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PREPARATION OF NEW SILICA HYBRID MATERIALS BY SOL-GEL PROCESS FOR HUMIC ACIDS IMMOBILIZATION

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Introduction

- Hybrid organic-inorganic materials derived through sol-gel process combine the properties of inorganic and organic compounds in one material.
- By tailoring the surface properties of inorganic materials, the desired functional groups can be generated on this silica surface having different roles (coupling agent, spacer).
- The attachment of humic acids to such silica hybrid particles leads to the formation of materials with interesting remediation properties which can use in environmental studies for eliminating metal ions or aromatic hydrocarbons from the contaminated water and for reducing chemical oxygen demand in wastewater etc.

Thesis objectives

Main objective:

- preparation and characterization of innovative silica hybrid materials, used as supports for humic acids immobilization.

Sub-objectives:

- obtaining silica particles by base-catalyzed sol-gel process;
- generating amino or epoxy functions on the silica particle surfaces, spaced by alkyl groups, which ensure a better access of humic acids to the connecting agents
- grafting the humic acids to the hybrid silica surface
- assessment of physico-chemical properties of the synthesized materials depending on the operating parameters.

Achievements

- ❖ Preparation of amino (APTES) and glycidyloxy (GPTMS) functionalized silica supports which were prepared by a two steps sol-gel process at room temperature, in alkaline medium:
 - ▣ Step 1: silica particles were synthesized from TEOS in ethanol, according to Stöber method
 - ▣ Step 2: after 2h of stirring, APTES or GPTMS (APTES/GPTMS:TEOS = 1:20 molar ratio) was added drop wise for 2h to the reaction mixture, then was further stirred for another 2h

- ❖ Characterization of silica hybrid materials with different methods:
 - ❑ Elemental Analysis (CHN)
 - ❑ Solid State ^{29}Si and ^{13}C CP/MAS Nuclear Magnetic Resonance (CP/MAS-NMR)
 - ❑ Fourier Transform Infrared Spectroscopy (FTIR)
 - ❑ Dynamic Light Scattering (DLS)
 - ❑ Laser Doppler Velocimetry (LDV)
 - ❑ Thermogravimetric Analysis (TGA)

- ❖ Preparation of amino and glycidyl silica supports functionalized with humic acids:
 - ❑ Preliminary coupling test using para hydroxybenzoic acid
 - ❑ Isolation of humic acids from Comandau peat
 - ❑ Testing the functionalization efficiency of the new prepared silica supports using two type of humic acids: one is commercially available (from Aldrich - Humic acid technical) and the other was extracted from the peat
 - ❑ Characterization of the obtained materials by FT-IR

General conclusions

- The amount of organic component from the final hybrid supports, evaluated by CHN and TGA analyses, depends on the co-precursors type, proving their successful grafting on the silica particles surface.
- By inserting spacing functions on the surface of the silica particles, zeta potential changed from negative to positive values, while the particle size measurements showed slight differences.
- FTIR spectra confirmed the integration of co-precursors into the final silica hybrid network.
- Solid state ^{29}Si and ^{13}C CP-MAS NMR spectra revealed the efficient functionalization of silica particles with amino or epoxy and with methyl groups.
- The synthesized hybrid particles can be successfully used for immobilization of the humic acids of different origin.

Conferences

□ Oral communication

- **Sándor Mónika**, Cristina Lavinia Nistor, Violeta Purcar, Szalontai Gábor, Rusandica Stoica, Szőke Anna-Mária, Dan Donescu, Fazakas József, *Synthesis of epoxysilane functionalized silica supports for humic acids immobilization*, Chemistry priorities for a sustainable development - PRIOCHEM - 10th Edition, 30-31 October 2014, Bucharest, Romania
- **Sándor Mónika**, Fazakas József, Bartalis Ildikó, Szőke Anna Mária, *Chemical characterization of peat from Comandău and its alkaline extracts*, International Conference NATURA – ECON 4, Environmental Dynamics under the Impact of Economic Trends – Realities and Perspectives; 7th March 2014, Sfântu Gheorghe, Romania

□ Poster

- **Sándor Mónika**, Cristina Lavinia Nistor, Violeta Purcar, József Fazakas, Cristian Petcu, Cătălin-Ilie Spătaru, Marius Ghiurea, Raluca Ianchiș, Raluca Șomoghi, Dan Donescu *Preparation of aminopropyl hybrid silicas by the sol-gel method for immobilizing humic acids*, The XXXIII-rd Romanian Chemistry Conference, 01-03 October 2014, Călimănești-Căciulata, Romania

Future publications

- writing an article about the preparation of amino hybrid silica particles functionalized with humic acids (partially completed manuscript)
- writing an article about the preparation of glycidyloxy hybrid silica particles functionalized with humic acids (manuscript under preparation)

Thank you for your attention!

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