physicochemical properties of these materials are very interesting and attractive because the composed of both tetrahedral  $AlO_4$  and  $PO_4$  units.

In our previous study we have synthesized  $AlPO_4$  with electrochimecal method at differents voltages. The properties of this material was caracterizad by using spectrsopic techniques as XRD, FT-IR, to be used in adsorption of drugs

**keywords :**  $AlPO_4$ , electrochimecal synthesis, DRX, , FT-IR, Adsorption, drugs.

[T4-PP 83]

**ELECTROCHEMICAL CHARACTERIZATIONS OF BIOMEDICAL ALLOYS SS AISI 316L FOR SURGICAL IMPLANTS IN SIMULATED BODY FLUIDS.**

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**Abstract:**

Biomedical prosthetic devices are used in human body to carry out the functions that are no longer performed by the original human parts. Metals are used in the human body mainly for orthopedic purposes and their degradation by wear and corrosion must be negligible so that they can be used for various practical applications [1]. Among the various metallic materials that are used for orthopedic devices, 316L stainless steel (SS) is one of the most commonly used. It is frequently employed for temporary devices in orthopedic surgery because of its low cost and acceptable biocompatibility [2]. However, under some conditions this alloy suffers localized corrosion and releases significant quantities of iron to its neighboring tissue thus inducing fibrosis around the implant [2]. To overcome this problem the prostheses should have high corrosion resistance and at the same time the best adhesion to the tissue so that a stable biological bond with the bone is formed.

The aim of this research is to study the Tribological and Electrochemical behavior of AISI 316L stainless steel for Surgical Implants (Total hip prosthesis). The electrochemical study is focused on the influence of solution chemistry on the passive behavior using electrochemical techniques, Potentiodynamic curves, potentiostatic tests, and the open circuit potentiodynamiques (OCP); the aim was to evaluate one of the most important properties of an implant material namely its corrosion rate. In order to simulate natural biological conditions; (Ringer's solution and Physiological solution 9gL<sup>-1</sup> of NaCl) at Ph 7.4 thermostatically controlled at 37°C was used as the electrolyte.

**Keywords:** AISI 316L SS, Orthopedic prostheses, Biomaterials, Ringer's solution, 9g l<sup>-1</sup> NaCl

**Reference**

- [1] A. Duran, A. Conde, A. Gomezcoeds, T. Dorado, C. Garcia, J. Mater. Chem. 14 (2004) 2282.  
[2] M. Vallet Regi, I. Izquierdo Barba, F.J. Gil, J. Biomed. Mater. Res. 67A (2003) 674.

[T4-PP 84]

**ANTIOXIDANT PROPERTIES OF OXALIS SERNUA AERIAL PART  
EXTRACTS**

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**Abstract:**

Many polyphenols are derived from natural food products, they are often considered safer and more easily integrated into life style changes than conventional pharmaceutical drugs, and share beneficial effects against a wide range of diseases. This study was designed to examine the antioxidant activity of *Oxalis sernua* aerial part extracts using different tests. The extraction of phenolic compounds was conducted using solvent of increasing polarity resulting in three extractions; chloroform extract (Ch.E), ethyle acetate extract (EtA.E) and aqueous extract (Aq.E). The results obtained from antioxidant activities using ABTS radical scavenging and ferrous ion chelating assays showed that Ch.E has the highest inhibition with  $0.03 \pm 0.00$  mg/ml and  $0.19 \pm 0.02$  mg/ml respectively. The EtA. E revealed the highest inhibition in B-carotene assay with 85.45%. In conclusion, extracts have strong antioxidant activities and may have a potential health benefit.

**Keywords:** *Oxalis sernua*, Antioxidant activity, Polyphenols, ABTS, Ferrous ion chelating.

[T4-PP 85]

**PHOTONIC CRYSTAL BASED BIO-SENSOR FOR DNA ANALYSIS OF  
AUTOMATED CANCER DETECTION**

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**Abstract:**

The field of electrochemistry as applied in the detection and cure cancer cells is not new. While used in therapy, the tumor area is treated with direct current. Cancer is a generic term used to classify cause of a huge set of diseases. These diseases primarily have a common symptom of uncontrolled cell proliferation which cannot be controlled by the normal cell kinetics regulation mechanism. In case of cancer, a normal cell multiplies continuously without control, leading to the development of tumors or an abnormal rise in the number of dispersed cells like the blood corpuscles. Occurrence of cancer may happen in any organ or tissue of the body.

Damage to DNA is the root cause of cancer. In case of a normal cell, a damaged DNA gets either repaired or the cell ceases to exist. In case of cancer, the damaged DNA continues to make new cells even though the body may not require them. This results in unwanted tissues and manifests as either tumors or cancer.

Due to the fact that molecules and nanoparticles are much smaller than the wavelength, their absorption are quite small at Terahertz frequencies, making it difficult to detect them unless they are present in large quantities. However, it has recently been demonstrated that their absorption can be enhanced by placing the macros inside nano-slot resonators. This yields a dramatically increased absorption coefficient, allowing smaller quantities to be detected.

In this paper, we propose an new electrochemical detection system of cancer cells based on 2D photonic crystals. The proposed platform uses ring shaped holes Ring Resonator configuration. It is aimed at DNA analysis of normal and cancerous blood cells. As the refractive index of normal and cancer infected tissue is inferred, the sensor can easily differentiate normal tissue and cancer infected cervical tissue. The transmission characteristics of light in the biosensor under different refractive indices that correspond to the cervical types of cancer cell sample refractive index cause resonant wavelength shifting at the output terminal. This highly sensitive automated system may be used for early cancer detection due to a change in DNA properties without the need to human intervention, and thus reducing errors and increasing the precision.

**Keywords:** Silicon nitride, Nanocavity, optical biosensor , Surface characterization,

[T4-PP 86]

**STRUCTURE, THEORETICAL AND ELECTROCHEMISTRY  
INVESTIGATION OF A TETRADENTATE COPPER SCHIFF BASE  
COMPLEX**

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**Abstract:**

Metal complexes with Schiff bases as ligands have played a considerable attention in the development of coordination chemistry because they have high stability and important properties in different oxidation states. Particularly, there has been significant interest in the coordination chemistry of copper with multidentate Schiff base ligands, because such complexes have small size, higher nuclear charge and thus have a great affinity for coordination. Some square planar Cu (II) complexes with tetradentate N<sub>2</sub>O<sub>2</sub> donor Schiff base ligands have been extensively studied in the past years.

Therefore, Schiff base ligands derived from dehydroacetic acid (dha= 3-acetyl-4-hydroxy-6-methyl-2H-pyran-2-one) are widely used to coordinate various metal ions because of the high stability of their complexes. Furthermore, studies have shown that such compounds and their complexes have very interesting biological such as antimicrobial, antifungal, antitumor and herbicides, pharmacological and medicinal properties.

Our group is interested to the study of [Cu<sub>2</sub>L<sub>2</sub>H<sub>2</sub>O], H<sub>2</sub>O. We describe the synthesis of Schiff base copper complex derived from dehydroacetic acid. As an extension of our work, a spectroscopic studies, crystal structure, electrochemical investigation, AFM study and quantum chemical calculations of this new binuclear complex were cautiously examined.

**Keywords:** Metal complexes, electrochemistry, AFM, Quantum chemical calculations (DFT).





[T4-PP 87]

**SYNTHESIS AND CHARACTERIZATION OF NEW  
TETRATHIAFULVALENE (TTF) CONTAIN A PYRIDINE GROUP**

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**Abstract :**

A number of new asymmetrically substituted tetrathiafulvalene were successfully synthesized as new electron donor compound. The chemical structures of these compounds were investigated using several spectroscopic methods. The synthesis of these donors was achieved by using  $\pi$ -electron-rich pyridine ligands in combination with an alkylthiodithiolone (thione). Their electrochemical properties have been investigated by cyclic voltammetry at ambient temperature.

[T4-PP 88]

**SYNTHESIS, CHARACTERIZATION AND EVALUATION OF  
ANTIOXIDANT ACTIVITY BY ELECTROCHEMICAL ASSAY OF  
SOME SCHIFF BASES**

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**Abstract :**

Tetradentates Schiff base ligands  $\text{H}_2\text{L}^1$  and  $\text{H}_2\text{L}^2$  were synthesized respectively from 1,2-diaminobenzene and 1,2-ethylenediamine which were condensed on salicylaldehyde in ethanolic solutions. These synthesized compounds were characterized by the physicochemical methods such as FT-IR,  $^1\text{H}$  NMR, elemental analyses, molar conductance and UV-Vis spectrophotometry. Antioxydant activity of the ligands was evaluated by superoxide antioxidant assay using cyclic voltammetry (CV). The superoxide radical scavenging assay was carried according to the method described by E.I. Korotkova et al (2002) modified by C. Le Bourvellec (2008). Cyclic voltametry (CV) measurements were performed using a Potentiostat-Galvanostat ; Voltalab PGZ 301 model at room temperature. The CV experiments of the oxygen reduction were carried out in dimethyl sulfoxide (DMSO) practically saturated with oxygen with  $0.1 \text{ mol.L}^{-1}$  tetra-n-butylammonium perchlorate (TBAP) as supporting electrode, scanning in negative range -1000 to -400 mV at a scan rate of  $50 \text{ mVs}^{-1}$ . The antioxidant activity was assessed from the change in the anodic current of the voltammograms and the results were expressed in percentage as a  $\text{O}_2^{\bullet-}$  inhibition (I%).

In conclusion, of all the compounds tested,  $\text{H}_2\text{L}^1$  had the lowest  $I_{\text{pa}}$  ( $1.83 \cdot 10^{-6}$ ) and a powerful anti-radical activity (88.84 %) at  $10^{-3} \text{ M}$  in comparison with standard antioxidant ascorbic acid (84.32 %) at same concentration.

**Key words:** Schiff base ligands, Characterization, Cyclic voltametry, superoxide radical.

**Reference**

E. I. Korotkova, Y. A. Karbainov, A. V. Shevchuk, Study of antioxidant properties by voltammetry. *Journal of Electroanalytical Chemistry*, **518** (2002) 56–60.

C. Le Bourvellec, D. Hauchard, A. Darchen, J.L. Burgot, M.L. Abasq, *Talanta*, **75** (2008) 1098–1103.



[T4-PP 89]

## STUDY ON THE STRUCTURE AND PROPERTY OF LEAD TIN ALLOY AS THE POSITIVE GRID

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### Abstract:

Pb–Sn binary alloys with different contents of tin (0–2wt.%) were investigated as the positive grid of a lead acid battery. The microstructure of Pb–Sn alloys was observed using a polarizing microscope. The morphology of the corrosion layers and corroded surfaces of Pb and Pb–Sn alloy electrodes were analyzed by scanning electron microscopy (SEM) following the corrosion test. The electrochemical properties of Pb–Sn alloys in sulfuric acid solution were investigated by cyclic voltammetry (CV), open circuit potential (OCP), electrochemical impedance spectroscopy (EIS), and linear sweeping voltammetry (LSV). The results indicate that the introduction of tin results in grain refinement, increased corrosion resistance.

**Keywords:** Microstructure, corrosion, resistance, Pb–Sn alloys, battery

[T4-PP 90]

**SYNTHESIS, CHARACTERIZATION, ELECTROCHEMICAL  
PROPERTIES AND BIOLOGICAL ACTIVITY OF 1-[(4-  
HYDROXYANILINO) METHYLIDENE]-NAPHTHALEN-2(1H)-ONE  
AND ITS BINUCLEAR COMPLEX OF COBALT**

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**Abstract:**

The non-symmetrical bidentates Schiff bases derived from aromatic amines and 2,4-pentanedione and their metal complexes have a wide variety of applications in many fields as biological, medicinal, analytical chemistry, catalysis, electrocatalysis and sensors where they are currently associated with the use of modified electrodes. The non-symmetrical multidentates Schiff bases and their metal complexes have been used to inhibit the mild steel corrosion in several industrial media. The non-symmetrical character will probably induce more properties such as those of biological activities. In azomethine products, the bond C=N is essential for biological activity since it is reported that several of this compounds have remarkable antibacterial, antifungal, anticancer and diuretic activities. The nature of the metal ion in Schiff bases complexes and the sequence of donor sites of the corresponding ligands affect highly the pharmacological activity of these species.

In this study, we report the synthesis, structural and spectral characteristics of Co (II) complex  $Co_2(L^2)Py_3Cl_4$  where  $L^2$  was obtained by the condensation of 2-hydroxy-1-naphthaldéhyde and 4-aminophenol. This Schiff base and its Co (II) complex were characterized by  $^1H$ NMR,  $^{13}C$ NMR, EA, IR, UV-Vis, XRD, and CV. The Conductance measurements indicate that these compounds have not electrolytic character. The electrochemical behaviors of the ligand ( $HL^2$ ) and its Co(II) complex have been investigated on glassy carbon electrode in dimethylformamide in the presence of 0.1 M  $Bu_4NClO_4$ . The redox system of Co (II) complex seems to be consistent with a quasi-reversible system. The bidentate Schiff base ( $HL^2$ ) and its Co(II) complex have a high antibacterial activity towards the Escherichia coli Gram (-), Staphylococcus aureus : Gram (+) and Candida albicans(fungus).

[T4-PP 91]

**SYNTHESIS, SPECTROSCOPIC (FT-IR, UV-VIS, RMN)  
CHARACTERIZATIONS AND ELECTROCHEMICAL STUDY OF A  
NEW  $\alpha$ -AMINOPHOSPHONATE DERIVATIVE**

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**Abstract:**

Organophosphorus compounds have a wide application in the industry, medicinal chemistry. In recent years, the synthesis of phosphonates compounds has attracted significant attention, due to their multiple uses in many vital areas.  $\alpha$ -aminophosphonates are the analogs of natural amino acid and some of them exhibit broad-spectrum biological activities, such as anti-HIV, antibiotic, antibacterial, and antiviral agents, anticancer, inhibition of activities of enzymes and anti-virus activities. Furthermore,  $\alpha$ -aminophosphonates are used in agricultural industry as fungicidal, herbicidal agents and plant growth regulators. A component synthesis of  $\alpha$ -aminophosphonates by reaction of aromatic-substituted aldehyde, aromatic amine and triethylphosphite at 76 °C catalyzed by organocatalyst with excellent yield and short reaction time. The structure of the synthesized derivative was characterized and confirmed by the FT-IR and <sup>1</sup>HNMR. On the other hand, the electrochemical characterization of the investigated derivative has been effectuated using cyclic voltamperometry method.

**Keywords:**  $\alpha$ -aminophosphonates, Synthesis, Kabachnik–Fields, electrochemical characterization.

## Topic 5: Physical and Analytical Electrochemistry.

[T5-PP 92]

### ELECTROCATALYTIC REDUCTION ABILITY TOWARDS BROMOCYCLOPENTANE OF ELECTRODEPOSITED POLYPYRROLE FILMS DERIVED FROM 3-BROMOPROPYL-N- PYRROLE

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#### Abstract:

Three monomers 6-[3'-N-pyrrolpropoxy]-2-hydroxyacetophenone (**1**), 5-(3'-N-pyrrolpropoxy)-2-hydroxyacetophenone (**2**) and 4-(3'-N-pyrrolpropoxy)-2-hydroxyacetophenone (**3**) were synthesized and their poly(pyrrole) films were successfully electrodeposited on glassy carbon (GC) and Indium tin oxide (ITO) conductive electrodes by anodic oxidation in acetonitrile solutions containing  $n\text{-Bu}_4\text{N}^+\text{ClO}_4^-$  (TBAP 0.1 M). These films, currently called modified electrodes (noted ME), were obtained by the successive cycling at the appropriate potentials. These films contain chelating sites such as carbonyl group bearing the phenolic function which could play an important role in coordination chemistry. The electrodeposited poly(pyrrole) films on the ITO conductive glass electrodes offer some analytical advantages as the optical and electronic properties. Consequently, these new materials of electrodes were characterized by cyclic voltammetry while the morphology of these films was studied by FT-IR spectroscopy, scanning electron microscopy (SEM), dispersive energy X-ray spectroscopy and atomic force microscopy (AFM). The AFM studies show that the morphology of polypyrrole (PPy) films, electrodeposited on ITO surface, depends on the specific structure of the compound deriving from the monoalkylated dihydroxyacetophenone **1**, **2** and **3**. The coordination of copper was performed by electroreduction reaction in presence of ligand (**3**) and copper acetate salt. The resulting electrode material was tested towards the electrocatalytic activity in the reduction of bromocyclopentane.

**Keywords:** Poly(pyrrole) films, SEM and AFM, Copper complex, Electrocatalytic reduction.

[T5-PP 93]

**EFFECT OF PALLADIUM PARTICLES SIZE AND DISPERSION ON  
THE RESPONSE TO ASCORBIC ACID ON POLYPYRROLE THIN  
FILMS**

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**Abstract:**

Catalysts based on electronic conductive polymers (ECP) with noble metal inclusion are of great interest for numerous modern technological applications as electrocatalysis and sensors. In numerous studies it has been reported that catalytic performances of these materials can be correlated by the synergic effect between the metal and polymer matrix.

In this respect, the present work exploits the electrochemical deposition method for the insertion of palladium (Pd) microparticles in polypyrrole (PPy) films grown on silicon (Si) substrate under galvanostatic control. The deposition of palladium was carried out from a solution of PdCl<sub>2</sub> under potentiostatic conditions employing 60, 120 and 180 s deposition times. The size and dispersion of the metallic particles on the polymer surface were analyzed in detail by SEM and AFM techniques and correlated with the catalytic properties of the composite film towards the oxidation of ascorbic acid in phosphate buffer solution (PBS, 0.1 M pH 7.0). Results reveal that Pd particles possess a nearly rectangular shape and are uniformly dispersed on the PPy surface. Although important oxidation currents are recorded for the different samples, the best electrocatalytic behavior is attained with film made up of smallest Pd particles (less than 0.5 μm in size). From this study, it can be concluded that small Pd particles may enhance significantly the electroactivity of PPy film for the oxidation of ascorbic acid.

**Keywords:** Ascorbic acid, Electrodeposition, Palladium, Polypyrrole

[T5-PP 94]

**EFFET D'INCORPORATION DU TETRATHIAFULVALENE TTF SUR  
UNE ELECTRODE MODIFIEE PAR UN FILM DE POLYMERE  
ORGANIQUE CONDUCTEUR LE POLYTERTHIOPHENE**

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**Résumé :**

Dans cette recherche, nous avons étudié le comportement électrochimique et spectroscopique, du film de polyterthiophène modifié par incorporation d'un composé organique donneur d'électron le tétrathiafulvalène (TTF). L'objectif de cette étude est axé surtout sur la synthèse de nouveau polymère conducteur, l'étude entre la relation, structure/propriétés, et l'amélioration de leur stabilité.

La voltampérométrie cyclique montre un petit décalage de potentiel de réduction de polymère vers des potentiels négatifs après la modification (incorporation du TTF), cette constatation nous a permis de confirmer l'état d'existence de TTF dans le polyterthiophène peut être sous un autre forme (incorporé ou lié au polymère).

La caractérisation des films polyterthiophènes obtenus après l'incorporation de TTF par la spectroscopie d'impédance obtenue pour les différentes valeurs de potentiel, montre une diminution de la résistance en présence de TTF par rapport à celle de polyterthiophène non dopé et par conséquent une augmentation de la conductivité de système électrode/polyterthiophène modifié par incorporation du TTF.

La spectroscopie uv-visible des films du TTF/ polyterthiophène déposé sur une lame d'ITO montre également une diminution de l'absorbance de film de polyterthiophène dopé par TTF comparable à celle de spectre de polyterthiophène non dopé. Cette diminution de l'absorbance montre une variation des propriétés de polyterthiophène par incorporation avec le TTF.





[T5-PP 95]

**ELECTROSYNTHESIS AND CHARACTERIZATION OF  
POLYPYRROLE-SILVER NANOCOMPOSITE**

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**Abstract:**

Metal-polymer composites have attracted much attention recently due to their interesting properties and have been widely used in practical applications [1,2]. In addition to the advantageous properties of metals and polymers, these composites exhibit many new characteristics that the single-phase materials do not have.

In the present study, polypyrrole-silver nanocomposite films were fabricated in situ by a rapid electrochemical polymerization by cyclic voltammetry in pyrrole aqueous solution containing well-dissolved silver nitrate in a conventional three electrode cell using SCE, platinum foil, and platinum disc as reference, auxiliary and work electrode respectively.

The synthesized composite films was characterized by cyclic voltammetry, electrochemical impedance spectroscopy, scanning electron microscopy, X-ray diffraction, and UV-vis absorbance spectroscopy, and conductivity of the nanocomposite was carried out by standard four-probe method.

The results showed that the Ppy-Ag composite films were synthesized successfully and showed better electrochemical characteristics than the pure Ppy electrode. Compared with the pure ppy film, the composite film was a highly porous film. Silver nanoparticles have an obvious improvement effect which makes the composite films more active sites for faradic reaction, lower resistance than pure ppy and facilitates the charge transfer in the composite films.

**Keywords:** polypyrrole, silver, electrochemistry, nanocomposite

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[T5-PP 96]

**EFFECTS OF VARIOUS OPERATING PARAMETERS ON  
THE ELECTROSYNTHESIS OF FERRATE (VI)**

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**Abstract:**

In the past decade, there has been huge interest in Ferrate (VI) due to its increasing applications. Presently, it can be used in green organic synthesis processes as a safer alternative to other highly toxic oxidizing compounds, and for water treatment to replace chlorine, hydrogen peroxide or ozone. Fe (VI) is an efficient oxidizing agent for the transformation of antibiotics, endocrine disruptors, and toxins to relatively non-toxic products. An additional advantage of Fe (VI) is an efficient coagulant for removing metals from solutions. Moreover, Fe (VI) is an effective disinfectant. Ferrate (VI) has also been recently used in an alkaline super-iron battery as cathode material.

Ferrate (VI) is not only one of the most powerful oxidizing agents ; it also has a negative environmental impact. Numerous researchers have attempted to synthesize Fe (VI) using thermal, wet chemical and electrochemical methods. In fact, the development of the existing chemical synthesis processes is hindered by the use of hazardous chlorinated compound. However, the electrochemical synthesis of Fe (VI) has been given more attention because it can be a simple, rapid, low costs and a relatively clean process with high Fe (VI) product. It may be considered environmentally friendly because it involves one-step process to convert Fe (0) to Fe (VI) without using harmful chemicals. This is due to the fact that it does not require high temperature or oxidizing agents and it is suitable for large scale production. Attempts to electrochemically synthesize Fe (VI) compounds have been mainly focused on the anodic dissolution of an iron metal anode into an alkaline solution at high potential.

However, little progress has been made in the efficient synthesis of Fe(VI) electrochemically because the process is strongly influenced by the anode material composition, the electrolyte concentration, temperature, and cell arrangement. One of the purposes of this paper is to define optimum operating conditions for the electrochemical ferrate (VI) production. NaOH concentration, current density and temperature were taken as three main parameters that influence ferrate (VI) production in an electrochemical cell.

**Keywords :** ferrate(VI), super oxidant, green oxidant, electrosynthesis of ferrate (VI)



[T5-PP 97]

**NON ENZYMATIC GLUCOSE SENSOR USING NICKEL (II)-SCHIFF  
BASE COMPLEX CARBON PASTE ELECTRODE.**

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**Abstract:**

Glucose is an important biomolecule for the human body; it is extensively used in food and beverage industry. The development of accurate, reliable and low cost devices for glucose detection presents one of the important subjects in scientific research [1]. The use of glucose oxidase enzymes (GOx) as modifier has extensively studied and shows high selectivity and sensitivity but this technology has some drawbacks, including high-cost, the difficulty of enzyme immobilization processes, oxygen dependency, and instability [2]. The development of non enzymatic glucose sensor attracts more interest due to the direct detection and the advantages of low cost, simplicity and good sensitivity.

In the present work, we report the use of carbon paste modified with nickel Schiff base complex as composite material for the detection of glucose in alkaline medium. Our electrode material was characterized with scanning electron microscopy (SEM), energy dispersive X-Ray spectroscopy (EDXS), cyclic voltammetry and chronoamperometry. The catalytic activity of our modified electrode was performed in presence of glucose in the range of 100 $\mu$ M to 15mM and the resulting catalytic current shows linear dependency with glucose concentration, we obtain very good sensitivity, good selectivity and excellent electrode stability.

**Keywords:** Ni(II)-Schiff base complex, Modified graphite paste electrode, Cyclic voltammetry, non enzymatic glucose sensor.

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[T5-PP 98]

**ELECTRODEPOSITION ET CARACTERISATION D'UN FILM DE  
Ag<sub>2</sub>O SUR L'ARGENT DANS UN MILIEU BASIQUE**

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**Résumé :**

Le but de cette étude est la détermination de la résistance de l'oxyde d'argent Ag<sub>2</sub>O élaboré par voie électrochimique. Pour réaliser cette investigation, des méthodes électrochimiques ont été mise en œuvre. Il s'agit du potentiel en circuit ouvert, la voltammétrie cyclique, la chronoampérométrie et la spectroscopie d'impédance électrochimique. Les résultats ont montré que l'application d'un potentiel égale à 0.28V/ECS sur l'électrode d'argent immergé dans une solution d'hydroxyde de sodium de concentration 0.1M permet le recouvrement total de la surface réactive, ces couches minces sont passives, homogènes et stables. En outre, la cinétique de croissance de l'oxyde est décrite par une équation logarithmique traduisant un processus contrôlé par la diffusion des espèces à travers le film. L'épaisseurs de film a été déterminée par coulométrie.

**Mots-clés:** Couche mince, Oxyde d'argent (Ag<sub>2</sub>O), Voltammogramme cyclique, Chrono ampérométrie

[T5-PP 99]

**PROPRIETES ELECTROCHIMIQUES ET SPECTROSCOPIQUES DES  
OLIGOMERES DU THIOPHENE ET PYRROLE**

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**Résumé :**

Les polymères conducteurs possèdent des liens délocalisés (souvent dans un groupe aromatique). Presque tous les polymères conducteurs connus sont des semi-conducteurs grâce à leur structure en bandes.

Le caractère conducteur d'un matériau organique est dû à la présence d'un système conjugué, c'est à dire à l'alternance de liaisons simples et doubles entre atomes de carbone le long de la chaîne polymérique.

Les différentes méthodes d'analyse électrochimiques et spectroscopique utilisées; montre que le type de germination qui a lieu peut être dû à une croissance tridimensionnelle instantanée. La diffusion des ions dans la solution devient l'étape limitante pour la réaction de croissance du film déposé.

La formation d'un film isolant dans quelques cas de mélanges indique l'occupation des sites active dans la matrice polymérique.

**Mots clés:** Oligomères, voltampérométrie cyclique, impédancemétrie, IR.

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[T5-PP 100]

**A NOVEL FERROCENYL-SUBSTITUTED NICKEL COMPLEX  
DERIVED FROM 5-CHLOROMETHYL-2-HYDROXYACETOPHENONE  
AND N-FERROCENYLMETHYLANILINE: SYNTHESIS, SPECTRAL  
AND EFFECT OF SOLVENTS POLARITIES ON THE  
ELECTROCHEMICAL BEHAVIOR OF  $Fc^+/Fc$  REDOX COUPLE.**

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**Abstract**

The synthesis, spectroscopic and structural characterization, as well as the electrochemical behavior of a neutral Ni(II)-centered organometallic Salen. The final Schiff base complex containing two redox centers ferrocene is the N,N-Bis[5-(N,N-ferrocenmethylaminophenyl)methylsalicylidyne]-1,2-diaminoethane of Nickel(II). The tetradentate Schiff base ligand (**IV**) was synthesized through the reaction of N-methyl-N-ferrocenmethylaminophenyl-2'-hydroxyacetophenone (**III**) with stoichiometric amount of 1,2-diaminoethane in absolute ethanol. Compound **III** was prepared by reacting 5-chloromethyl-2-hydroxyacetophenone (**I**) with N-ferrocenmethylamine (**II**) with presence of sodium hydrogenocarbonate ( $NaHCO_3$ ) in tetrahydrofuran solution. Heating a mixture of the Schiff base ligand (**IV**) in presence of a stoichiometric amount of forhydrated Nickel(II) acetate salt, in absolute ethanol at 50 °C under nitrogen atmosphere afforded the expected tetradentate Ni(II)-Schiff base complex. All the synthesized compounds were obtained in good yields (60%). Their compositions and purities were estimated from elemental analysis while, their molecular structures were elucidated with FT-IR, UV-Vis, and <sup>1</sup>H and <sup>13</sup>CNMR. For the nickel complex, the Ni<sup>II</sup> is tetracoordinated in a distorted square planar environment, with two nitrogen and two oxygen atoms as donor sites. The electrochemical behavior of the synthesized compounds was investigated by cyclic voltammetry. This method shows greater anodic shifts of the reversible redox process ascribed to the  $Fc/Fc^+$  couple of the ferrocenyl donor fragment of compound (**III**) in dichloromethane (**DC**) medium compared to the other polar medium acetonitrile (**AN**), dimethylformamide (**DMF**) and dimethylsulfoxide (**DMSO**).

**Key words:** Organometallic salen, N-ferrocenylmethylamine, Tetradentate Schiff base, Cyclic voltammetry.



**[T5-PP 101]**

**ETUDE DE L'ACTIVITE ELECTRO-CATALYTIQUE DE DEPOTS DE  
NICKEL RANEY DOPEES AU PLATINE SUR LA REACTION DE  
DEGAGEMENT D'HYDROGENE EN MILIEU ALCALIN  
CONCENTRE.**

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**Résumé :**

Dans ce travail, l'effet de l'incorporation de particules de platine dans des dépôts à base de Ni-Raney sur leur activité électro-catalytique vis-à-vis de la réaction de dégagement d'hydrogène (RDH) en milieu alcalin concentré a été effectuée en utilisant des techniques électrochimiques telles que la polarisation cathodique dynamique et la spectroscopie d'impédance électrochimique (SIE). La caractérisation a été effectuée au moyen de microscopie électronique MEB couplé à la microanalyse, EDX, et de diffraction aux rayons-X. Cette étude nous a permis à montrer que des électrodes à base de Nickel de Raney étaient très actifs par rapport au Ni électrolytique classique [1] d'une part et que la réaction de dégagement de l'hydrogène (RDH) sur ces dépôts spongieux était contrôlée suivant le mécanisme de réaction de Volmer- Hyrorwsky [2]. La modification de ce type d'électrode par déposition de particule de Pt améliore d'avantage l'activité et la stabilité par une augmentation notable de la résistance contre corrosion en milieux agressifs.

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[T5-PP 102]

**EFFECT OF THE USED SOLVANT NATURE ON THE COPPER  
ELECTRODEPOSITION KINETICS**

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**Abstract:**

Nowadays copper is largely used in the electronic industry to manufacture semiconductor based products. This is related to the recent replacement of Al with Cu in microelectronic interconnections production, which increased the performances in terms of lower electrical resistance, higher thermal conductivity and lower cost [1, 2]. As a side effect, the need of developing economically affordable and reliable processes to plate copper having the required features became a priority. Copper electroplating is commercially carried out using aqueous solutions [3] due to the high solubility of the corresponding metal salts resulting in high conductivity of the electrolytes and consequent good throwing power. Nonetheless, some issues such as their toxicity, corrosivity (pH <<1) and environmental impact led to the investigation of less dangerous electrolytes, like ionic liquids [4]. The use of these novel solvents is a promising way to solve the cited issues. Among the air and water stable ionic liquids, glycerin-based electrolytes were used due to their moisture stability, low price, biodegradability and non-toxicity.

The present work investigates for the first time the influence of the physical properties of glycerin on the deposition of copper on indium tin oxide (ITO)-coated conducting glass substrate, in particular the relation between chemistry, viscosity, conductivity of the solutions and the quality of the obtained Cu layer. The electrochemical study by cyclic voltammetry shows a difference in the deposition process of Cu from glycerin compared to the aqueous solution. On the other hand, the use of glycerin as solvent has reduced the cathodic peak current density which means that the deposition rate has been decreased. This decrease of rate deposition caused a remarkable change in the morphology and the microstructure of Cu deposits. The pattern of X-ray diffraction (XRD) peaks confirms that the films are pure Cu and all samples have a cubic structure with a notable change in preferential orientation, crystallite size and lattice parameter.

**Keywords:** Cu, glycerin, electrodeposition, nanostructures, copper.

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[T5-PP 103]

**SYNTHESIS, CHARACTERIZATION AND APPLIED OF NOVEL  
NICKEL (II)-SCHIFF BASE COMPLEX IN OXIDATION OF  
METHANOL AND BENZYL ALCOHOL**

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**Abstract :**

The development of new methods for the selective catalytic oxidation of alcohols to aldehydes and ketones is an important goal for chemical synthesis. Various systems for the catalytic oxidation of alcohols involving Schiff base complexes as catalysts are reported in the literature. The catalytic activity of these complexes varies with the type of ligands, metal ions and coordination sites [1-2]. In particular, numerous investigations have been made for the electrocatalytic oxidation of methanol and benzyl alcohol, the use of which is very diversified. Copper (II) and nickel (II) complexes are the two main types of complexes used in the oxidation of methanol and benzyl alcohol, the reaction mechanisms of which are complex [3-7].

In this work, we present the results of the synthesis and physicochemical characterization (CCM, Elemental Analysis, SM, <sup>1</sup>H NMR, UV-Visible, IR, and molar conductivity) and the electrochemistry of a new Schiff base ligand and its nickel complex. The catalytic activity of this complex on the oxidation of an aliphatic alcohol (methanol) and another aromatic (benzyl alcohol) is examined for different concentrations for the two alcohols. The study is carried out in a solution of dimethylformamide (10-1 M DMF / Bu 4 NPF 6 ) in the presence of 10<sup>-3</sup> M of the complex as a catalytic site. The study is carried out on a glassy carbon electrode and at a scanning speed of 25mV/s under oxygenated atmosphere and at ambient temperature.

This catalyst oxidizes both alcohols at low potential values suggesting high selectivity and stability under mild experimental conditions of temperature and pressure. Keywords : Catalytic oxidation, alcohol, oxidation, Schiff bases Ni(II) complexes.

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## Topic 7: Nanomaterial for Electrochemical and Photovoltaic Applications.

[T7-PP 104]

### NUCLEATION AND GROWTH MECHANISM OF Cu<sub>2</sub>O-N THIN FILMS ONTO FTO ELECTRODE USING THE ELECTROCHEMICAL DEPOSITION PROCESS

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#### Abstract:

Cyclic voltammetry and chronoamperometry were used to study the deposition process of Cu<sub>2</sub>O thin films on fluorine tin oxide-coated glass (FTO). The current transient data and predicted values were analyzed by using the Scharifker Hills nucleation models under different electrolyte concentrations. The effects of the Cu<sub>2</sub>O nucleation mechanism on electrochemical, microstructural and photoelectrical properties of Cu<sub>2</sub>O were investigated by Mott-Schottky analysis (MS), Photocurrent measurements (PC), X-ray diffraction (XRD), ultraviolet visible spectrophotometry (UV) and photoluminescence spectroscopy (PL). Many electrochemical parameters were investigated such as transfer coefficient, diffusion coefficient, cathodic and anodic charges, nucleation rate etc. Nucleation and growth mechanisms were also studied with different concentrations of Cu<sup>2+</sup> to elucidate the dependence of the nucleation mechanism of Cu<sub>2</sub>O on the concentration of Cu<sup>2+</sup>. It was found that the nucleation changes from progressive to instantaneous with the increase of Cu<sup>2+</sup> concentration. Under the instantaneous nucleation mechanism, the prepared Cu<sub>2</sub>O displayed a uniform structure with high crystallite size. Compared to the progressive nucleation, the purity of the Cu<sub>2</sub>O was higher, a narrow optical bandgap, and high photoluminescence intensity. The Cu<sub>2</sub>O had a high carrier density and a low charge transfer resistance. It exhibits superior photoelectric performance.

**Key words:** Electrodeposition, Cyclic voltammetry, Nucleation, Growth, Cu<sub>2</sub>O,

**[T7-PP 105]**

**CONTRIBUTION A L'ETUDE D'ENERGIE PHOTOVOLTAÏQUE  
DANS LA REGION DE BORDJ BOU ARREDJ**

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**Résumé :**

La promotion et le développement des énergies renouvelables en Algérie s'inscrivent dans un cadre de développement durable ayant comme objectif : la protection de l'environnement en favorisant le recours à des sources d'énergie non polluantes ; la contribution à la lutte contre le réchauffement climatique en limitant les émissions de gaz à effet de serre ; la participation à la politique nationale en matière d'aménagement du territoire par la valorisation des gisements énergétiques renouvelables. Nous prenons comme zones test des hauts plateaux telliens représentée par la région de Bordj Bou Arredj comme projet réalisé: (Ras El Oued, Ain Taourt) avec la contribution de l'entreprise (Condor) qui fabrique les panneaux de solaire. Ce projet ressort la nécessité de connaître l'évolution et l'introduction des matérielles technologies modernes comme énergie solaire donne beaucoup dans l'amélioration la production et le rendement dans le domaine agricole aussi pour l'élevage. Pour une meilleur planification et évolution de la céréaliculture dans les zones considérée, la méthodologie qui nous avons adopté, créer une complémentarité entre énergie solaire pour l'irrigation des zones agricole par l'eau aux se base sur les bombes d'électricité qui alimente par les panneaux solaire.

**Mots Clés:** Bordj Bou Arredj; énergie photovoltaïque; élevage; terre agricole; électricité

[T7-PP 106]

**SYNTHESIS AND PHOTOELECTROCHEMICAL  
CHARACTERIZATION OF COMPOSITE MATERIALS  
(MEH-PPV+TiO<sub>2</sub>)**

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**Abstract:**

Poly[2-methoxy-5-(20-ethylhexyloxy)-*p*-phenylenevinylene] (MEH-PPV), [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) and titanium dioxide (TiO<sub>2</sub>) nanoparticles (n-type) were dissolved, mixed and deposited by physical methods (spin-coating) on indium tin-oxide (ITO) substrate. The incorporation of the titanium dioxide nanoparticles changed the morphology and increased the roughness of polymers film (MEH-PPV/PCBM), and the photocurrent density of the composite (MEH-PPV/PCBM +n-TiO<sub>2</sub>) was higher than that of single MEHPPV/PCBM film. The study showed that the presence of n-TiO<sub>2</sub> particles in the polymeric film improves the photoelectrochemical properties of MEH-PPV/PCBM composite.

**Keywords:** Electrochemical property, Hybrid coating, Conducting polymer; Titanium dioxide



[T7-PP 107]

## ELABORATION ET CARACTERISATION D'UNE BICOUCHE ORGANIQUE/INORGANIQUE POUR DES APPLICATIONS DANS LES DIODES

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### Abstract:

Ce travail est basé sur la synthèse et la caractérisation des films minces bicouches ZnO/P3HT. Nous avons commencé par l'électrodéposition et la caractérisation de films minces d'oxyde de zinc (ZnO) et avec le revêtement par centrifugation pour les dépôts de P3HT et enfin l'évaporation de l'aluminium sous vide pour former les contacts de la diode (ITO/ZnO/P3HT/Al). Les matériaux obtenus sont caractérisés par des méthodes électrochimiques (voltampérométrie cyclique, chronoampérométrie), des méthodes morphologiques et spectroscopiques (AFM, DRX, UV-visible).

Les mesures photoélectrochimiques montrent que le semi-conducteur déposé (ZnO) est le type n. La caractérisation I-V de la structure bicouche ITO/ZnO/P3HT/Al montre les caractéristiques d'une diode.

**Mots-clés:** Oxyde de zinc, P3HT, caractérisation I-V, diodes

[T7-PP 108]

**EFFECT OF ANNEALING TREATMENT ON THE STRUCTURAL AND OPTICAL PROPERTIES OF ZN DOPED ITO THIN FILMS PREPARED BY SOL-GEL METHOD.**

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**Abstract:**

The Zn doped ITO thin films which commonly called IZTO were deposited by sol-gel spin-coating method onto glass substrates. The content of Zn in ITO solution is taken 1 at. %. For studying the stability of prepared films, an annealing treatment in air atmosphere on Zn doped ITO films was performed at 300, 400, 500 and 600°C for 1 hour. The structural parameters, such as texture coefficient ( $T_C$ ), grain size ( $D$ ), dislocation density ( $1/D$ ) and lattice constant were obtained from XRD analysis. The texture coefficient showed that for annealing temperature at 400 and 500°C, the IZTO films exhibited a stable cubic bixbyite structure with orientation along (400) plane. This orientation changed from (400) to (222) plane for 600°C. When increasing annealing temperature to 600°C, the grain size increased to 28nm and the lattice parameter decreased from 10.16 to 10.09 nm that indicates the presence of minimum dislocation density in heated films. The FTIR analysis carried on the IZTO films showed the presence of functional groups of In-O binding at  $595\text{cm}^{-1}$  which corresponds to the  $\text{In}_2\text{O}_3$  bixbyite structure. The optical properties from transmission spectra showed that all films have a transmittance higher than 90% in ultraviolet-visible region. In conclusion, the present results showed that the obtained thin films could be used in a photovoltaic application with the investigation of their electrical properties.

**Keywords:** Zn doped ITO, structural and optical properties.



**[T7-PP 109]**

## **HYBRID SOLAR CELLS USING A ZINC OXIDE AND POLYMER**

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**Abstract:**

We describe a new method heterojunction hybrid polymer solar cells based on composite films of zinc oxide (ZnO) and a polymer polyaniline (PANI) thin films were synthesized by electrochemical on a conducting ITO glass substrate. The physicochemical characterizations were made using Fourier transform infrared spectroscopy, UV-Vis spectroscopy. From the cyclic voltammetry measurements, the main reactions that take place on the electrode during the processes of electropolymerization are identified. The compact aspect of the layer is observed on the top of films by Atomic force microscopy (AFM).

**Keywords:** Composite, conducting polymers, polyaniline, electropolymerization.

[T7-PP 110]

## INFLUENCE OF ANODIZATION TIME ON THE GROWTH OF TiO<sub>2</sub> NANOTUBES AND THEIR PROPERTIES

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### Abstract:

In recent years, nanostructured materials have acquired great interest because of their many properties that make them active components in several applications. Highly ordered Titanium dioxide nanotubes (TiO<sub>2</sub>/NTs) have great importance in photovoltaïque application due to their one-dimensional (1D) nature and the high specific surface area. The synthesis techniques of self-organized TiO<sub>2</sub> NTs have been investigated using sol-gel technique, hydrothermal processes and electrochemical anodization method. Among these processes, electrochemical anodization of titanium in fluorinated electrolytes is a simple method to control the dimensions of NTs by varying anodization parameters. In this paper, the influence of anodization time on the morphology, structural and optical properties of TiO<sub>2</sub> NTs grown by electrochemical anodization process in ethylene glycol electrolyte containing 0.3wt% Ammonium fluorides (NH<sub>4</sub>F) and 2wt% ultrapure water at 60V for different anodization time (01, 02 and 03h) will be studied. The characteristics of these nanotubes were investigated using a scanning electron microscope (SEM), Energy Dispersive Spectroscopy (EDS), X-ray diffraction (XRD) and Ultraviolet-visible spectroscopy (UV).



[T7-PP 111]

## ELABORATION ET CARACTERISATION DES COUCHES MINCES Ni-Mo OBTENUES PAR ELECTRODEPOSITION

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### Résumé :

L'étude des couches minces représentent une thématique de recherche en plein essor du fait de leurs nombreuses applications dans différents domaines. Elles constituent une classe de nanomatériaux dont les propriétés dépendent étroitement des conditions d'élaboration.

Les revêtements Ni-Mo sont utilisés en tant que matériaux de protection dans les industries automobile et aéronautique et pour les pièces des machines fonctionnant à hautes températures et dans des environnements agressifs. Ces alliages sont caractérisés par une grande résistance à la corrosion, des propriétés mécaniques importantes et de bonnes propriétés catalytiques pour la réaction de dégagement d'hydrogène (HER) en milieu alcalin.

Dans ce travail, des couches minces Ni-Mo ont été élaborées par électrodéposition sur un substrat en Cu dans une solution acide de citrate de sodium à température ambiante en appliquant différentes densités de courant. Les dépôts ainsi obtenus ont été caractérisés par des techniques expérimentales complémentaires: la Microscopie Electronique à Balayage (MEB) associée à la microanalyse par Energie Dispersive des rayons X (EDX) et la diffraction des RX (DRX). Les résultats obtenus par MEB montrent que les revêtements Ni-Mo obtenus par application de courant direct (DC) à différentes densités de courant présentent une bonne qualité de surface et une morphologie relativement homogène. Les analyses par EDX montrent la présence du Ni et du Mo dans les couches déposées. Les résultats de la DRX révèlent le caractère nanocristallin des couches ainsi que la formation des différentes phases de Ni et de Mo. Les variations des paramètres structuraux et microstructuraux des différentes phases en présence seront discutées en fonction de la densité de courant.

**Mots clés :** Electrodéposition, couches minces Ni-Mo, DRX, EDX, MEB.

[T7-PP 112]

**PREPARATION OF N-TYPE Cl-DOPED Cu<sub>2</sub>O BY  
ELECTROCHEMICAL DEPOSITION FOR A P-N HOMOJUNCTION  
THIN FILM SOLAR CELL**

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**Abstract:**

Cu<sub>2</sub>O is a p-type semi-conductor with a band gap of 2.1 eV, which is adapted for solar energy conversion and other advanced applications. Moreover, it is based on non-toxic and abundant elements and can be deposited by low cost electrochemical route. In this work, chlorine doped Cu<sub>2</sub>O was achieved by adding copper chloride to the electrodeposition alkaline Cu (II) citrate electrolyte. The X-rays diffraction (XRD) showed that Cl-doped Cu<sub>2</sub>O films are pure and have a monocrystalline structure. The M-S plots demonstrate that the conduction type of the Cu<sub>2</sub>O films changes with increasing the carrier density by increasing CuCl<sub>2</sub> concentration in the deposition bath. The electrical resistivity of the as-deposited undoped film is in the order of  $5 \times 10^3 \Omega \cdot \text{cm}$  and decreases to order of  $10^2 \Omega \cdot \text{cm}$  when chlorine ions were incorporated in Cu<sub>2</sub>O lattice. This work is progressing to fabricate homo-junction p-Cu<sub>2</sub>O/n-Cu<sub>2</sub>O/FTO using the undoped and the Cl-doped Cu<sub>2</sub>O-n films with 0.01, 0.02 and 0.03 M of CuCl<sub>2</sub>, the I-V characteristics of homo-junctions shows electrical rectification behavior in dark with high photo-response. The optimal Cl concentration incorporated in the Cu<sub>2</sub>O film was found to be 0.01 M, which present a high photo-response. The optimized resistivity and the carrier density were approximately  $97.2 \Omega \cdot \text{cm}$  and  $2.05 \times 10^{20} \text{cm}^{-3}$ , respectively.

**Key words:** Electrodeposition, Semiconductor, Homojunction, Cu<sub>2</sub>O, Current-Voltage,

[T7-PP 113]

**NEW INVESTIGATION OF THE ELECTRODEPOSITED ZNS  
NANOSTRUCTURES BY CHRONOAMPEROMETRY PROCESS:  
EFFECT OF THE ZINC SULFATE CONCENTRATION**

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**Abstract:**

ZnS is one of the most important n-type semiconductor materials, with a wide direct band gap (3.5eV-3.7eV) [1] and a large exciting bending energy of 60 meV at room temperature [2]. It is used in a wide range of applications such as sensors, optoelectronic, photoelectrical and photovoltaic. This is mainly due to the nontoxicity, easy preparation and low cost of its development. However, with increasing demands of component with better performance, ZnS has consequently drawn a growing interest over the last decade in all its forms: thin films and powder. This material has been deposited using various technique [3]. In our work ZnS thin films was deposited on ITO glass substrate by electrodeposition technique.

The ZnS nanostructures was deposited from an aqueous solution containing  $10^{-3}$  M  $\text{Na}_2\text{S}_2\text{O}_3$  and  $10^{-3}$  or  $10^{-4}$  M of  $\text{ZnSO}_4$ . First, the electrochemical study is devoted to explaining the formation mechanisms of ZnS. The Morphological observation was carried out by scanning electron microscopy (SEM). It shows a significant change in the surface area of the obtained films as a function of the  $\text{ZnSO}_4$  content. Structural analysis by XRD indicates that all the samples crystallize with Zinc blende structure, with the (200) preferred orientation. While gap energy is in the order of 3.8eV estimated from, Tauc plots (UV-Vis measurements).

**Key word:** ZnS, nanostructures, XRD, UV-Vis.

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[T7-PP 114]

## ETUDE DES PROPRIETES OPTIQUES DE DIOXYDE DE TITANE

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### Résumé :

La déposition du film de dioxyde de titane (TiO<sub>2</sub>) par voie électrochimique et ses études optiques et photovoltaïques a été réalisée dans le but de développer coût-efficacité des procédés de synthèse alternatives et améliorer le rendement quantique des cellules photovoltaïques à base de (TiO<sub>2</sub>). L'élaboration des films de (TiO<sub>2</sub>) ont été réalisées dans une solution aqueuse de nitrate de potassium.

La caractérisation de film élaboré a été réalisée par méthodes électrochimiques (Voltampérométrie cyclique, Spectroscopie d'impédance Electrochimique (SIE)), spectroscopie UV-visible, les mesures des résistivités et les mesures des photocourants comme application dans les cellules photoélectrochimiques.

Les films de (TiO<sub>2</sub>) déposés résultent une amélioration dans les propriétés optiques et les réponses photoélectrochimiques.

**MOTS-CLES :** (TiO<sub>2</sub>), semi-conducteur, photocourants, résistivités.

[T7-PP 115]

**ELECTROCHEMICAL BEHAVIOR, OF  
POLY(TERTHIOPHENE+SEXITHIOPHENE)  
COPOLYMER COATINGS ON TRANSPARENT ITO GLASS  
SUBSTRATE**

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**Abstract :**

Conducting polymers have been studied during the last two decades as important semiconductor materials because of their excellent chemical and physical properties (1–3). Thus they can be used in various applications such as organic field effect transistors (OFETs) (4), polymer light-emitting diodes (PLEDs) (5), solar cells (6) and chemical and electrochemical sensors (7). Recently, more interest has been focused on various conjugated polymers such as polythiophene, polypyrrole and conjugated polymers. These materials have rapidly gained significant attention, due to the existence of  $\pi$ ,  $\pi'$ -linkages in their monomers, which make the whole poly terthiophene type chain grow regularly and leads in very interesting electronic, electrochromic and optical properties (8–10).

This work presents the synthesis, electrochemical and spectroscopic characterization of the copolymer: (P3T- $\pi$ 6T) obtained from the terthiophene (3T) and Sexithiophene (6T) at a platinum electrode or indium tin oxide ITO glass electrodes. The so obtained films were characterized using cyclic voltamperometry (CV), impedance spectroscopy measurement (EIS), UV-visible (UV-VIS), energy dispersive X-ray (EDX), scanning electron microscopy (SEM), to study their electrochemical properties. These modified electrodes can be used in various applications, such as light emitting diodes (LEDs) and photovoltaic cells.

**Keywords:** Terthiophene ; Sexithiophene copolymers; photovoltaic cells.

[T7-PP 116]

## ELECTROSYNTHESIS OF POLYANILINE / CARBON NANOTUBE NANOCOMPOSITE FOR ELECTROCHEMICAL SUPERCAPACITORS

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### Abstract:

Conducting polyaniline (PANI) has the highest environmental stability and is recognized as the only conducting polymer stable in air [1]. However, PANI has low conductivity, which limits its electrochemical performance and applications in developing electrical devices. To overcome this problem, carbon nanotubes (CNTs) have been assessed as potential candidates owing to their unique structure and excellent mechanical, electrical, and thermal properties as well as their high surface area [2,3].

The objective of this work is to prepare composites from the two materials to combine the advantages of PANI and CNTs.

The CNTs/PANI composite films were formed in situ by electrochemical polymerization of an aniline solution containing CNTs of 0.8 wt.%, with a three-electrode system using a SCE and platinum plates as the reference, counter and work electrode respectively.

The electrochemical performances of these composite films as active supercapacitor electrodes were investigated with cyclic voltammetry (CV) electrochemical impedance spectroscopy (EIS), and chronopotentiometry (CP) in 0.1 M H<sub>2</sub>SO<sub>4</sub>.

It was found that the CNTs/PANI composite films showed much higher specific capacitance (SC), better cyclic stability and more promising for applications in supercapacitors than a pure PANI film electrode.

**Keywords:** composites, electrochemistry, polyaniline, CNTS, supercapacitor.

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[T7-PP 117]

## AMELIORATION MORPHOLOGIQUE DES FILMS DE ZnO ELECTRODEPOSE SUR ITO

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### Résumé :

L'oxyde de Zinc (ZnO) avec différentes morphologies ont été élaborés sur l'oxyde d'étain dopé Indium (ITO) en utilisant deux techniques d'électrodépositions différentes. La méthode d'électrodéposition classique avec un choix du courant contenue pendant un temps bien définie et la deuxième méthode en mode pulse qui est besoin d'un courant pulsé suivit d'un nombre de pulsation bien déterminer.

L'analyse par la diffraction des rayons X (DRX) a présenté une diminution de taille des grains des films de ZnO préparés par la méthode pulse que la méthode classique d'électrodéposition. La caractérisation des échantillons par microscopie à force atomique (AFM) montre une modification notable de la morphologie avec une diminution de la rugosité (RMS) lors de la méthode pulse.

Les changements morphologiques et l'amélioration optique des films de ZnO obtenues par plusieurs procédés donnent une nombreuse ides sur les applications photonique.

**Mots-clés :** Morphologie, ZnO, Electrodeposition, pulse, films minces.

[T7-PP 118]

**STUDY OF STRUCTURAL PROPERTIES OF CSVT DEPOSITED  
CuIn<sub>0.8</sub>Ga<sub>0.2</sub>S<sub>2</sub> LAYERS SOLAR CELLS**

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**Abstract:**

Chalcopyrite compound family has received considerable attention because of their device applications in photovoltaic [1-2]. CuIn<sub>x</sub>Ga<sub>1-x</sub>Se<sub>2</sub> absorber layers have achieved conversion efficiencies more than 22% [3]. In this paper, the quality of CuIn<sub>0.8</sub>Ga<sub>0.2</sub>S<sub>2</sub> nanoparticles elaborated by close spaced vapor transport (CSVt) technique has been studied. Layers were grown on glass substrate at various substrate temperatures (480 °C and 500 °C) [4-5]. The surface morphology is characterized by scanning electron microscopy (SEM) and revealed the good crystalline of layers. The layers thicknesses estimated by cross-section with SEM were about 2.7 to 5.8µm. The nanoparticles chemical composition analyses were determined by Energy Dispersive Spectroscopy (EDS) and were found to be quasi-stoichiometric. The layers structure was analyzed by using X-Ray Diffraction (XRD) and it was found that layers exhibited highly crystalline chalcopyrite structure, with a preferential orientation in the (112) direction.

**Keywords:** CSVt, CuIn<sub>0.8</sub>Ga<sub>0.2</sub>S<sub>2</sub>, SEM, EDS, chalcopyrite structure.

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[T7-PP 119]

**MATERIAUX HYBRIDES POLYOXOMETALATE-PORPHYRINE ;  
GENERATIONDES PHOTOCOURANTS**

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Ferhat Abbas Sétif

**Résumé :**

La fabrication des matériaux hybrides ayant une activité et une stabilité améliorées pour la génération de photocourant reste encore un challenge majeur. Dans ce sujet nous avons tenté d'élaborer des matériaux hybrides nanostructurés à base de polyoxométallates, de nanoparticules métalliques (POM@M NP) et de colorants permettant de combiner des propriétés catalytiques différentes et représentent une voie intéressante à explorer en vue d'applications énergétiques. Au cours de ce travail, les propriétés photoélectrochimiques d'électrodes fonctionnalisées par des systèmes hybrides inorganiques-organiques à base de POMs ont été étudiées, en se focalisant plus particulièrement sur les performances de ces matériaux pour la génération de photocourant.

Ces copolymères hybrides POMs-porphyrine sont été directement obtenus par l'oxydation électrochimique de la 5,15-ditolyldiporphyrine (**H<sub>2</sub>T<sub>2</sub>P**) en présence de différents groupes pyridyles.

Dans un second temps et après les études spectrales, morphologiques, conductivité et les réponses photoélectrochimiques de ces films des POMs@MNP (M= Au, Ag) peuvent être incorporés sur ces copolymères de porphyrines cationiques via une réaction de méthanèse (échange des contre anions PF<sub>6</sub><sup>-</sup> par des groupements POMs@MNP (M= Au, Ag) en vue d'améliorer leurs réponses photoélectrochimiques. L'incorporation de nanostructures du type P<sub>5</sub>W<sub>30</sub>@AgNPs en surface du film, conduit à une augmentation spectaculaire du photocourant. Des résultats pertinents ont été observés attribués à la présence de nouveau de transfert d'électron photoinduit en cascade : de la porphyrine vers le pyridinium, puis du radical pyridinium vers P<sub>5</sub>W<sub>30</sub> ce qui retarde la recombinaison des charges. L'augmentation de l'efficacité peut aussi être due également au plasmon de résonance des nanoparticules P<sub>5</sub>W<sub>30</sub>@MNPs (M = Ag, Au) qui améliore considérablement l'excitation électronique mais aussi probablement la conductivité du film sous illumination.

**Mots clés :** Porphyrin, POMs, Nanoparticules, Photocourant...

[T7-PP 120]

**POROUS SILICON MULTILAYERS FABRICATED BY  
ELECTROCHEMICAL ANODIZATION PROCESS.**

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**Abstract :**

Porous silicon (PS) is an excellent material for engineering; it can be produced by electrochemical etching of silicon wafer in solution based on hydrofluoric acid. It consider as suitable material to produce multilayer structures since one can change refractive indices and thicknesses of the PS layers etched by changing the current density and etching time during the anodization process. Bragg mirror is one-dimensional photonic band gap structure that controls the propagation of electromagnetic wave through photonic crystal. Several studies have used it as back reflector to enhance optic confinement of low energy photons in thin silicon solar cells. In this context, a theoretical model relying on the Bruggeman's effective medium approximation (BEMA) and stratified medium theory with its matrix representation was established to model the reflectance spectra and to choose the parameters of bi-layer periodically stacked (index of layers and its porosities, numbers of bi-layers, Bragg wavelength) that allow to improve photons reflection in wave range 800-1200 nm. Calculation results show that the bandwidth Bragg mirror is wader for layers having higher refractive index contrast. The Numbers of bi-layers contributes in improvement of the maximum reflectivity. In the other hand, Bragg mirror with ten bi-layers was fabricated by electrochemical etching of crystalline silicon wafer using current density of 5 and 200mA/cm<sup>2</sup> in HF-containing solution. Maximum measured reflectivity for the prepared mirror achieves approximately 95% in 800-1200nm region spectral.

**Keywords:** Electrochemical etching; Porous silicon; Bragg mirror; Reflectivity; Porosity.

[T7-PP 121]

## SYNTHESIS AND CHARACTERIZATION OF Mn DOPED FTO THIN FILMS DEPOSITED BY SPRAY PYROLYSIS

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### Abstract:

Transparent conductive oxides (TCOs) are interesting materials for several applications, because of their important properties, such as electrical conductivity and transparency in the visible region. This make them ideal candidates for optoelectronics, photovoltaic and catalytic applications. Among the different TCOs, SnO<sub>2</sub> films doped with fluorine (FTO) seem to be the most appropriate for these applications, owing to their low electrical resistivity, high optical transmittance in the visible region and a high infrared reflectivity. This material finds numerous applications, some of which includes liquid crystal displays, light emitting diode, dye-sensitized solar cells, thick-film sensor, transparent electrodes in solar cells, flat panel displays, and heat mirrors.

In this work we report on the properties of Mn-doped FTO thin films synthesized by spray pyrolysis method. X-ray diffraction analyzes of the FTO: Mn thin films showed that the prepared thin films are polycrystalline and have a tetragonal structure with (200) as a preferred orientation. The doping of FTO thin films with Mn shows significant changes in the transmittance characteristics in the visible range. The average transmittance of these doped films is higher than 90%. The photoluminescence (PL) behavior of Mn-doped FTO thin films was also studied. The obtained results showed that the PL intensity peaks increased when the doping concentrations was increased. The results obtained in this work have shown that electrical properties of the FTO: Mn thin films where greatly improved making them very suitable for ohmic contacts in devices for photovoltaic applications.

**Keywords:** FTO Thin films, Mn doping, Photoluminescence, Resistivity, photovoltaic applications.

[T7-PP 122]

## INFLUENCE DE NOMBRE DE TREMPAGE DU MATERIAU COMPOSITE SUR LA QUANTITE DE COBALT INCORPOREE DANS UN FILM DE POLYPYRROLE FONCTIONNALISE

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### Résumé :

Ce travail consiste à l'élaboration de nouveaux matériaux d'électrodes composites par la complexation et l'électroréduction du cobalt dans un film de poly [acide 4-(pyrrol-1-yl) benzoïque]. Le dépôt du film de polymère à la surface de l'électrode en ITO est obtenu par oxydation électrochimique du monomère en milieu organique.

L'incorporation des particules du cobalt dans le film de polymère est réalisée par trempage de l'électrode modifiée dans une solution de chlorure du cobalt pour complexer les cations cobalteux, suivi d'une électroréduction dans une solution aqueuse pour précipiter le cobalt sous forme des particules métallique dans le film de polymère.

L'analyse d'une même électrode sur laquelle, ont été fait, plusieurs opérations successives de trempage, de réduction, et d'oxydation, montre que les propriétés physicochimiques de l'électrode modifiée obtenu restent inchangées même après plusieurs opérations, suggérant, ainsi, une bonne stabilité du matériau composite obtenu, ce qui rend possible son application comme matériau d'électrode pour l'électrosynthèse de certains composés organiques.

Nous avons ensuite caractérisé notre matériau composite par voltamétrie cyclique, microscopie électronique à balayage et par spectrométrie à fluorescence X.

**Mots-clés :** électrodes modifiées, polypyrrole fonctionnalisées, cobalt.

[T7-PP 123]

**ELECTRODEPOSITION AND CHARACTERIZATION OF INDUIM  
FROM CITRATE ELECTROLYTE-EFFECT OF POTENTIAL ON THE  
NUCLEATION MECHANISM**

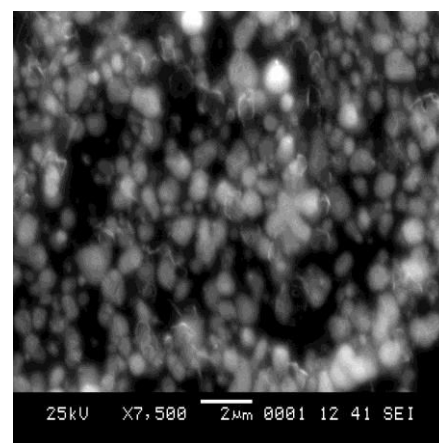
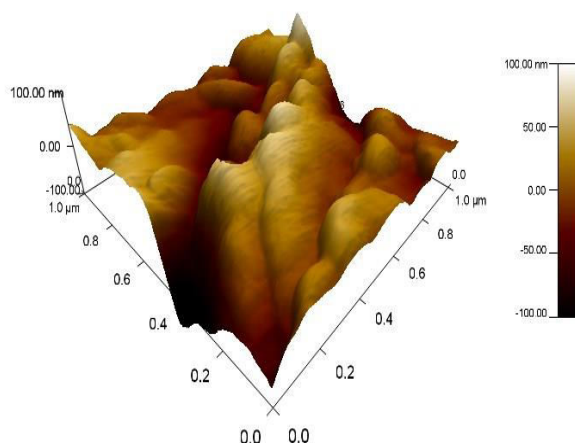
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**Abstract:**

In this work, cyclic voltammetry and chronoamperometry were used to study the influence of potential on the electrocrystallization kinetics of electrodeposited indium on platinum and ITO (indium tin oxide) substrates from acid medium (PH=4.2) containing indium trichloride  $\text{InCl}_3$  and sodium citrate at ambient temperature. The potentiostatic current transients were analyzed according to Scharifker-Hills model. The morphological characterization of the deposits was carried out by Scanning Electron Microscopy (SEM), whereas the Atomic Force Microscopy (AFM) was used to investigate its roughness. The results shown that the nucleation mechanism of (In) on the two substrates is instantaneous with a three-dimensional growth of the hemispherical nuclei. The nucleation density ( $N_0$ ) and the time max ( $t_m$ ) evolved exponentially with applied potential, whereas the max of current density ( $i_m$ ) is laniary evolved with the applied potential, the grains of the deposits obtained at -1.2 and -1.3 have spherical forms with nanometer size and possesses less rough surface.

**Keywords:** electrodeposition; nucleation; indium; thin film.



[T7-PP 124]

## EFFECT OF ANNEALING TEMPERATURE ON THE PROPERTIES OF Cu<sub>2</sub>O THIN FILMS PREPARED BY ELECTROCHEMICAL DEPOSITION

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### Abstract:

Cuprous (Cu<sub>2</sub>O) and cupric (CuO) oxides are considered to be the materials of large strategic potential from the viewpoint of their application in photovoltaics. In this work, cuprous oxide thin films have been grown by electrodeposition technique onto FTO coated glass substrates from a sulfate medium at 60 °C and pH of 11. CuO thin films were obtained on FTO substrates using an electrodeposited Cu<sub>2</sub>O thin films followed by annealing at 300 to 500 °C for 1 h. X-ray analysis showed that the increasing of temperature up to 500 °C favored the formation of monoclinic CuO from cubic Cu<sub>2</sub>O electrodeposited on FTO with a high crystallinity. The average crystallite size decreases from 63.89 to 43.25 nm by raising the temperature to 400 °C, then increased when the deposition was annealed at 500 °C. UV-Vis analysis was used to determine the optical properties of Cu<sub>2</sub>O and CuO nanostructures. The transmission of our samples is high; it is in the order of 70 % in the visible range and the optical band gap value of thin films decreases from 2.5 to 1.9 eV with increasing annealing temperature. The effect of annealing on the microstructure of copper oxides thin films was investigated with atomic force microscopy (AFM). AFM images showed that the grains size is increased whereas the surface roughness is increased by increasing in annealing temperature. Photo-electrochemical measurements of both oxides confirmed that these films behave as a semiconductor of p-type and presents high photo cathodic-generated currents of 0.45 mA/cm<sup>2</sup> of Cu<sub>2</sub>O film annealing at 500 °C, which exhibited a two order increase over the as-deposited sample. We attributed the enhanced photo-electrochemical current to the improved crystallinity and reduced defects for the annealed Cu<sub>2</sub>O films.

**Keywords:** Annealing, Cu<sub>2</sub>O, CuO, Electrodeposition, Nanostructures.

[T7-PP 125]

## OPTICAL PROPERTIES OF Cd<sub>x</sub>Zn<sub>1-x</sub>S THIN FILMS DEPOSED BY CHEMICAL BATH DEPOSITION

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### Abstract:

Thin films of Cd<sub>x</sub>Zn<sub>1-x</sub>S were deposited on glass substrates by chemical bath deposition for photovoltaic applications. The thin films CdZnS were synthesized by chemical bath (CBD) with different deposition protocols for optimized the parameter of deposition as the temperature, time of deposition, concentrations of ion and pH. Surface morphology, optical and chemical composition properties of these films CdZnS were investigated by SEM, EDS, XRD, AFM and spectrophotometer. The transmittance is 80% in visible region 300 nm – 1000 nm; it has been observed the grain size is between 90 nm and 150 nm. It is observed that the transmittance and the shape are change with the concentration of Zinc in the solution. This result favors of application these films in solar cells; the chemical analysis with EDAX gives information about the presence of Cd, Zn and S elements. This result favors of application these films in solar cells.

**Keywords:** CdZnS, chemical bath, thin films, solar cells.

[T7-PP 126]

**GROWTH AND CHARACTERIZATION OF ELECTRODEPOSITED  
Cu<sub>2</sub>O THIN FILMS**

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**Abstract:**

Cuprous oxide (Cu<sub>2</sub>O) is a low-cost, environmentally benign and important p-type semiconductor with a direct bandgap of 1.9–2.5 eV. Owing to its suitable band gap, high absorption coefficient in the visible region, high mobility and large diffusion length of minority carriers, Cu<sub>2</sub>O is promising for photovoltaic cells, catalysis, transistors, chemical sensor, photocathode for H<sub>2</sub> production and electrodes for lithium batteries. Among the various techniques available for the preparation of Cu<sub>2</sub>O thin films, the method of electrodeposition is an attractive technique because of its simplicity and usefulness [1-4].

In this work, Cu<sub>2</sub>O thin films were potentiostatically electrodeposited on FTO glass substrate at different applied potentials from aqueous cupric sulfate solution with citric acid at 70 °C. The effects of deposition potential on the electrochemical, surface morphology, structural and optical properties of Cu<sub>2</sub>O thin films were investigated. During cyclic voltammetry experiences, the potential interval where the electrodeposition of Cu<sub>2</sub>O is carried out was established. The Mott–Schottky (M-S) plot demonstrates that all the films are p-type semiconductors, the flat-band potential and the acceptor density for the Cu<sub>2</sub>O thin films are determined. Atomic force microscopy (AFM) images reveal that the applied potential has a very significant influence on the surface morphology and size of the crystallites of thin Cu<sub>2</sub>O. The XRD measurements indicated that all the obtained films display a Cu<sub>2</sub>O cubic structure with a strong preferential orientation of the (111) direction. The optical transmission spectra in the UV-Visible domains revealed the highest transmission (70 %), and their calculated gap values increased from 1.93 to 2.31 eV, with increasing potentials.

**Keywords:** Cu<sub>2</sub>O, electrodeposition, semiconductor, applied potential.

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[T7-PP 127]

**SYTHESIS AND CHARACTERISATION OF NANOSTRUCTURED p  
AND n-TYPE CUPPROUS OXIDE (Cu<sub>2</sub>O) FOR PHOTOVOLTAIC  
APPLICATIONS.**

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**Abstract:**

In this work, we report the synthesis and characterization of high quality p- and n-type nanostructured cuprous oxide (Cu<sub>2</sub>O) semiconducting thin films onto ITO substrate. Potentiostatic electrochemical technique was used to study the deposition of the thin film from a friendly low-cost lactic acid to give p-type or acetate-based electrolyte to give n-type photo-active thin films. The optimum experimental conditions (temperature, pH and applied potential) were determined. The characterization of the deposits was carried out by means of X-ray diffractometry (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM) and UV-Vis spectroscopy. The band gap of the films was ranging from 2.1 to 2.4 eV which is suitable for solar cell application.

**Keywords:** Electrodeposition; Thin films; Cu<sub>2</sub>O; Solar cells.

[T7-PP 128]

**In<sub>2</sub>Se<sub>3</sub> BUFFER LAYER GROWN BY PVD TECHNIQUE FOR THIN  
FILM SOLAR CELLS BASED ON CZTS ABSORBER**

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**Abstract:**

Recently, there is a significant increase in the interest for III-VI materials, which find applications in the electronic industry in photovoltaic applications [1, 2]. Indium selenides are III-VI compounds, In<sub>2</sub>Se<sub>3</sub> exists in three different structures: a defect cubic structure  $\alpha$ -In<sub>2</sub>Se<sub>3</sub>, which transforms into a defect spinel,  $\beta$ -In<sub>2</sub>Se<sub>3</sub>, at 693 K and into a layered structure,  $\gamma$ -In<sub>2</sub>Se<sub>3</sub>, at 1013 K [3]. In this work, we reported the effect of substrate temperature on the structural and optical properties of In<sub>2</sub>Se<sub>3</sub> nanoparticles deposited on soda-lime glass substrates by physical vapor deposition technique (PVD) at various substrate temperatures.

The In<sub>2</sub>Se<sub>3</sub> material used for deposition was synthesized from its constituent elements. It was found that all samples exhibit one phase which corresponds to  $\beta$ -In<sub>2</sub>Se<sub>3</sub> phase. Values of band gap energy of the films obtained at different substrate temperatures vary in the range of 2.38-2.80 eV and decrease with increasing substrate temperature.

**Keywords:** III-VI materials, In<sub>2</sub>Se<sub>3</sub>, nanoparticles, PVD.

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[T7-PP 129]

**ETUDE DE LA CROISSANCE DES NANOFILS DE ZNO SANS ET  
AVEC LA COUCHE TAMPON DE ZnO**

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**Résumé :**

L'oxyde de zinc (ZnO) est un semi-conducteur de type n à large bande interdite (3.37 eV), une grande énergie de liaison de l'exciton de (60 meV) [1]. ZnO a suscité un intérêt croissant en raison de sa capacité à former une variété de nanostructures comme : couches minces, réseaux ordonnés de micro- et nano-plots, réseaux libres de nanofils [2] ce qui lui rend un matériau largement utilisé dans diverses applications technologiques telles que les cellules solaires, optoélectronique, les diodes électroluminescentes, les lasers...ect [3-4].

Dans ce travail, nous avons étudié la croissance des nanofils de ZnO déposés directement sur un substrat de verre semiconducteur d'ITO à partir d'un bain mixte de nitrate et de chlorure de zinc avec (150 nm) et sans (0 nm) une couche tampon de ZnO. La variation des propriétés électrochimiques, structurales et optiques des nanofils de ZnO déposés sans et avec la couche tampon de ZnO a été étudiée. Les mesures de Mott-Schottky confirment que le ZnO a une conductivité de type n, un décalage du potentiel de la bande plate ( $E_{fb}$ ) et une augmentation du nombre de porteur de charge ( $N_D$ ) de  $4,27 \times 10^{20}$  à  $9,27 \times 10^{20}$  ( $\text{cm}^{-3}$ ) est observé en présence de la couche tampon de ZnO. Pour la spectroscopie d'impédance électrochimique (SIE), une résistance de transfert de charge de 100.4 kΩ a été obtenue. Les spectres de diffraction des rayons X indiquent que les nanostructures de ZnO électro-déposées sans et avec la couche tampon présentent une structure hexagonale avec une orientation préférentielle selon l'axe (002). En outre, la cristallinité des échantillons est améliorée en présence de la couche tampon de ZnO et la taille des grains a augmenté de 94,86 à 97,23 nm.

La spectroscopie UV-Visible montre une forte transmittance de l'ordre de 80% et un gap optique de 3,16 et 3,37 eV pour les dépôts avec et sans la couche tampon de ZnO, respectivement.

**MOTS – CLES :** ZnO, nanofils, Mott-Schottky, couche tampon.

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[T7-PP 130]

**EFFECT OF Co CONTENT ON STRUCTURAL PROPERTIES OF  
ELECTRODEPOSITED NANOCRYSTALLINE Co-Ni THIN FILMS**

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**Abstract:**

Nanocrystalline Co-Ni thin films were produced on Cu substrate by electrodeposition method from chloride-sulfate aqueous bath with different Co content in the bath and addition agents including boric acid and thiourea.

The chemical composition, crystalline structure and morphology of the electrodeposited Co-Ni films were investigated using Inductively Coupled plasma-Optical Emission Spectrometry (ICP-OES), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) techniques respectively.

The chemical analysis results confirm the formation of the Co-Ni coatings according to an anomalous codeposition phenomenon. The composition of all electrodeposited alloys depends on Co content in the bath. The SEM observations show that the coatings surfaces are uniform without cracks.

The X-ray diffraction profile fitting through the Rietveld refinement using MAUD software reveals that the coatings are nanocrystalline with the presence of Co and Co(Ni) phases. At low Co content, the FCC structures are the dominant phases. Above 50 at.% Co, the deposits exhibit a large percentage of HCP structures at the expense of FCC structure. The refinement results reveal that the crystalline lattice parameters of Co and Co(Ni) are strongly dependent on the Co content in the bath.

**Keywords:** Nanostructured coating, Co-Ni alloys, Electrodeposition, Rietveld analysis, anomalous codeposition

[T7-PP 131]

## ÉLABORATION ET CARACTERISATION D'UN MATERIAU COMPOSITE PALLADIUM-POLYPYRROL Pd-PPY/ITO

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### Résumé:

Un nouveau matériau composite à base de film de poly (pyrrole) (PPy) contenant des particules de palladium (Pd) a été synthétisé par une méthode électrochimique utilisant une procédure en deux étapes. Tout d'abord, le film de PPy a été déposé sur un substrat d'oxyde d'indium et d'étain (ITO) par oxydation du monomère pyrrole dissous dans l'eau (H<sub>2</sub>O) contenant du perchlorate de lithium (LiClO<sub>4</sub>) comme sel de support. Ensuite, l'incorporation de particules de Pd sur le film de PPy a été réalisée par immersion de l'électrode modifiée dans une solution de Pd. L'électrode obtenue (PPy-Pd / ITO) a été caractérisée par voltammétrie cyclique (CV), chronoampérométrie. L'étude voltampérométrique du film de PPy obtenu montre un pic anodique et cathodique caractéristiques de l'oxydation et de la réduction du polymère formé, les courants de ces pics augmentent au cours du cyclage, attestant la formation du film de polymère sur la surface de l'électrode.

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**ELECTROCHEMICAL AND PHOTOCONDUCTING PROPERTIES OF  
ITO/POLYBITHIOPHENE–MnO<sub>2</sub> COMPOSITE FILMS**

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**ABSTRACT:**

Polythiophenes constitute a particularly important class of conjugated polymers, which has been extensively studied for the relation between the geometrical structure and the optic and electronic properties. They are, furthermore, chemically and thermally stable materials, and are very attractive for exploitation of their physical properties [1]. The observation of photocurrents from polythiophene-modified metal electrodes has been reported by several groups of investigators [2, 3].

In this work, a composite thin film containing polybithiophene (PBTh) and manganese dioxide (MnO<sub>2</sub>) on indium tin oxide (ITO) glass substrates was prepared by electro-polymerization of bithiophene in the presence of MnO<sub>2</sub> nanoparticles. The films ITO/PBTh–MnO<sub>2</sub> are characterized by AFM and SEM, the analyses show an increase in roughness and the incorporated MnO<sub>2</sub> particles are of size in the range of 50 nm.

As a result, the optical gap is shifted by the incorporation of MnO<sub>2</sub> nanoparticles from 2.23 eV for ITO/PBTh to 2.03 eV for ITO/PBTh–MnO<sub>2</sub>. The photocurrent measurements indicate that the ITO/PBTh–MnO<sub>2</sub> films show a value that is three times higher than that of polybithiophene substrate, so that such a composite can be used as a new active material in solar cells.

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[T7-PP 133]

## ELECTROCHEMICAL SENSOR FOR HYDRAZINE DETERMINATION USING CARBON PASTE ELECTRODE MODIFIED BY AgO NANOPARTICLES.

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### Abstract:

In recent years, green nanotechnology is gaining more attention due to its ecofriendly and economical approach to nanoparticle synthesis [1]. As a result, researchers in the field of nanoparticle synthesis have turned to biological systems like plant extracts [2]. In this study, we synthesized silver (Ag) nanoparticles by green voice using *Rosmarinus officinalis* plant extract at room temperature. These nanoparticles were calcined, and characterized using scanning electron microscopy (SEM) coupled with EDX and, using X-ray diffraction. Scanning electron microscopy (SEM) shows spherical shape of nanoparticles was obtained, X-Ray diffraction (XRD) pattern shows face cubic center (fcc) structure and the average crystallite size was calculated to be about 19 nm. These nanoparticles were used to modify carbon paste electrode for the study of the electrocatalytic determination of Hydrazine by cyclic voltammetry and chronoamperometry techniques. The presence of this nanoparticles in carbon paste exhibit a higher electrocatalytic activity of hydrazine oxidation in phosphate buffer (pH = 8) at 435 mV/ECS. Results obtained show an excellent electrocatalytic activity toward hydrazine with the best sensitivity and good selectivity, than the prepared electrode can be used as an electrochemical sensor for uric acid.

**Keywords:** Nanoparticles, hydrazine, carbon paste electrode and *Rosmarinus officinalis*.

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