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High performance electrochemical sensor based on task specific ionic liquid mixed palm shell activated carbon for trace level Cd (II) detection

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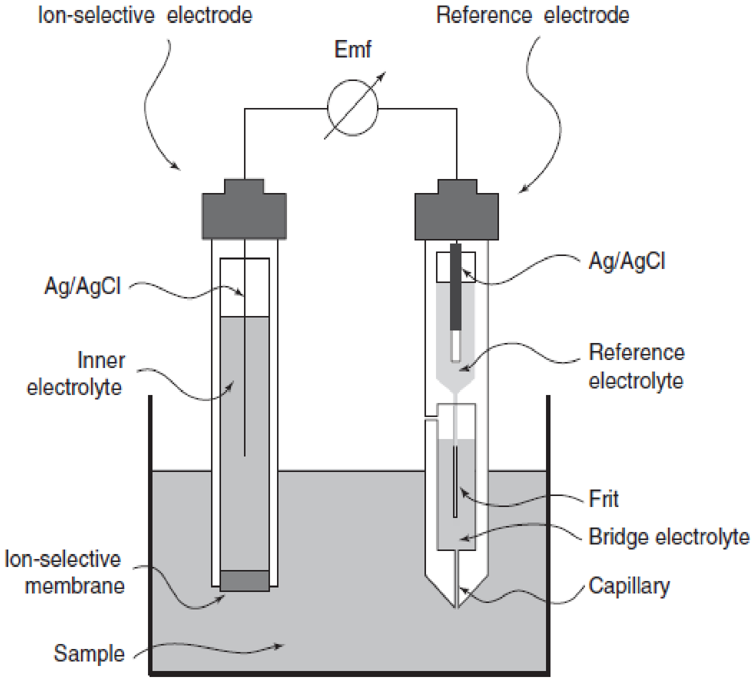
Overview

- The contamination of water resourses by heavy metal is a serious worldwide environmental problem. Numerous metals such as cadmium, mercury, chromium, lead, etc. are known to be toxic.
- The investigation of heavy metal ions concentration has been of most importance in environmental issues.
- Many of analytical techniques including atomic spectroscopy (AS), inductively coupled plasma and a variety of electrochemical techniques have been used for the analysis of heavy metals in real samples.
- Potentiometry is the most commonly used of the electrochemical techniques. We shall restrict our work to ISEs.

Ion Selective Electrodes

What is Ion Selective Electrodes?

Ion Selective Electrodes (ISEs) is a potentiometric sensor where used to measure some of the most critical analytes on environmental samples.



Component of ISEs

Ion selective electrode consists of some components.

These components are:

- The polymeric matrix, or carbon materials (graphite, activated carbon, carbon nanotubes).
- The ionophore (membrane - active recognition).
- The membrane solvent (Plasticizer).
- The ionic additives.

Properties of ISEs

According to the IUPAC recommendation, the essential properties of an ion-selective electrode are characterized by parameters like:-

- Detection limit
- Linear response range
- Slope
- Selectivity
- Sensitivity

IUPAC, Analytical Chemistry Division, Commission on analytical nomenclature, Recommendations for Nomenclature of Ion-Selective Electrodes, Pure Appl. Chem. 48 (1976) 127-132.

Advantages of ISE with other analytical methods

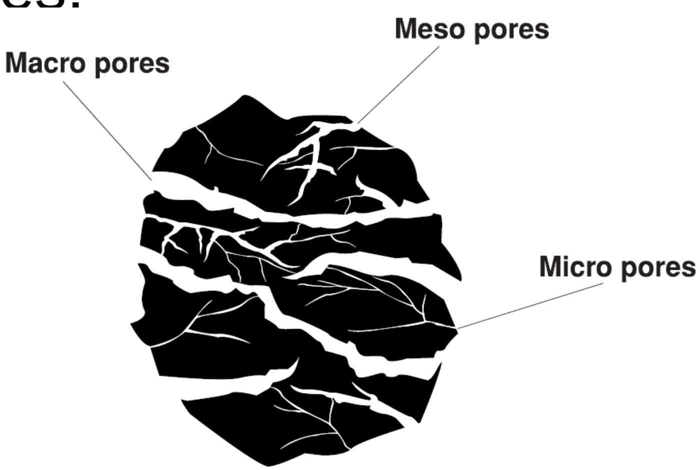
When compared with other analytical methods, ion-selective electrodes are very attractive for determination of many chemical species because they have many advantages such as:

- Low cost.
- Ease of preparation.
- Sensitivity.
- Selectivity
- Good precision

Why Palm Shell Activated Carbon ?

This material is easily available at low cost and have become an extremely popular theme in recent electrochemical sensing research, due to their:

- Good electrochemical properties.
- Good electrical conductivity,
- High surface area,
- High porosity,
- Good electrochemical stability



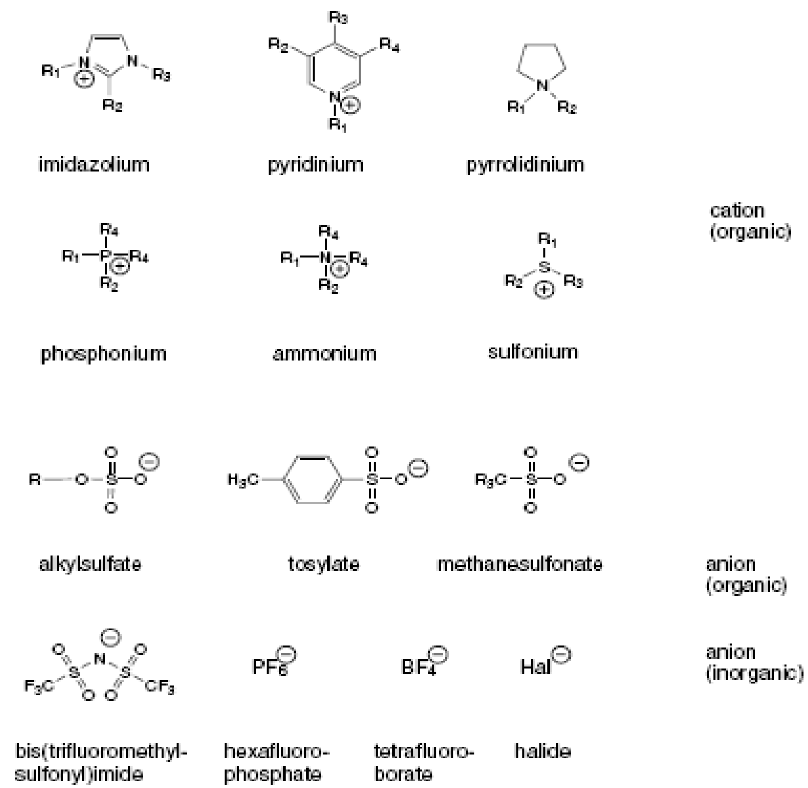
The schematic of internal pore structure in activated carbon

What is task specific ionic liquids (TSILs)?

- TSILs are subclass of ILs.
- ILs are air and water stable liquid organic salts.
- ILs are composed from an organic cations
- And either an organic or inorganic anion.

The physical and chemical properties of IL as well as TSIL

- Highly polar,
- Conductivity,
- Viscosity,
- Lewis acidity
- Hydrophobicity



The structure of some of ionic liquids.

Problem Statement

- The determination of low-level contaminants in ground water has become a major issue during the last several decades.
- The continuous monitoring of water pollutants in the field requires portable fast response sensors with sufficient sensitivity and high selectivity.
- The determination of pollutants in ground water requires new and improved techniques for rapid and low-cost monitoring.
- Although, their easy fabrication, simple usage, and low cost, ISEs suffer from long response times, low response sensitivity, interference by a number of metal ions, long equilibration times and short lifetimes.
- As a result, the development of new ISE materials that can address some of these limitations is a worthwhile and challenging topic of research.

Objectives and scope of work

- The ultimate goals of this study are to increase the sensitivity and selectivity of the proposed electrodes by minimizing the previously mentioned undesirable electrode processes. Additionally, the application of plasticizer-free electrodes can eliminate the leaching of the electrode solvent and sensing components, improving the electrode lifetime.
- The main objective of this study is to develop and evaluate the performance of novel ion selective electrode for the determination of Cd(II) in aqueous solutions.
- The research were include preparation of the ISEs, determination of their analytical characteristics, and their applications in the monitoring of ion concentration in drinking water samples.

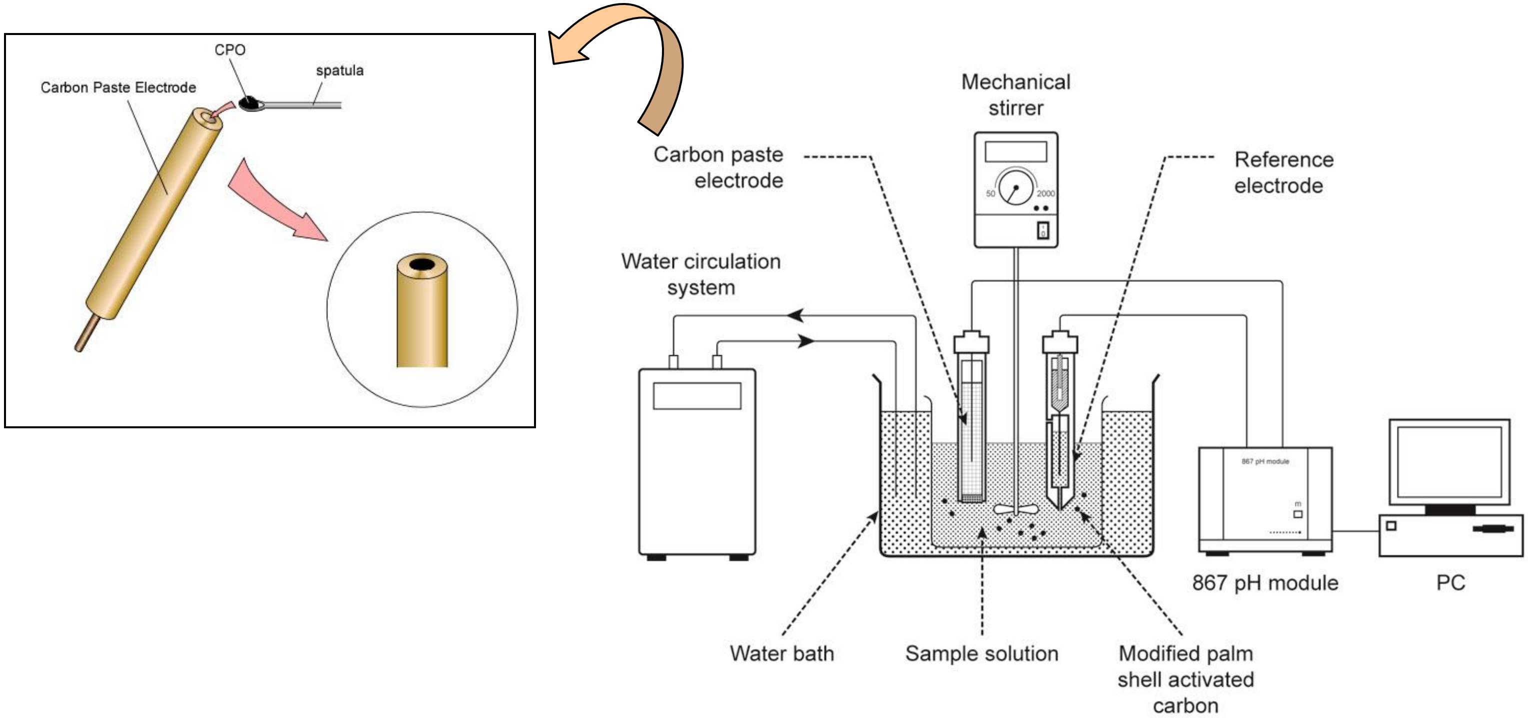
Significance of the study

This work reports significant, novel results pertaining to the detection and measurement of cadmium in aqueous media. These novelties can be summarized as follows:

- To the best of our knowledge, we are the first to propose this electrochemical sensor, based on a task specific ionic liquids carbon paste electrode, to detect cadmium in aqueous solution.

- More importantly, this work is the first to demonstrate that TSILs can have dual functions, acting as an ionophore and plasticizer. This finding will likely have a strong impact on potentiometric sensor technology because ionophores and plasticizers are usually different materials.

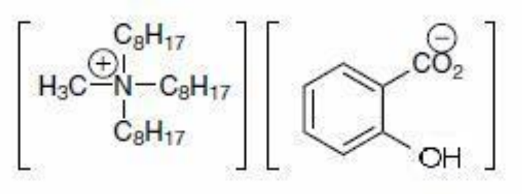
Methodology



Potentiometric experimental setup

Methodology

- In this work, the application of TOMAS in ion selective electrode in order to fully understand its functions as both ionophore and plasticizer in potentiometric sensor.

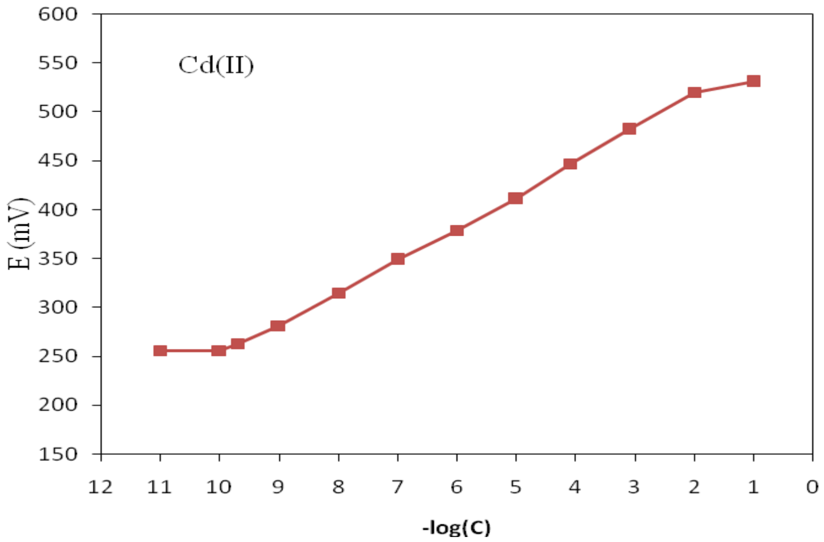


Structure of TOMAS

RESULTS AND DISCUSSION

Response of Cd (II) electrode

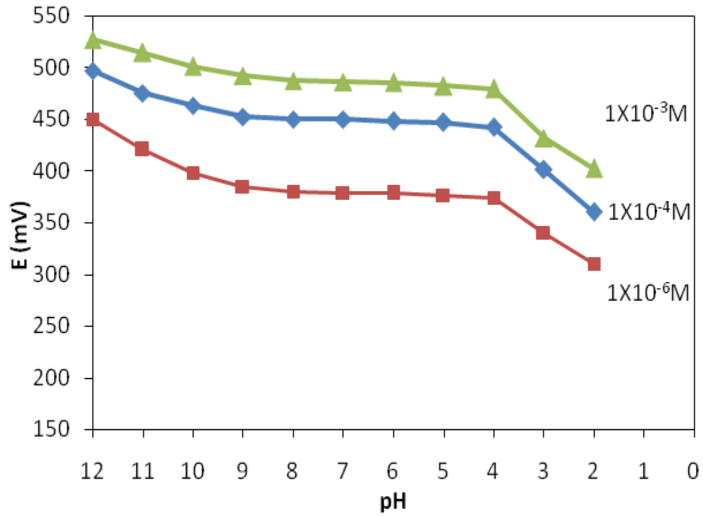
- The slope: 30.9 mV/dec closes to (29.5 mV/dec for bivalent cation).
- Response concentration range of 1.0×10^{-9} to 1.0×10^{-2} M.
- Detection limit: 1×10^{-10} M



The calibration curve for a modified palm shell activated carbon paste electrode over a wide range of solution Cd^{2+} .

RESULTS AND DISCUSSIONS

Effect of pH on the potential response.



Effect of pH on the potential response of Cd^{+2} palm shell activated carbon paste electrode.

From the figure we can see the electrode potential remain almost constant in the pH range of 4-9 this due to protonation of carboxylic group and formation of hydroxy complexes

RESULTS AND DISCUSSIONS

Potentiometric Selectivity Coefficients

The influence of interfering ions on the response behavior of an ion-selective electrode has usually been described in terms of selectivity coefficient. The values of the selectivity coefficients K^{pot}A,B of the proposed electrodes towards different species (B), were determined by the matched potential method.

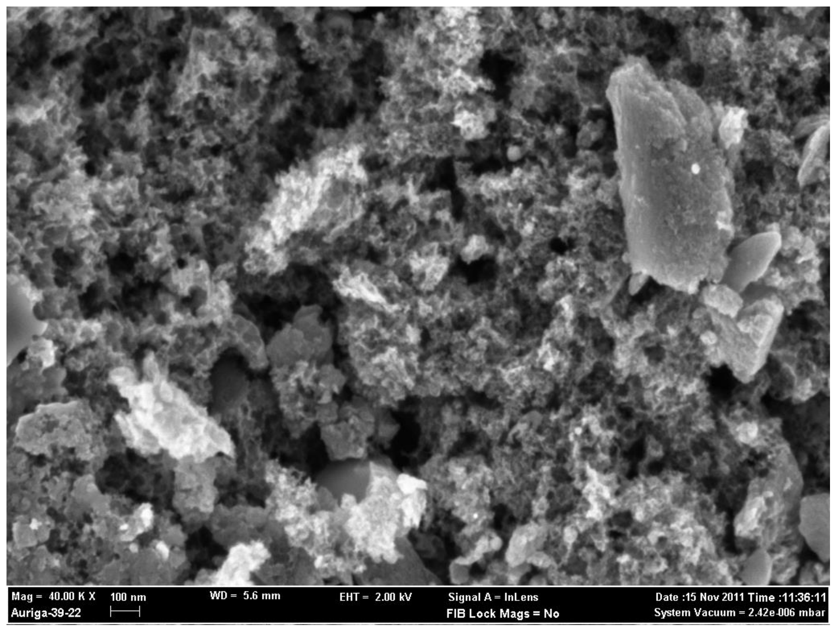
Selectivity coefficient values of various interfering ions with Cd(II) electrode using matched potential method (MPM).

Interferent ion, B	-log KpotCd^{2+},B	Interferent ion, B	-log KpotCd^{2+},B
Cu^{+2}	3.26	Na^{+}	4.71
Hg^{+2}	2.47	K^{+}	4.54
Ca^{+2}	3.95	Ni^{+2}	3.29
Mg^{+2}	4.15	Cr^{+3}	4.21
Zn^{+2}	3.83	Co^{+2}	3. 37
Al^{+3}	3.74	Ag^{+}	3.10
Fe^{+3}	2.98	Pb^{+2}	4.02

The table show that the electrodes exhibited better selectivity for Cd ion over a wide variety of other metal, this due to the stability of the complex between metal and TSILs

Results and Discussions

Scanning electron microscopy results



The typical morphological features of pure palm shell activated carbon (PSAC).

We can see the surface have porosity properties and smooth surfaces

RESULTS AND DISCUSSIONS

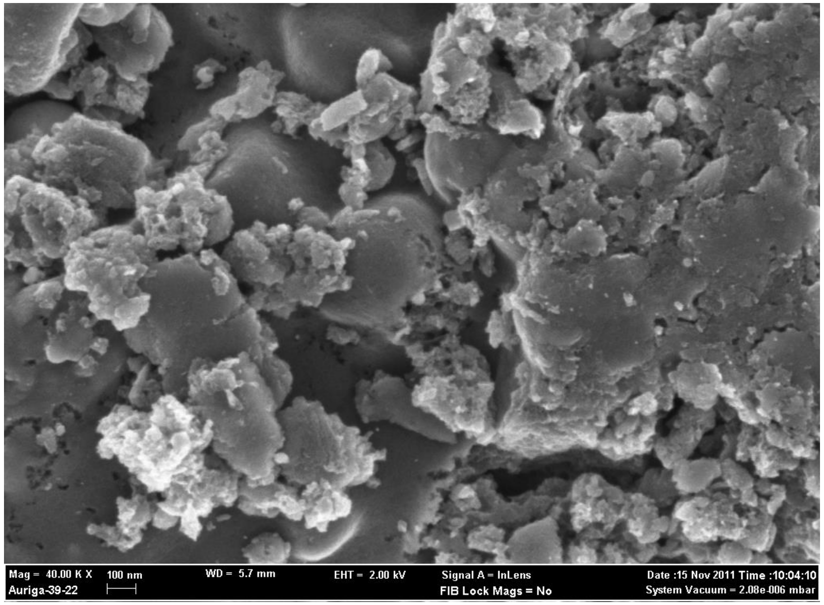


Fig. shows the SEM image of the paste film based on TOMAS.

We can see the active TSILs on the paste film are more uniformly distributed on the electrode surface

RESULTS AND DISCUSSIONS

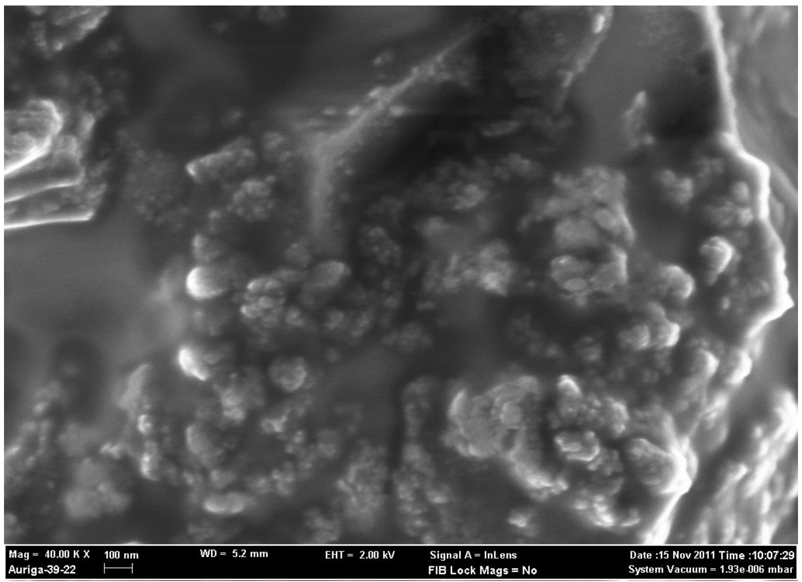


Fig. shows the SEM image of the electrode based on TOMAS after dipped in 1x10^{-4} M Cd^{+2} solutions.

We can see aggregates of white needles on the surface that mean complexes formed between TSILs and metal ions

RESULTS AND DISCUSSIONS

Analytical Applications

Sample[a]	Cd (II) (mgL^{-1})			
	Proposed electrode	ICP	RSD%	Recovery%
(1)	0.575	0.55	3.19	104.6
(2)	0.464	0.446	2.90	104.2
(3)	0.480	0.491	1.55	97.8
(4)	0.423	0.412	1.92	102.8

This table show analytical results of Cd in real water samples. We can see the results obtained by proposed electrodes are good agreement with those obtained by ICP

CONCLUSION

- The results presented here demonstrate the utility of TSILs as both plasticizer and ionophore in preparation of new ion selective electrodes for determination of heavy metal ions.

- Palm shell activated carbon modified with trioctylmethylammonium salicylate (TOMAS) was used for the potentiometric determination of cadmium ions in water samples.

CONCLUSION

Several important points have been highlighted in this work as follow:

- The new sensors for trace measurements of mercury and cadmium ions are suitable for in site monitoring with high detection limit.
- The modified electrodes show excellent potentiometric response, sensitivity, selectivity and stability.
- The electrode was successfully applied for determination of cadmium content in drinking water samples.


Thank You!