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GLASSY CARBON ELECTRODES MODIFIED WITH ORDERED MESOPOROUS SILICA FOR LEAD DETECTION





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INTRODUCTION

Ordered mesoporous silica (OMS) from the classes MCM-41 and SBA-15 [1] has attracted the scientists interest due to their large specific surface areas, ordered structure, inert framework, non-toxicity, and hydro/thermal stability, which allow them to be used as catalyst support, as well as adsorption, sensing or drug delivery systems.

EXPERIMENTAL



AIMS

The aim of the work was to prepare modified glassy carbon electrodes coated with a composite matrix consisting of four types of original functionalized OMS and an ion-exchange polymer (Nafion) in order to detect Pb (II) ions.

The role of NH₂- functional groups grafted on the silica surface on the electrochemical behavior of the modified electrodes was also investigated

SYNTHESIS OF MESOPOROUS MATERIALS

The functionalized MCM-41-NH₂ and SBA-15-NH₂ were prepared using APTES through a post-functionalization method.

PREPARATION OF GC MODIFIED ELECTRODE

The cleaned glassy carbon electrode (GCE) surface was covered with 5 µL OMS suspension (2mg/mL) in SDS 1% and 5 µL of a 2.5 % Nafion solution. The modified electrode was dried 2 h at room temperature.



MORPHO-STRUCTURAL MEASUREMENTS

Table 1. Characteristics of the various mesoporous silica samples.

Sample	^a S _{BET} (m²/g)	^b d _{Des,BJH} (Å)	^c V _{pDes,BJH} (cm³/g)	^d a (Å)
MCM-41	1061	27	1.41	45
MCM-41-NH ₂	894	21	0.98	46
SBA-15	880	67	1.25	117
SBA-15-NH ₂	373	55	0.65	119



Figure 1. Characterization of MCM-41, MCM-41-NH₂, SBA-15, and SBA-15-NH₂ ordered mesoporous silica samples. A) SAXS patterns and B) TEM image.

[a] Surface area calculated by the BET method, [b] Pore diameter from desorption branch calculated by BJH method, [c] Pore volume from desorption branch calculated at $p/p^{\circ}=0.99$ by BJH method, [d] Lattice parameter obtained by SAXS.

ELECTROANALYTICAL MEASUREMENTS





Table 2. Electroanalytical parameters of thevarious GCE modified with mesoporous silicaand Nafion.

Electrode	Sensitivity (A/M)	Detection limit (µM)	Linear domain (µM)	R²/N*
GC/Nafion	1.32 ± 0.063	1.68	0.5-6	0.9927/12
GC/SBA- 15/Nafion	3.36 ± 0.038	0.72	0.5-5	0.9993/10
GC/SBA-15- NH ₂ /Nafion	4.82 ± 0.063	0.24	0.5-6	0.9983/12
GC/MCM- 41/Nafion	2.84 ± 0.037	1.19	0.5-6	0.9976/12





SWASV recorded at GC/SBA-15-NH₂/Nafion electrode in 0.1 M acetate buffer (pH 4.4) in presence of Pb(II), and in their absence (A); Calibration curve and the corresponding linear fit (B).

CONCLUSIONS

© The analytical parameters of the prepared electrodes determined by square wave anodic stripping voltammetry were very promising, recommending the use of the modified electrode for real samples analysis.

© Ordered mesoporous silica immobilized on GCE improve the electroanalytical parameters of GCE/Nafion electrodes due to their large surface area and to their adsorption ability.



Bibliography

[1] El-Salamony R. A., Gobara H. M., Younis S. A., Journal of Water Process Engineering, 2017, 18, 102-112.

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