



Adsorption of heavy metal ions and epoxidation catalysis using a new polyhedral oligomeric silsesquioxane

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HIGHLIGHTS

- ▶ The synthesis of a new silsesquioxane chemically modified (T₈-Pr-ATD) was described.
- ▶ The new adsorbent showed to be an effective sorbent for metal ions in solutions.
- ▶ The new adsorbent presented a high large adsorption capacity.
- ▶ The adsorption process equilibrium condition is reached at time lower than 10 min.
- ▶ The new material T₈-Pr-ATD-Mo and Si-Pr-ATD-Mo exhibits great catalytic activity.

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ABSTRACT

The objective of this research was the preparation of a silsesquioxane functionalized with eight chloropropyl chains (T₈-PrCl) and of a new derivative functionalized with a pendant linear chain (2-amino-1,3,4-thiadiazole – ATD; T₈-Pr-ATD). The two nanostructured materials were characterized by ¹³C and ²⁹Si NMR, FTIR and elemental analysis. The new nanostructured material, octakis[3-(2-amino-1,3,4-thiadiazole)propyl] octasilsesquioxane (T₈-Pr-ATD), was tested as a ligand for transition-metal ions with a special attention to adsorption isotherms. The adsorption was performed using a batchwise process and the organofunctionalized surface showed the ability to adsorb the metal ions Cu (II), Co (II), and Ni (II) from water and ethanol. The adsorption isotherms were fitted by Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D-R) model. The kinetics of adsorption of metals were performed using three models such as pseudo-first order, pseudo-second order and Elovich. The Langmuir and Elovich models were the most appropriate to describe the adsorption and kinetic data, respectively. Furthermore, the T₈-Pr-ATD was successfully applied to the analysis of environmental samples (river and sea water). Subsequently, a new nanomaterial was prepared by functionalization of the T₈-Pr-ATD with a Mo (II) organometallic complex (T₈-Pr-ATD-Mo). Only a few works in the literature have reported this type of substitution, and none dealt with ATD and Mo (II) complexes. The new Mo-silsesquioxane organometallic nanomaterial was tested as precursor in the epoxidation of cyclooctene and styrene.

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1. Introduction

Novel compounds based on nanocage silsesquioxano cores have been the subject of much attention in recent years [1–4]. Through their eight Si vertices, these nanoplatfoms may be covalently linked to a plethora of organic or organometallic groups which

may serve as sorbents of metal ions from aqueous and non-aqueous solutions [5–9].

Polyhedral oligomeric silsesquioxanes (POSSs), (RSiO_{1.5})_n with *n* = 6, 8, 10 etc., are nanoplatfoms with one to eight reactive or nonreactive organofunctional groups (R) anchored to the eight possible vertices of the cubic silsesquioxane. Octahedral POSS (*n* = 8) are the most important members of this family due to its extensive application in the field of research in polymers [10–14].

In the POSS, the cubic silica core is rigid “hard particles” with diameter 0.53 nm and a spherical radius of 1–3 nm including peripheral organic units [1,2,10–12]. Reviews on this field were published by Baney et al. [12], Calzaferri [13], and Li et al. [14].

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