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CONTENT

<i>Volume I</i>	
<i>Organizer</i>	IV
<i>Comittes</i>	V
<i>Sponsors</i>	VI
<i>Plenary Lecture</i>	1
<i>Chemical Thermodynamics</i>	57
<i>Spectroscopy, Molecular Structure, Physical Chemistry of Plasma</i>	71
<i>Kinetics, Catalysis</i>	157
<i>Nonlinear Dynamics, Oscillatory Reactions, Chaos</i>	253
<i>Electrochemistry</i>	361
<i>Biophysical Chemistry, EPR investigations of biosystems,</i>	419
<i>Photochemistry, Radiation Chemistry</i>	



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NANOSTRUCTURED ORGANIC-INORGANIC HYBRID SMECTITE BASED COMPOSITES

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ABSTRACT

This paper represents a survey of the investigation conducted in the Center for Catalysis and Chemical Engineering of the Institute of Chemistry, Technology and Metallurgy, Belgrade, during last decade regarding nanostructured organic-inorganic hybrid smectite based composites and their application as adsorbents and modifiers of glassy carbon electrode.

INTRODUCTION

Nanostructured organic-inorganic hybrid smectite based composites (HC) are among the most promising contemporary materials for various applications. HC obtained by interaction of quaternary alkyl ammonium cations (QAAC), organic anions and biopolymer chitosan were investigated as adsorbents of pertechnetate, anionic textile dyes, of phenol and its nitro derivatives and nicotine. Comparison of bare glassy carbon electrode (GCE) and GCE modified with different HC was performed [1-24].

EXPERIMENTAL

The first step in synthesis of nanostructured organic-inorganic hybrid smectite based composites (HC) is preparation of Na⁺-enriched homoinic smectite (Na-S) as a precursor material. The Na-S was obtained by replacing of naturally present exchangeable cations in the interlamellar space of smectite with solely Na⁺ [2, 25]. The HC were synthesized by intercalation of selected (bio) organic cations into smectite structure. Intercalation was performed with quaternary alkylammonium cations (QAAC) with different smectite/QAAC ratios and different QAAC. The intercalation with biopolymers i.e. chitosan was also performed in order to test green alternative to QAAC.

The QAAC used for synthesis of HC were: hexadecyltrimethylammonium (HDTMA), dodecyltrimethylammonium (DDTMA), tetramethylammonium (TMA) and benzyltrimethylammonium (BTMA) cations. The QAAC/smectite ratios were defined as multiples of cation exchange capacities (CEC) of smectite in the range from 0.2 to 2.0. The samples were denoted according to QAAC and multiples of CEC i.e. 1.0 TMA-S. The HC were obtained by intercalation of QAAC into the interlamellar space of smectite. The arrangement of the intercalated QAAC depended on the length of intercalated alkyl cation chains and the amount of the intercalated cations forming monomolecular layer, lateral bimolecular layer, pseudo-trimolecular layer and paraffin type of layer [12, 26, 27]. The HC with organic anion dodecyl sulfate was obtained by interaction with acidic smectite surface, developed by acid modification and denoted as DS-S_A [22].

The bio-polymer used for synthesis of HC was high molar mass chitosan (deacetylated chitin or poly(D-glucoseamine), av. $M_w = 342500 \text{ g mol}^{-1}$). Chitosan/smectite HC had chitosan intercalation in mono- and bi-layer arrangement. Synthesis was performed using chitosan solution of pH=4.9 that enabled presence of ammonium groups in chitosan structure, and preserved the smectite structure [20, 28, 29]. The samples were denoted according to chitosan layer formation as Cml-S and Cbl-S for monolayer and bi-layer formation in smectite structure, respectively.

Chemical composition of starting smectites was obtained by ICP-OES, while composition of organic phase (C, H and N) in HC samples was determined by elemental analysis [12, 20]. The intercalation of organics into smectite structure was monitored by X-ray diffraction (XRD). The FTIR was used to confirm the electrostatic interaction of positively charged organic species with negatively charged sites of smectite [12, 20]. The point of zero charge was used to estimate the surface acidity of the synthesized HC [5, 12, 21]. Textural properties were determined by low temperature N₂ physisorption in order to correlate porous structure with the adsorptive and electrochemical properties [2, 3, 6, 7, 10, 16]. The morphology of HC was analyzed by SEM [5, 17]. The process of adsorption was monitored using UV-Vis spectrophotometry and inductively coupled plasma (ICP). The electrochemical behavior of GCE modified with HC was analyzed using cyclic voltammetry, multisweep cyclic voltammetry and square wave voltammetry.

RESULTS AND DISCUSSION

Adsorption study

The review of comprehensive adsorption investigations is summarized in Table 1. The amount of adsorbed dye (AO10, AY99 and RB5) at equilibrium time (q_e) increased with: (i) increase of QAAC/CEC ratio, and (ii) number of C-atoms present in applied QAAC. Generally, it could be assumed that adsorption of textile azo dyes strongly depends on arrangement of intercalated organic cation in smectite interlamellar space. The organic cations were incorporated in smectite structure in different layered structures. Monolayer, bi-layer, pseudo-trilayer and paraffin type of arrangements were found in 0.2 HDTMA-S, 0.5-HDTMA-S, 1.0-HDTMA-S and 2.0 HDTMA-S, respectively. The adsorption mechanism of acid azo dyes on samples with amount of QAAC \leq CEC was mainly through different organic interactions. Adsorption of azo dyes on hybrid nanocomposites with QAAC $>$ CEC is governed by additional electrostatic interaction i.e. via attraction of $-\text{SO}_3^-$ groups of dyes with ammonium groups [12, 29]. The amount of adsorbed dyes at equilibrium time was the highest for AY99 and the lowest for AO10. It was found [12] that developed surface area was less significant for the adsorptive properties of QAAC-smectite nanocomposites toward organic contaminants than arrangement of organic phase in composite. The highest adsorption efficiency toward investigated dyes showed 2.0 HDTMA-S, although this sample possessed the least pronounced textural properties [12].

Jović-Jovičić et al. [2, 3, 5, 12] showed that there is no competition between acid dyes (AO10, AY99 and RB5) and Pb^{2+} in simultaneous adsorption from bi-component solutions. Moreover, the adsorptions of dye molecule or Pb^{2+} become the new adsorption sites, enhancing adsorption from their bi-component solutions [2, 5, 12].

The 0.5 HDTMA-S and 1.0 HDTMA-S were tested as multifunctional adsorbent for three-component solution of dyes (AO10, AY99 and RB5), three-component solution of toxic metallic cation (Pb^{2+} , Ni^{2+} and Cd^{2+}) as well as their hexa-component solution. Both samples showed the highest adsorption affinity toward AY99 among dyes and Ni^{2+} among cations. The 1.0 HDTMA-S was found to be more efficient multifunctional adsorbent of hexa-component solution, showing synergistic effect in simultaneous adsorption of textile azo dyes and metal cations [14].

Among QAAC-smectite nanocomposites the 1.0 TMA-S was found to be the most efficient toward nicotine adsorption. In this sample intercalated TMA^+ cations formed pillar-like arrangement within smectite interlamellar space that led to developed textural structure and enhanced specific surface

Table 1. The review of adsorption studies on smectite nanocomposites

Adsorbate	Adsorbent	q_e [mg g ⁻¹]	Ref
AO10	0.2 HDTMA-S	2.2	2
	0.5 HDTMA-S	5.4	2
	1.0 HDTMA-S	22.8	2
	2.0 HDTMA-S	100.0	2
	2.0 DDTMA-S	12.3	2
	2.0 TMA-S	2.5	2
	Cml-S	21.9	21
AY99	0.2 HDTMA-S	18.3	3
	0.5 HDTMA-S	103.5	3
	1.0 HDTMA-S	142.3	3
	2.0 HDTMA-S	253.7	3,4
	2.0 DDTMA-S	88.9	3
	2.0 TMA-S	21.4	3
	Cml-S	121.8	21
RB5	0.2 HDTMA-S	2.7	12
	0.5 HDTMA-S	11.9	12
	1.0 HDTMA-S	54.7	12
	2.0 HDTMA-S	193.6	12
	2.0 DDTMA-S	39.5	12
	2.0 TMA-S	1.0	12
	Cdl-S	166.7	15
Cml-S	87.1	21	
Nicotine	1.0 TMA-S	58.4	23
Pb ²⁺	0.2 HDTMA-S	51.3	2
	0.5 HDTMA-S	45.0	2
	1.0 HDTMA-S	43.4	2
	2.0 HDTMA-S	31.9	2
	2.0 DDTMA-S	60.4	12
	2.0 TMA-S	63.3	12
AO10, Pb ²⁺	0.2 HDTMA-S	1.8, 50.1	2
	0.5 HDTMA-S	10.9; 44.2	2
	1.0 HDTMA-S	36.2; 36.8	2
	2.0 HDTMA-S	99.3; 29.5	2
AY99, Pb ²⁺	0.2 HDTMA-S	30.7; 50.6	3
	0.5 HDTMA-S	90.1; 40.0	3
	1.0 HDTMA-S	219.2; 21.7	3
	2.0 HDTMA-S	243.6; 22.7	3
	2.0 DDTMA-S	195.7; 47.9	3
	2.0 TMA-S	30.7; 67.0	3
RB5, Pb ²⁺	0.2 HDTMA-S	8.0; 63.0	12
	0.5 HDTMA-S	22.0; 40.3	12
	1.0 HDTMA-S	66.4; 21.1	12
	2.0 HDTMA-S	168.3; 10.5	12
	2.0 DDTMA-S	55.5; 42.1	12
	2.0 TMA-S	3.7; 63.8	12
AO10, AY99, RB5	0.5 HDTMA-S	8.3; 68.1; 3.9	14
	1.0 HDTMA-S	19.1; 138.3; 25.0	14
Ni ²⁺ , Cd ²⁺ , Pb ²⁺	0.5 HDTMA-S	41.1; 27.0; 56.0	14
	1.0 HDTMA-S	18.2; 18.0; 39.4	14
AO10, AY99, RB5 Ni ²⁺ , Cd ²⁺ , Pb ²⁺	1.0 HDTMA-S	19.0; 151.2; 57.2 7.6; 7.9; 31.1	14
phenol	2.0 HDTMA-S	172.0	19
2-nitrophenol	2.0 HDTMA-S	500.4	19
3-nitrophenol	2.0 HDTMA-S	476.8	19
4-nitrophenol	2.0 HDTMA-S	597.7	19
⁹⁹ Tc(VII)	1.0, 1.5 and 2.0 HDTMA-S	523; 1295 and 1295 MBq g ⁻¹	13

area that favored nicotine adsorption [23].

The Cml-S with monolayer arrangement of chitosan showed adsorption affinity toward investigated dyes in the following order: AY99 > RB5 > AO10. It was found that adsorption was most efficient in acidic medium, probably due to electrostatic attraction between protonated active sites of Cml-S with anionic groups of dyes [21]. The adsorption of RB5 was also studied on Cdl-S, the bionanocomposite with intercalated bilayered arrangement of chitosan chains. The additional cationic sites ($-\text{NH}_3^+$) present in bilayer structure, could be the reason for almost two times higher amount of adsorbed RB5 on Cdl-S than on Cml-S [15, 21].

The removal of radioactive $^{99}\text{TcO}_4^-$ from aqueous solution at room temperature was investigated on HDTMA-S adsorbents with different HDTMA⁺ cation loading. The samples with HDTMA/S value > 1 were able to adsorb significant amount of $^{99}\text{TcO}_4^-$ [13].

The adsorption of phenol and its nitro derivatives was investigated on 2.0 HDTMA-S. Among investigated phenols the 4-nitrophenol (NP) had the highest adsorption capacity ($q_e=597.7 \text{ mg g}^{-1}$) [19].

For all investigated adsorption systems, the adsorption kinetics followed pseudo-second order kinetics model, while adsorption isotherms were best described by Langmuire isotherm [1, 13, 19, 21].

Electrocatalysis

The electrochemical performance of glassy carbon electrode (GCE) modified with different HC was tested.

The phenol electrooxidation showed similar behavior on 0.2 HDTMA-S, 0.2 DDTMA-S and 0.2 TMA-S, with the highest electrochemical response with 0.2 TMA-S. The major difference was found between Tafel slopes, indicating different types of adsorption [6].

Phenol determination was performed on series of HDTMA-S based electrodes in acidic and alkaline solutions. It was proved that HDTMA/S ratio plays the key role in phenol oxidation [7, 9]. Sensitivity of phenol oxidation was improved with increasing of HDTMA⁺ loading, while deactivation was observed only in electrode with 0.2 HDTMA-S [7]. It was also confirmed that presence of HDTMA⁺ in electrode materials allowed the analyte preconcentration and prevent phenol polymerizations on electrode surface [9].

The electrooxidation of 4-nitrophenol was investigated using electrodes based on three series of HC: (i) TMA-S composites with different TMA/S ratios in range from 0.2 – 2.0 [10]; (ii) series of different QAAC (HDTMA⁺, DDTMA⁺, TMA⁺) with constant QAAC/S ratio of 0.2 [10] and (iii) series with aromatic BTMA⁺ cation with 0.2-2.0 BTMA⁺/S ratios [8, 16].

The results showed that current density of anodic peak at 1.2 V assigned to 4-NP oxidation depended on type and amount of QAAC loading. The electrode contained 0.2 TMA-S showed the highest signal for 4-NP oxidation, with detection limit of $1 \cdot 10^{-6} \text{ mol dm}^{-3}$ [10].

The electrooxidation of 4-NP using electrodes based on BTMA-S showed that increasing of BTMA⁺ loading lead to the increase of electrochemical activity and stability [8, 16]. The linear dependence of current density of 4-NP concentration was observed for electrode with 2.0 BTMA-S [8].

The Cbl-S was used for glassy carbon electrode modification and used for detection of phenol and its nitro derivatives from acidic and alkaline solutions [20]. In acidic solution the investigated electrode proved to be applicable for the simultaneous detection of phenol, 2-NP and 4-NP, although with high rate of passivation. On the other hand, in the alkaline solution the rate of passivation was very low, but all three analytes could not be detected simultaneously [20].

GCE modified with DS-S_A was used for electrooxidation of nicotine [25] and testing of electrochemical behavior of immobilized hemoglobin in alkaline solution [22]. The intensity of the nicotine oxidation peak was lower on the GCE-DS-S_A than on the bare GCE, and the onset and peak potentials were significantly shifted towards less positive values. The nicotine peak intensity increased gradually with the increase of the accumulation time improving the sensitivity of GCE-DS-S_A [25].

Investigation of immobilized hemoglobin (Hgb) on GCE-DS-S_A revealed that presence of DS resulted in partial oxidation/denaturation of Hgb as well as formation of hemichrome. In alkaline medium, two reduction peaks were observed ascribed to reduction of Fe(III)/Fe(II) heme pair and highly reduced HgbFe(I). The peaks current were enhanced in presence of H₂O₂ in alkaline solution, and linear response to concentration was observed [22].

CONCLUSION

The variety of different nanostructured organic-inorganic hybrid smectite based composites (HC) was synthesized. Their chemical and phase composition together with morphological and textural properties was determined. The most important feature that influenced HC adsorptive and electrochemical properties was the arrangement of (bio) organic phase within inorganic aluminosilicate structure. HC with high amounts of intercalated quaternary alkyl ammonium cations (QAAC) showed superior adsorptive potential toward pertechnetate and anionic textile dyes. HC obtained with chitosan although with somewhat lower adsorption capacity represent green alternative to HC with QAAC. Glassy carbon electrode modified with

different HC improved electrochemical response toward phenol, its nitro derivatives and nicotine.

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