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SYNTHESIS AND CHARACTERIZATION OF MODIFIED SILICA HYBRID NANOCOMPOSITES

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Introduction

Environmental problems lead to invention innovative nanocomposites, which can adsorb, degrade and prevent further growth of pollutants. The requirements for protective hybrids are connected with their compatible, high reactive structures, long-term stability and purification effect. Silica-chitosan nanocomposites are proven as high reactive adsorbents of different pollutan. Low mechanical stability in different media leads to modification with synthetic polymer, which can improve the resistance and extend pollutant sorption activity.

Main goal

The goals of the study were sol-gel synthesis and structural characterization of modified silica hybrid nanocomposites for environment purposes. Tethaethylothosilicate (Si) was used as silica matrix former. Natural (chitosan, CS) and synthetic (hydroxyethylmethacrylate, HEMA) polymers were used as modifiers of silica network, which can enhance sorption activity and plasticity of final nanocomposite. Quantity of CS/HEMA vary between 5 and 20%. Influence of nature and quantity of organic polymers on silica network, as well as final structure characteristics were investigated.

Structural characterization

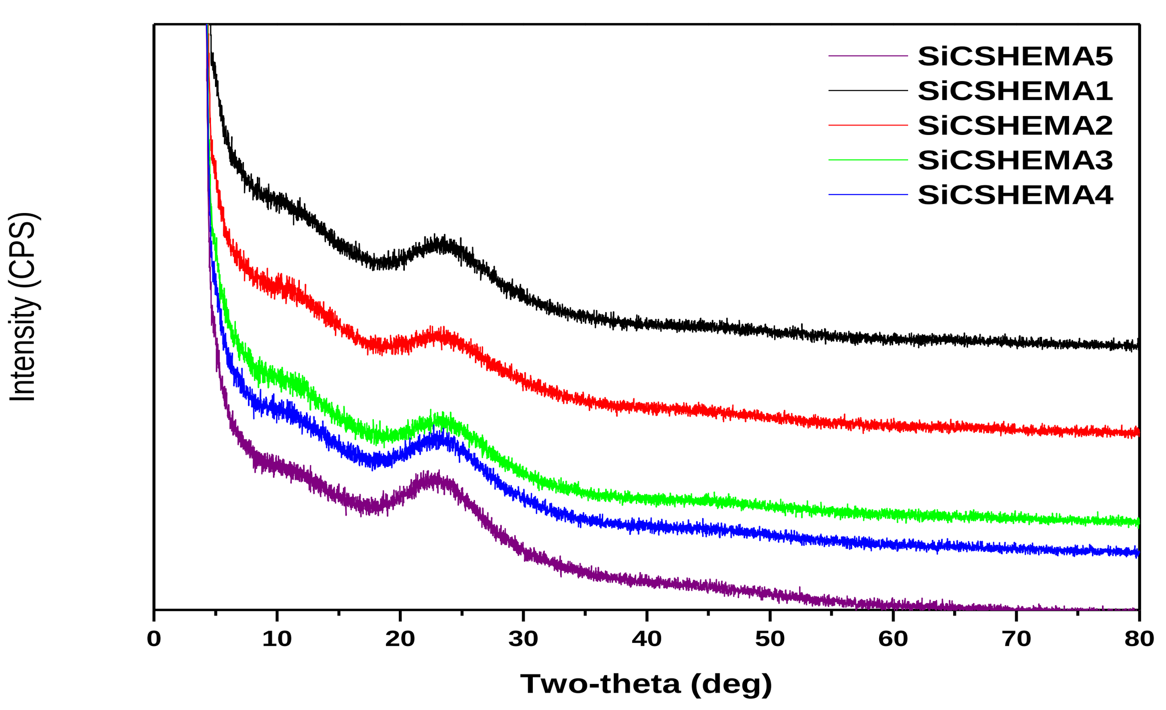

Fig.1 XRD patterns of synthesized hybrid nanocomposites

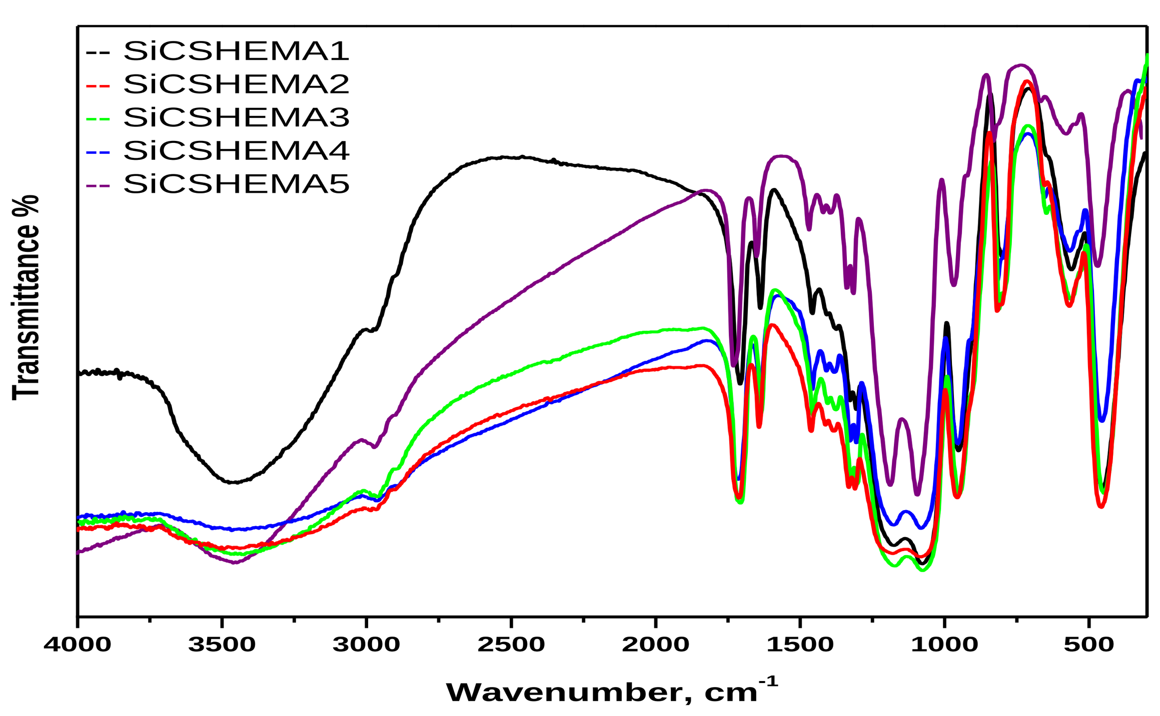

Fig. 2 FTIR spectra of synthesized modified silica nanocomposites

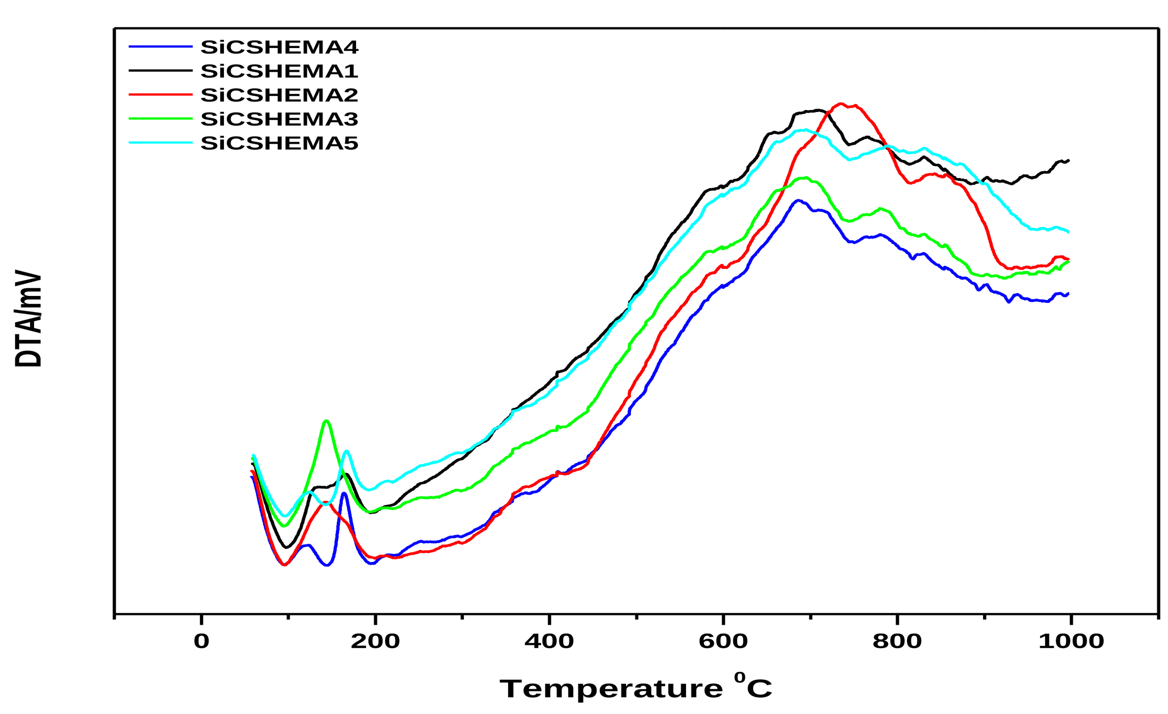

Fig. 3 DTA curves of synthesized hybrid nanocomposites

- From XRD patterns are observed two intensive peaks around 23 and 12 2θ. The halo around 23 2θ showed, that synthesized hybrid nanocomposites are in amorphous state, as a result of low temperature sol-gel synthesis. Other intensive peak around 12 2θ is connected with participation of chitosan in composite structure. XRD investigations of chitosan showed, that the polymer can exist in three forms–amorphous (12 2θ), hydrated crystalline (14 2θ) and anhydrous crystalline (23 2θ). Presence of peak in 12 2θ showed, that in synthesized hybrid materials, the used chitosan is in amorphous state. In comparison on XRD results of pure silica material and synthesized hybrids there is no difference of intensity of halo (23 2θ). This is evidence, that in obtained hybrid exist only amorphous chitosan.
- FTIR spectra showed the formation of Si-O-Si network and existence of Si-OH bounds due to hydrolysis and polycondensation reactions of silicate precursor. The structure of natural polymer (chitosan) consist carbon structural units with free reactive –OH and amide/amino groups. Formation of chitosan chain between separate units is ascribed with existence of peak at 1100 cm^{-1}. Presence of NH_2 and $NHCOCH_3$ groups are associated with peaks in the diapason 1400-1500 cm^{-1}. The structure of added synthetic polymer (HEMA) is characterized with C=C and C=O bounds. The existence of these two bounds are ascribed with peaks at 1640 and 1700 cm-1. The peak of C=C bound is overlapping with H-OH bounds.
- DTA analysis established structural transformation of obtained structures during thermal treatment. DTA curves established intensive endothermic reactions (50-200 ^{0}C), which are associated with partial degradation of organic polymers, as well as removal of residual water. In this temperature diapason removal of water molecules can be connected with poly condensation of Si-OH groups. After this with increasing the temperature further degradation of organic units and transformations of SiO_4 tetrahedral structures is observed.
- SEM micrographs of synthesized hybrid nanocomposites, with participation of 5% and 20% organic components are shown on fig. 4. The results of SiCSHEMA1 mixture showed, that synthesized hybrid formed homogeneous, smooth non-crack structure. With increasing organic quantity formation of nanoparticles is observed (SiCSHEMA5). Formed nanoparticles with size between 30 and 50 nm are evenly distributed on the surface and in the volume. EDS analysis showed tendency of increasing carbon contain with increasing organic quantity.
- AFM topographic pictures confirm the results for surface structures from SEM analysis. Evenly distribution of organic polymers is clearly visible from AFM results for SiCSHEMA5.

Surface Volume

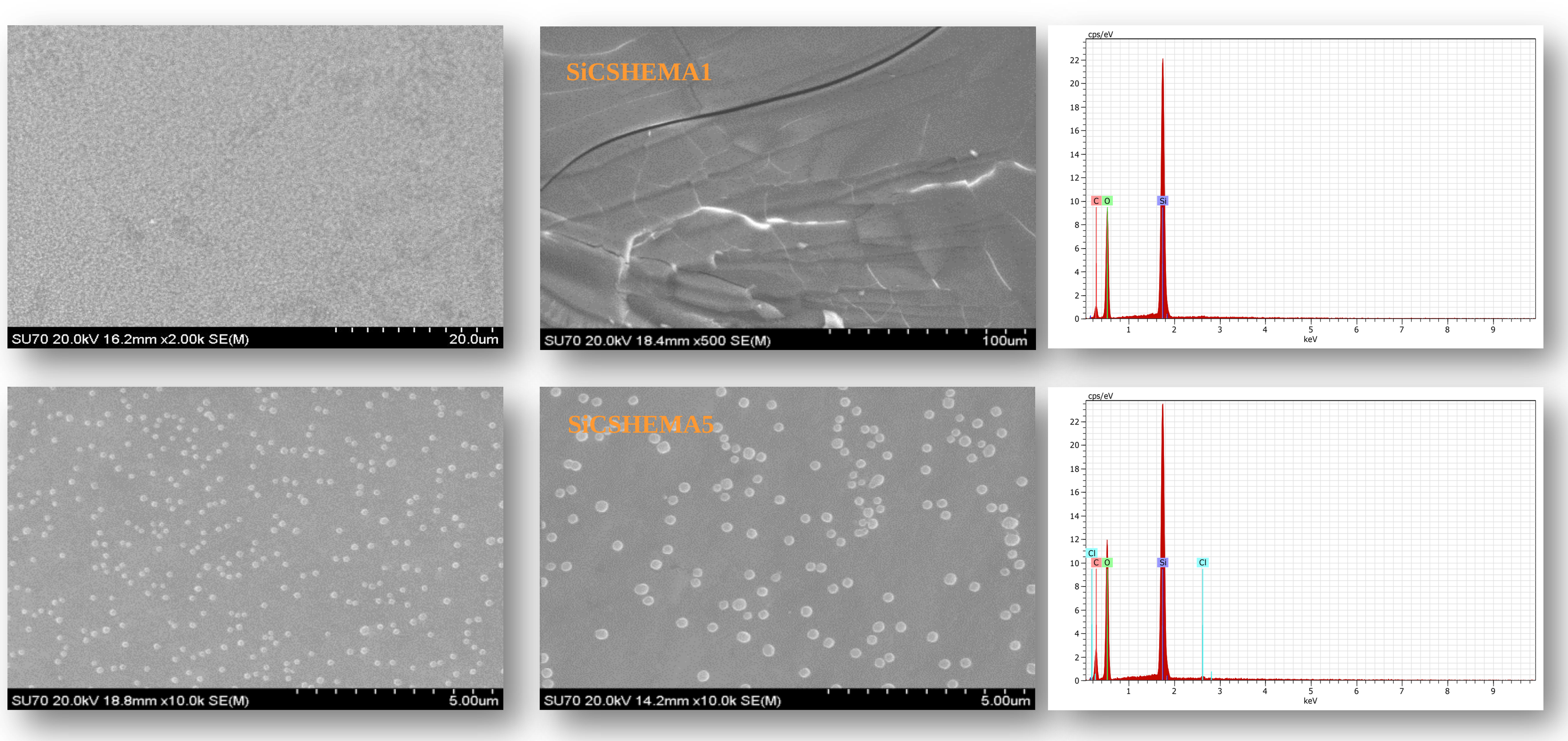

Fig. 4 SEM micrographs and EDS analysis of innovative hybrid nanocomposites

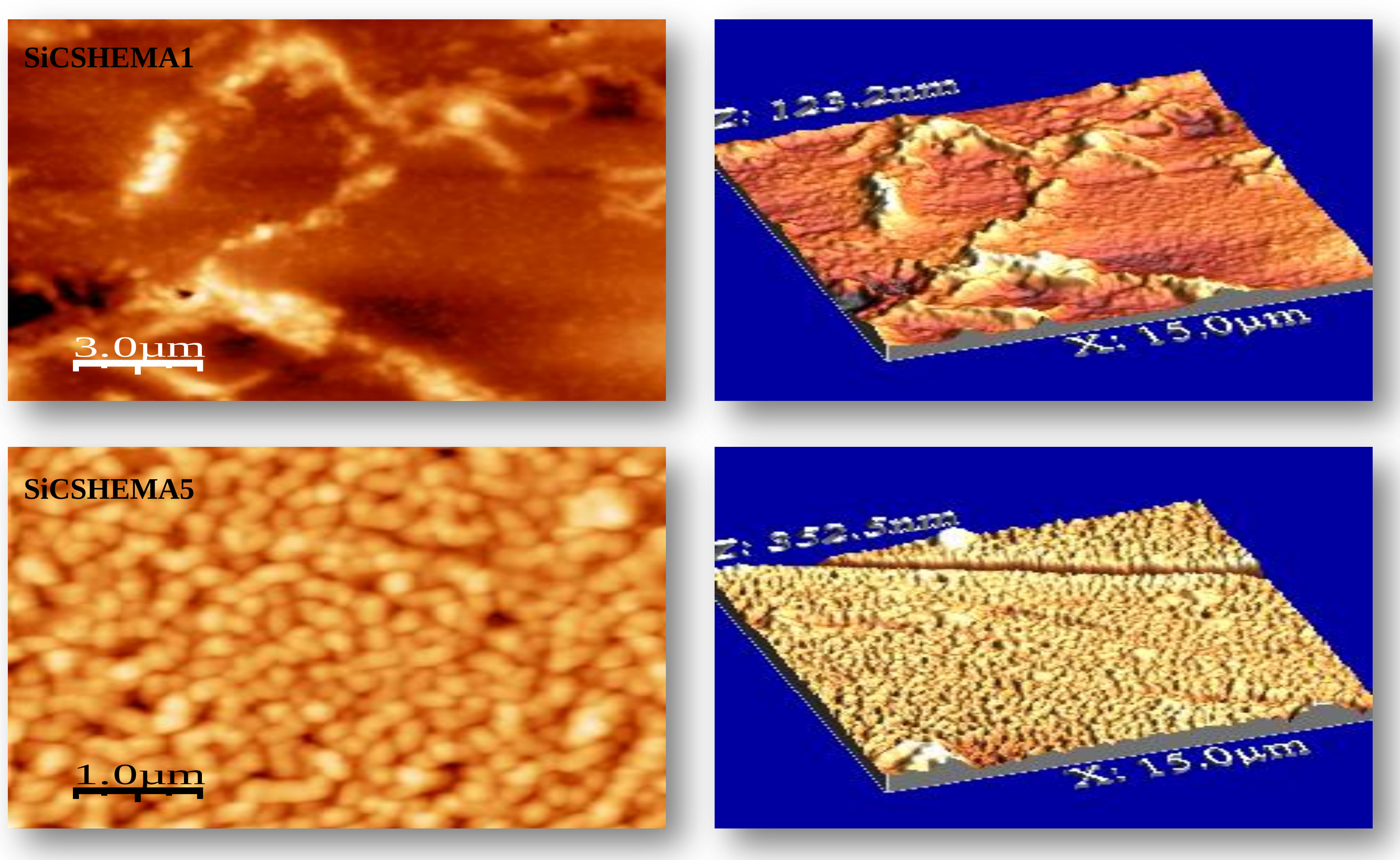

Fig. 5 AFM topographies of synthesized hybrid nanocomposites

Potential application

The innovative sol-gel nanocomposites, based on silica materials and modified with chitosan and hydroxyethylmethacrylate exhibit unique structure and properties. Formation of stable, homogeneous structure, as well as presence of high reactive groups in it were established from the used analysis. Compatibility of used components lead to formation of unique nanocomposite with improve properties. Formation of silica network allows introduction and evenly distribution of organic additives and formation of stable hybrid. Compatibility between two polymers (CS and HEMA) and with silica matrix, lead to formation of potential sorbents with high reactive contact surface. Specific structure of obtained hybrid nanocomposites, abundance of used components and easy technique of preparation ensure their potential application as protective materials for environmental safety.

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