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## LAYERD INORGANIC-ORGANIC TALC LIKE HYBRID WITH HETEROCYCLIC CHELATING LIGAND INSIDE THE LAMELLAR SPACES – SYNTHESIS, CHARACTERIZATION AND CATION REMOVAL

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**Abstract-** Layered inorganic-organic hybrid based on talc like magnesium phyllosilicate was synthesized with the heterocyclic chelating ligand inside the lamellar spaces. The talc like magnesium phyllosilicate with organic moiety was characterized by IR, solid state NMR for both <sup>13</sup>C and <sup>29</sup>Si nuclei and XRD. The synthesized hybrid material was used for Pb<sup>2+</sup> cation removal from aqueous solution using adsorption isotherms.

Self-assembled inorganic-organic layered hybrids materials are usually synthesized from one step sol-gel route. An interesting feature associated with these hybrid materials is the presence of organophilic functionalities distributed inside the interlayer cavities [1]. Such kind of materials are useful in different fields e.g. as metal ion extractions, chromatographic determinations, catalysis etc. The synthesis of organofunctionalized layered inorganic-organic hybrid was based on magnesium-phyllosilicate model, in which pendant chains have nitrogen and sulfur atoms, that act as chelating agents for cation complexing from aqueous solutions. A new silylating agent was initially prepared by the reaction of 3-aminopropyltrimethoxysilane and 2-thiophenecarboxaldehyde in presence of triethylamine in methanol. This new silylating agent reacted with Mg(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O in methanol by sol-gel process, that lead to the formation of proposed talc like layered magnesium-phyllosilicate hybrid, and was characterized by, IR, solid state NMR for <sup>13</sup>C and <sup>29</sup>Si nuclei and XRD (Fig. 1).

The IR spectra showed typical stretching from both inorganic and organic moieties at 1034 and 1131 cm<sup>-1</sup> due to Si-C, Si-O-Si and 2930 cm<sup>-1</sup> for CH stretching of organic moiety. A band at 518 cm<sup>-1</sup> was attributed to inorganic part of Mg-O. Solid-state <sup>13</sup>C NMR spectrum showed all the expected peaks from the propyl carbon chain with a signal at 144 and 125 ppm, which is attributed to the carbon atoms of C=C bond of thiophene ring. For <sup>29</sup>Si, the signal were assigned to T<sup>3</sup> [RSi(OSi)<sub>3</sub>], T<sup>2</sup> [(RSi(OSi)<sub>2</sub>(OH)] and T<sup>1</sup> [(RSi(OSi)(OH)<sub>2</sub>] at –68 ppm, -56 ppm and –48 ppm, respectively, confirming the covalent bonding of the organic moiety to the inorganic backbone. The XRD shows a peak in the 001 plane at  $2\theta = 5.17^{0}$ , confirming the phyllosilicate like structure. The synthesized hybrid material was tested for Pb<sup>2+</sup> removal from aqueous solution (Fig. 1)

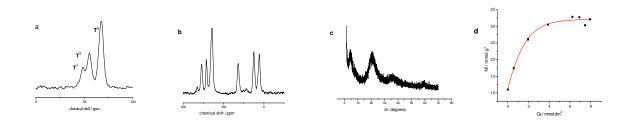


Figure 1: Solid state NMR for <sup>29</sup>Si nuclei (a), solid state NMR for <sup>13</sup>C nuclei (b), XRD of hybrid material (c), adsorption isotherm for  $Pb^{2+}$  cation removal from aqueous solution (d).

[1] M. G. Fonseca and C. Airoldi, J. Mater. Chem., 10 (2000) 1457-1463.