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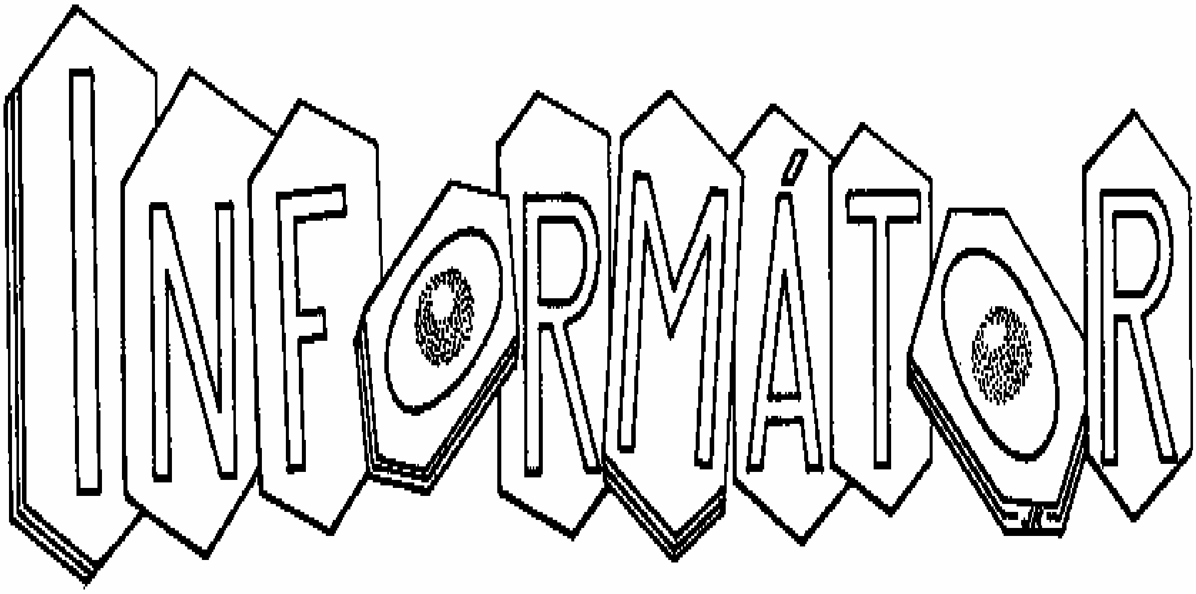

INFORMÁTOR

Book of Abstracts

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Theme A

Phyllosilicates in the Sediment-Forming Processes

PHYLLOSILICATES IN THE SEDIMENT-FORMING PROCESSES: WEATHERING, EROSION, TRANSPORTATION, AND DEPOSITION

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EXTENDED ABSTRACT

Contents: 1. Introduction. - 2. Groups of phyllosilicates, their common species and structural states in the sediment-forming processes of the argillosphere. - 2.1. A basic structural and chemical classification of phyllosilicates. - 2.1.1. Neutral 1:1 structures: the kaolinite and serpentine group. - 2.1.2. Neutral 2:1 structures: the pyrophyllite and talc group. - 2.1.3. High-charge 2:1 structures, non-expansible in polar liquids: illite and the dioctahedral and trioctahedral micas, also brittle micas. - 2.1.4. Low- to medium-charge 2:1 structures, expansible phyllosilicates in polar liquids: smectites and vermiculites. - 2.1.5. Neutral 2:1:1 structures: chlorites. - 2.1.6. Neutral to weak-charge ribbon structures, so-called pseudophyllosilicates or hormites: palygorskite and sepiolite (fibrous crystalline clay minerals). - 2.1.7. Amorphous clay minerals. - 2.2. Order-disorder states, polymorphism, and polytypism. - 2.3. Interstratifications (mixed-layer structures). - 3. Phyllosilicates in the sediment-forming processes: effects of parent rocks and acting factors. - 3.1. Weathering. - 3.1.1. The importance of the physical/chemical weathering ratio and chemical maturity. - 3.1.2. Chemical stability and solubility of rock-forming silicates under the conditions of weathering (and volcanic glass). - 3.1.3. Crystallization of newly formed phyllosilicates. - 3.1.4. Phase diagrams. - 3.1.5. Weathering of rocks: a quantitative approach. - 3.2. Erosion and transportation: hydration, softening, swelling, physical disintegration, milling, abrasion, delamination, dispersion, and sorting. - 3.2.1. Movement of solid material in streams and its partial deposition. - 3.3. Deposition of phyllosilicates in different fluid dynamics of streams expressed by Reynolds (Re) and Froude (Fr) numbers. - 3.3.1. Deposition from unflocculated and flocculated suspensions. - 3.3.2. Deflocculation and coagulation of clay particles. - 3.3.3. Sediments rich in clay and silt phyllosilicates covering vast areas of seas and oceans. - 4. Conclusion. - 5. References.

The most important results cited from literature and from my research are summed up in eleven key points:

1 - The different and numerous irregularities in the structure of crystallizing phyllosilicates might have several causes. One of them lies in variable bond lengths between cations and shielding anions (O^{2-} in tetrahedra, and $O^{2-}+OH^{-}$ in octahedra). This variability is caused by the different sizes of central cations. The cation-anion bond length increases with the size of cations. The leading central cation in tetrahedra is Si, but it may occur partially substituted, for example, by larger Al or Fe^{3+} cations. The central cations in octahedra are mostly Al, Fe^{3+}, Mg, Fe^{2+}, rarely Li^{+} or other cations; in addition, vacancies exist among the octahedra if they are occupied with trivalent cations only. The larger the irregularity in the chemical substitution in tetrahedra and octahedra in a mineral species or group, the more variable the resulting cell dimensions, symmetry, interatomic forces and their local balance, deformation and distortion of octahedra and tetrahedra in the final structure can be. The result is that actual sheet and layer structures depart from the ideal hexagonal arrangement. The general phenomenon of the deformation of tetrahedra and tetrahedral sheets and octahedra and octahedral sheets leading to the crystallization of polytypes has been compiled and summarized in detail by Weiss (2004). Already in 1959-1963, Radoslovich with his coworkers (quoted in detail by Konta, 2005) drew the crystallographers' attention to the fact that the energetic state expressed in "local balance of forces" in layer structures may provide a means of structural control over polytypism. The first cause of order or disorder in the structure of phyllosilicates and the resulting polymorphs and polytypes as well as of the interstratification phenomenon lies in the chemical micromilieu acting during the crystallization. The second cause contributing to the diverse polymorph, polytype, and interstratification formations (including indexes of crystallinity) is the accepted low or higher thermal energy. Higher temperature and permeability acting for a longer span of time support better ordering.

2 - Vast areas of tills and tillites in arctic parts and adjacent areas of the Earth, together with debris in mountaneous and arid regions in other climatic belts, contain immense volumes of chemically almost intact phyllosilicates. They originate from different sedimentary, metamorphic, and igneous parent rocks. The composition and properties of parent rocks and minerals in the weathering profiles, the elevation, relief and topography of source areas and the climatic conditions, especially rainfall and temperature, control the resulting assemblage of phyllosilicates to be transported after erosion. A simple division of recent global soil zonality with the most typical phyllosilicates, practical for global sedimentology, was published by Chamley (1989). It can be summarized as follows: *lithosols* formed in very cold or hot-dry climate are a rich source of the original phyllosilicates contained in parent rocks; *podzols* typical of cool-wet climate contain, depending on horizons, any original phyllosilicates (Gjems, 1967) preserved according to their chemical persistence (expressed by index of corrosion I_{KO}) as well as newly formed 2:1 and 2:1:1 phyllosilicates and their irregular interstratifications, kaolinite and free silica, but podzols can form also in other climatic conditions; *brown and chestnut soils* formed in temperate-humid climate contain phyllosilicates inherited from parent rocks, as well as newly formed illite, vermiculite, smectite, and chlorite minerals including their irregular interstratifications, kaolinite and also allophane; *chernozems, vertisols and related soils,* rich in dioctahedral smectites with some admixtures of illite and irregular interstratifications of 2:1 structures, and in some location containing calcite, form in temperate to warm-subarid climate; *calcrete,* also called *caliche,* a lateral equivalent of vertisols developed on calcareous rocks, usually contains newly precipitated calcite, Mg-smectite, palygorskite, and sepiolite; *laterites and latosols* formed in hot-wet climate are rich in kaolinite, gibbsite and iron oxyhydroxides. Chemical decay decreases with the depth of weathering profiles. (Main minerals see Fig. 5.) Clastic material eroded from profiles and fragmented debris in horizons where physical fragmentation of primary constituents prevailed over their deeper chemical changes usually contains overwhelming amounts of phyllosilicates inherited from original rocks.

3 - The mutual comparison of the I_{KO} values (index of corrosion, Konta, 1984) of rock-forming silicates leads to a conclusion that a considerable volume of phyllosilicates in the sedimentary lithosphere, apparently comparable or still larger than that occupied by clastic quartz, has a source in preexisting phyllosilicates of different bedrocks (sedimentary, metamorphic, and igneous) affected by weathering and erosion. The enormously high accumulation of phyllosilicates in the sedimentary lithosphere is primarily conditioned by their high up to extremely high chemical stability in water-rich environments. This property is remarkable despite the fact that clay minerals in sediments are noted for their large specific and thus reactive surface area during the long-term contact with water. The high production of phyllosilicates in weathering crusts and soils under favourable weathering conditions, especially in tropical and subtropical climatic belts, is yet another but secondary source of clay minerals transferred into the sedimentary lithosphere. In geological literature, the newly formed phyllosilicates - crystallizing in soils as the dominating global source of argillaceous accumulations in the sedimentary lithosphere - have been overestimated for a long time. It is, however, difficult to differentiate the same phyllosilicate species of the size of physical clay originating in bedrocks from those newly formed in weathering crusts, jointly eroded, transported, and later occurring together in argillaceous sediments.

4 - *Volcanic glass* in contact with water is less stable and its argillization proceeds more rapidly, compared with the behaviour of crystalline primary silicates of the corresponding chemical composition. The removal of dissolved components from glass, however, is retarded due to a limited system of cracks and no dense cleavage system typical of most crystalline minerals. This is the reason why, despite their faster dissolution, volcanic glasses still better preserve their geochemical milieu than crystalline primary silicates (compare the results by Gilg et al., 2003).

5 - The dissolution of silicates in different dense rocks under weathering conditions and the sequential crystallization of newly formed phyllosilicates is strongly and for prolonged periods influenced by chemical microenvironments existing within the space of each primary mineral. The clay pseudomorphs after potassium feldspars, Na- or Na-Ca-feldspars, biotite or amphibole in deeply weathered igneous rocks, testify that the sequentially crystallizing clay mineral species, their chemistry, structural ordering, and morphology are under the influence of long-range outlasting geochemical microenvironments in the space of the original silicates (Proust and Velde, 1978; Konta, 1979, 1981; Konta et al., 1972). The clay pseudomorphs after primary rock-forming silicates in weathering crusts preserve the traces of chemical micromilieu of original minerals not only in the deep parts but also in the completely argillized upper parts of the regolith profiles. The movement of aqueous solutions is initially controlled by the fractures of different origin but mainly by the capillary system of the original cleavage and by the crystal structure of individual primary silicates. Sooner or later, the flow is combined with ion diffusion in the very dense capillary system within the newly formed clay matter. The narrow capillary and interlayer pathways, their tortuosity, and large specific surface area of newly formed clay matter retard any big scale equilibrium in dense igneous and metamorphic rocks. The individual chemical and microstructural micromilieu in each clay pseudomorph plays a big role during the entire sequential argillization of igneous and metamorphic rocks. The influence of chemical microenvironments of original rock-forming silicates and their clay pseudomorphs, however, can strongly or totally be reduced in rocks of higher porosity and permeability such as, for instance, in kaolinized arkoses. In soil horizons, the different chemical microenvironments after primary silicates are still more or less apparent despite the percolation of aqueous solutions is exceedingly open, accelerated, and the mutual mixing of chemical species in newly formed clay minerals much broader and easier (Wilson, 2004).

6 - Figures 6, 7 and 11 together with their explanations (Konta, 1992) show that most phyllosilicates in different rocks have a good chance to survive in the weathering crusts of climatic belts between 15° and 90° latitudes north and south of the equator. Even in the tropical humid belt along the equator, about 30° latitude wide, the phyllosilicates rich in aluminium, i.e. kaolinite and dioctahedral micas, have a chance to withstand the weathering. Smectites, vermiculites, chlorites, and all trioctahedral phyllosilicates are less resistant because they still contain more mobile cations (Na, Ca, Mg, Fe) in susceptible amounts.

These phyllosilicates can endure the strong chemical destruction even in the tropical belt if the elevation, the relief and the topography of an eroded region create a sufficiently higher physical/chemical weathering ratio of the exposed rocks.

7 - The erosion and transportation of the greatest part of the earth surface takes place in water. Phyllosilicates transported in contemporary streams have different sources. They occur as constituents in eroded fragments of various igneous, metamorphic, and sedimentary rocks, from where they are loosened. They also occur as newly formed clay minerals from weathering crusts and soils. Sedimentary rocks may contain phyllosilicates that already passed one or more weathering events due to their extremely high chemical stability. This counts especially for kaolinite and dioctahedral micaceous clay minerals rich in Al as the representatives of common mineral species in argillaceous sediments (see their I_{KO}, Figs. 6 and 7). The strong hydration capability of most clay aggregates and crystals of phyllosilicates (excepting talc and pyrophyllite) still leads to their softening, swelling, easy physical disintegration, and dispersion in fluvial currents. The eolian dust, volcanic ash, submarine volcanic effusives, and hydrothermal effluences can variably contribute to the composition of any sediment rich in phyllosilicates deposited in water.

8 - Coarser crystals of micas, chlorites, vermiculites or kaolinite in different stages of physical weathering disintegrate during continual transportation. The turbulent flow of any river, the impacts and pressure from harder, larger, and heavier fragments make the milling, grinding, and partition of all relatively soft phyllosilicates and their fine-grained aggregates easy. The microscopic or submicroscopic fissures are controlled by the best-developed crystal structure planes densely occupied by atoms, but not only along the basis. Phyllosilicates, therefore, are concentrated in clay and fine silt fractions and mostly occur as very thin, irregular flakes. Planar bedding and considerably low aggregate hardness of argillaceous sedimentary rocks and fissility or foliation of metamorphic rocks rich in fine-grained mica and chlorite enable their easy milling or abrasion and disintegration to still thinner and finer flakes of phyllosilicates. Coarser fragments of argillaceous rocks and crystals of phyllosilicates from bedrocks and soils are very sensitive to impacts and pressure from the fragments of co-transported harder and denser rocks and minerals in turbulent streams. This is the most important mechanical phenomenon supporting the enormous accumulation of lutite sediments rich in phyllosilicates in geological history. Table 2 shows a comparison among hardness, density, and cleavage of some common rock-forming minerals reflecting these properties in their rock fragments transported in rivers (see also Fig. 14). The softness of phyllosilicates and their aggregates still increases with hydration.

9 - The absence or very rare and extremely slight admixture of the phyllosilicate interstratifications containing expansible layers in suspended solids of 13 major world rivers studied can be explained by the following interpretation: These mixed-layer structures rapidly swell in the dynamic environments of rivers and their milling process, disintegrate, and disperse up to very fine almost monophase particles of nonexpansible (mainly micas and chlorites) and expansible (smectites and vermiculites) phyllosilicates. A considerable admixture of quartz is always present in river suspensions under sufficient current energy. Further detectable non-clay minerals in river suspensions studied are (Konta, 1985): potassium feldspar, acid plagioclase, amphibole, calcite, dolomite (and in the Waikato opal with cristobalite in organic remains). Quartz, acid plagioclase, and potassium feldspar are those primary clastic minerals that commonly occur in suspensions of the 13 rivers in various regions of the world. Amphibole is the fourth primary silicate sometimes occurring. The reason for the common occurrence of these four silicates accompanying the predominating phyllosilicates in river suspensions is that they are the most abundant among the minerals in crustal plutonic rocks (Wedepohl, 1969). The purely eolian and glacial sediments could theoretically contain enough preserved phyllosilicate interstratifications with expansible layers derived from eroded bedrocks or soils.

10 - After the erosion of weathering crusts, the original clastic material transported in fluvial currents is not only sorted but it undergoes strong abrasion, delamination, and dispersion similarly occurring in ball or pebble mills. The abrasion is very strong in the presence of big and hard enough rudite fragments (essentially above 2 mm). Crystals of phyllosilicates and fragments of rocks rich in phyllosilicates reflect their physical properties in relation to co-transported fragments of harder minerals and rocks. The most important physical properties of phyllosilicates and their fine-grained aggregates reflected during the transportation and settling are as follows: a) good cleavage of crystals, b) easy separability of small particles from clay pseudomorphs and fragments of aggregates of bedded, fissile or foliated rocks rich in phyllosilicates, but mainly c) their relatively low hardness in relation to other common rock-forming minerals and rock fragments co-transported in streams. In geological history, the easy fragmentation of the phyllosilicate matter transported in streams, together with the natural limitation of their crystal growth under the surface pT conditions, substantially contributes to a huge accumulation of lutite sediments rich in phyllosilicates (see Fig. 15). Coarser flakes, mostly fragments of micas, especially of muscovite, once transported by saltation or traction in streams occur in sands, sandstones, gravels, conglomerates or breccias of fluvial sediments, and in other near-shore sediments of lakes and seas.

11 - Phyllosilicates and accompanying detrital minerals in recent marine muds, covering vast areas of seas and oceans, as well as in lacustrine muds correspond with those transported in fluvial suspensions. The resulting quantitative ratios among deposited terrigenous minerals are controlled by the composition of eroded weathering crusts including their bedrocks, by the continuing disintegration and sorting of detrital material during transportation, and by the settling conditions in final depositional environments. Under new environmental conditions during burial, the phyllosilicates tend to new equilibria through chemical and structural changes.

Each of the key points, however, still requires a more thorough and deeper investigation. The compiled facts and new interpretations should inspire to new experimental activity and more thorough study of samples and processes in nature. The quoted literature, figures, and table 2 can be found in the complete paper, which contains 16 figures and 2 tables. It will be published in *Acta Geodynamica et Geomaterialia* (Prague, 2009).

After the completion of this extended abstract, I came across a remarkable paper on weathering processes by Meunier et al. (2007; *Clay Minerals,* vol. **42**, no. 4, 415-435). I recommend it to everybody who is interested in detailed research respecting a broader petrographic and geologic context.

MINERALOGY OF THE CLAY GOUGE ON PRAGUE FAULT

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The fault cataclastic (gouges) rocks were firstly described by Lapwort (1885). Later on many papers dealt with this theme either describing or classifying the rocks (e.g. Waters, Campbell, 1935; Hsu, 1955; Christie 1960, 1963; Reed, 1964; Spry, 1969; Higgins, 1971; Sibson, 1977). The fundamental work of Wise et al. summarizing a fault rock terminology was published in 1984. These authors divided the fault rocks into three basic types namely cataclasites, mylonites and metamorphic rocks. Dislocation clays together with breccia and micro-breccia belong to the cataclasites being considered as a typical product of tectonic movements occurring on dislocation planes. These clays are mostly considered as fine-grained low-temperature clays, the product of cataclase.

The area protects a rock outcrop representing the tectonic contact between older underlying clay shales of the Záhořany rock series and younger Skalka quartzites bordering alongside of the most important fault in the Prague territory, the Prague Fault. Skalka quartzites form massive to thick benches inset 40° towards SE. NW part of the outcrop is individualized as an anticline with limbs dipping 60 to 80° towards NW. Thickness of the benches varies. The beds superposed to quartzite are composed of a dark to blackish grey clay shale as a part of Dobrotivá rock series (Ordovician) (Röhlich, 1953). According to Vorel (1982) the fault caused a vertical movements with displacement magnitude around 900 m, but possibly 1000 to 1800 m in post-Ordovician period. The fault zone is filled with clay or sandy-silt to silty-sand matrix with scattered fragments of the surrounding rocks either shale or quartzite.

Clay and accompanying minerals were identified by X-ray diffraction analysis of the powder preparations with random orientation of particles and with preferred orientation of particles separated as <4 µm fractions.

The Prague fault is a tectonic boundary between underlying clay shales of Záhořany series and Skalka quartzite. Quartz, mica/illite, kaolinite, chlorite, sporadic feldspar and gypsum were identified in the powdered preparations by X-ray diffraction. The clay fraction (< 4 mm) is composed of mica/illite predomitaning over kaolinite and sporadic chlorite and gypsum.

Mica/illite has Kubler index (KI) in the range 0.7 (Δ2θ°). The mica/illite KI with the values in the range 0.55-0.75 (Δ2θ°) indicates a low temperature polytype 1Md on the basis of X-ray reflection area between 15-35 2θ°.

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Poster

GENESIS OF BENTONITE IN THE KREMNICA GRABEN (WESTERN CARPATHIANS)

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The most important bentonite deposits in Western Carpathians are located in Kremnica volcanotectonic graben in the Central Slovakia Volcanic Field. The deposits are hosted by heavily altered rhyolite and rhyolite tuffs of the Jastrabá Fm. (12.9-10.7 Ma) that occur S of the Kremnica low-sulfidation Au-Ag deposit, situated at the E side of a local horst along N-S trending faults. Alteration products show a clear N-S zonal arrangement. In the north, (Čertov Vrch) kaolinite predominates, while mixed-layer illite/smecite (rectorite) dominates in altered rhyolite tuffs and glassy rhyolite 2 km southward (Dolná Ves). These types of alteration show a clear input of hot hydrothermal fluids, probably flowing laterally out of the Kremnica hydrothermal system. A general decrease in temperature from N to S was proven by fluid inclusion and stable isotope data along the entire hydrothermal vein system (Koděra et al., 2007). Kaolinite-bearing alteration indicates the presence of acid groundwater, probably resulting from the condensation and dissolution of H_2S and CO_2-bearing vapours released by boiling of hydrothermal fluids in a greater depth. Some mineralised quartz veins are also present here, originated at 160-130°C based on fluid inclusions data. Illite/smectite deposits result from more neutral but still heated fluids (140-157°C; Uhlík & Majzlan, 2004). A N to S decrease in temperature was proven by increasing smectite component in this direction (6 % to 45 %). Decomposition of volcanic glass resulted in an enrichment of silica in the fluids. Smectite-dominated alteration occurs in southernmost parts of the system (Jelšový potok) and in its marginal parts (Kopernica in NW). Bentonites resulted here from alteration of tuffs and margins of rhyolite domes in fresh-water limnic environment. Formerly perlitic marginal parts of rhyolite domes often contain the best-quality smectite, but some smectite in rhyolite extrusives could also result from autohydrothermal alteration. Such an interpretation is consistent with stable isotope data of smectite in this setting. Smectite alteration changes into zeolite alteration with clinoptilolite and mordenite towards depth probably due to diagenesis and thermal effect of rhyolite extrusives. Bentonite deposits are often interlayered with limnic/lacustrine silicites (quartz or opal CT), resulting from episodical inflow of groundwaters enriched in silica derived from input of hydrothermal fluids from the N part of the system. Systematic increase of $\delta^{18}O$ values from N to S both in limnic silica and smectite clearly corresponds to decreasing temperature in this direction. Equilibrium fluids dominated by meteoric water range from 85° to 15°C and 50° to 20°C for limnic silica and smectite, respectively. Alternatively, this trend results from decreasing temperature of recrystallisation during burial and diagenesis.

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A SCHEME FOR THE CLASSIFICATION OF MICACEOUS MINERALS

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Micas are hydrated silicates with the layer crystal structure – a group of minerals with the extraordinary variable chemical composition of the general crystallochemical formula:

$$(K,Na)\ (Al,Fe,Mg)_{2\text{-}3}\ [(Si,Al)_4O_{10}](OH,F)_2\ .$$

In case of true micas univalent cations are placed in the interlayer space whereas divalent interlayer cations fill this space in brittle micas.

Micas can occur in a macrocrystalline form or in a size fraction below 4 µm. The latter are included in the subgroup of clay micas with the interlayer deficit of cations – the formula exhibits only 0.85-0.60 positive interlayer charges.

The scheme is here submitted in which micas are subdivided into macrocrystalline micas and clay micas by the horizontal boundary. According to the occupancy of octahedral positions, the macrocrystalline micas are differentiated among the subgroups of Mg-Fe-Al micas, Li-micas and micas with other cations.

The vertical boundary divides micas into trioctahedral and dioctahedral sections and each section is further subdivided according to the extent of Si substitution in the tetrahedral positions into the following types: Si_4, $(Si\ X)_4$, $Si_3\ X$, $Si_{2,5}\ Al_{1,5}$ (or $Si_{3,5}\ Al_{0,5}$), $Si_2\ Al_2$, where $X = Al, Fe^{3+}, B$.

Micas occur in various polytypic forms, that are designated by means of the Ramsdell indicative symbolics on the basis of the number of layers in the repeating unit along *c*-direction, e.g. 3T. The letters indicate the resulting lattice symmetry (M - monoclinic, T - trigonal, H - hexagonal, Or - orthorhombic).

A mineral of this group can be identified on the basis of basal reflection sequences 00ℓ and 060 diffractions. Basal *c*-spacing of micas amounts to 10 Å, lateral dimensions *a, b* fluctuate in the range of 5.2 - 5.4 Å, or ~9.0 - 9.3 Å respectively.

To identify a polytype we need the sequences of selected nonbasal X-ray diffractions. The intensity distributions of diffractions 20ℓ, 13ℓ and 02ℓ, 11ℓ are used.

Sometimes micas exhibit a tetrahedral and octahedral cation ordering. In the case of ditrigonal tetrahedral network positions T(1) and T(2) for tetrahedral cations are concerned. In octahedral network M(1) trans-positions and M(2), M(3) cis-positions can indicate the ordering of the octahedral cations.

Clay micas, illite (I) and glauconite (G) very often contain the smectite (S) component in various amounts, show some expansibility and create different sorts of interstratifications. Illite/smectites, according to Srodon, form a continuous sequence of interstratification: random, random/IS ordered, IS ordered, IS ordered/ISII ordered, ISII ordered – each type is related to specific range of expansibility.

The illite particles are at least 20 Å thick and they are composed of two 2:1 layers joined together by interlayer potassium. Ordered IS contains 50 % of the smectite units whereas ordered ISII 20 % of them. Such illite particle is ~ 40 Å thick. The 2:1 layers joined together by irreversibly fixed potassium produce the “fundamental” particle of interstratified illite/smectite. It is not possible to disaggregate mechanically this fundamental particle more.

NONTRONITES IN THE OKHOTSK SEA SEDIMENTS

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The composition, structure and peculiarities of crystal chemical transformations of clay minerals in context with geochemical processes in bottom sediments of North-east seas of Russia were investigated. Series of samples of clay substance of Ohkotsk Sea bottom from areas with essential various geochemical conditions were studied by powder X-ray diffraction, infrared and Mössbauer spectroscopy methods and indicated the presence of phyllosilicates. Analysis of obtained results showed that all clay substance is high-iron nontronite with Fe/(Fe + Al + Mg) ratios ranging from 0.83 to 0.88. Broad diffraction lines with a low intensity on the XRD pictures of starting material of different genesis are attributed to a high dispersity and poor structural order. Varying values of the basal reflections d_{001} in the ranges of 11 – 13 Å are caused by a degree of the cation fillings and hydrations of the interface space. The shifts of frequencies on IR-spectra are determined by isomorphism, it adjusts with the electron-microprobe analysis data. Ge99-43 nontronites as compared with Lv40-12 ones have a higher charge in tetrahedral layer and a level of the structural defects. Mössbauer spectroscopy indicates that some nontronites contain a small amount of Fe^{2+}. The partitioning of $^{VI}Fe^{3+}$ between cis (M2) and trans (M1) sites within the octahedral sheet was determined for nontronite of Lv40-12 and Ge99-43 series from Mössbauer data. It testifies about unstable and dynamic environment of crystallization resulting from postvolcanic hydrothermal manifestations. Textured-structural peculiar properties of the analyzed materials confirm that fact. According to the data of electron-microscope investigation during the crystallization of nontronites a significant role pertains to the bacteria which sorb Fe and Si, and under certain stoichiometric correlations between them promote the crystallization of nontronite. Sandy parts, gravel and solitary pebble of glacial delivery are found in some samples. The given impurity of the material introduced from continent is shown on diffractograms as quartz and feldspars. Taking into account that receipts of an ice material in the central areas of Okhotsk Sea is characteristic for cold time intervals (especially during glaciation), it is possible to assume, that the studied mineralization is rather young (in geological sense) formation.

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MICROANALYTICAL INVESTIGATIONS OF KAOLINITE PARTICLES

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White paper- or porcelain-clays should contain only small amounts of iron or titanium. These elements occur in such clays primarily in individual mineral-particles, including goethite, hematite, magnetite, biotite, pyrite, anatase, rutile, titanite or in Fe-containing clay minerals (illite, nontronite, IM-mixed-layers) which possibly may be reduced by leaching or particle separation. The amount of Fe or Ti that actually occurs within the kaolinite crystals is lesser known. The paper reports on the results of microanalytical studies of Fe and Ti in single kaolinite particles from different clay deposits.

The microanalysis was made on suspended specimens of the grain fraction < 2 µm of selected samples of different clay deposits, using the analytical TEM JEM1210, Jeol (120 kV, LaB_6-kathode) and LINK OXFORD-EDX-system (ISIS, S-ATW Si(Li)-detector). Analyses were performed on single isolated particles and portions of the particles which were not influenced by neighbouring particles. From each specimen up to 100 particles or particle-areas were analyzed, often more than one on the same particle.

The titanium is mostly fixed in very small individual particles of titanium oxide which are agglomerated on kaolinite. The iron content of the kaolinite particles varies between 0.0x to >1 %. In some cases there are very small Fe-bearing inclusions, overgrowths or intergrowths (Fe-bearing pyllosilicates) in the kaolinite to find in other cases the Fe-bearing kaolinites are very homogeneous. The different types of iron fixation will be shown in detail and there genesis discussed.

Theme B

Evaluation and Discussion of two Books Edited by Renown Editorial Houses

CONTRASTING BOOKS ON CLAY MINERAL SCIENCE – HOW SHOULD THEY BE JUDGED?

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The appearance of two new books covering clay mineral science in the last three years is an important event, particularly as one is a handbook and the other suggests that the answer to almost any clay mineral question can be found within it. Such are the *Handbook of Clay Science* and *Clays* published respectively by Elsevier (2006) and Springer (2005). At the time when these books were conceived the library world was a rather different place than it is now. Gone are the days when it did not matter for whom a book was published. Was it really for the benefit of the reader and not the publisher and author? At least the library would always purchase a copy however expensive it might be. Today with massive cutbacks in library funds, at least in the UK – for example the Geoscience Library at Cambridge University has suffered a 40 % reduction in its annual allocation: the subscriptions to many old favourites but little used journals had to be stopped, fewer books are now purchased, and these are largely concerned with the teaching courses. The reason for this approach – and I do think it is the correct one in the circumstances – is the recognition that the primary source of reliable data are the well refereed journals and monographs; whereas books today are, with exceptions, much less rigorously reviewed and refereed and are only a secondary source of information. This may be hard on books that are well conceived, researched, written and produced, but they are usually recognised for their worth: they benefit from their use as standard texts, and the likelihood of additional reprintings and editions where errors can be corrected and new developments can be included. A pertinent question for clay scientists to ask themselves – why are so few books on clay mineral science represented by additional reprintings or second editions. The *Handbook of Clay Science* and *Clays* should be examined and judged by the traditional and not always popular standards of quality, accuracy, balance, inspiration and user friendliness.

The Organizing Committee of the 18th Clay Conference in Czech Republic expects a valuable discussion on the most modern manuals on clay minerals or clay science. Although the Organizing Committee is well aware of the power and wide co-operation of the renown editorial houses with excellent scientists and authors, some critical views of the everyday book-users could be useful. The Czech National Clay Group supposes that evaluation and critical discussion of recent handbooks or manuals should be a quite legitimate part of scientific conferences. The organizers of the 18th Clay Conference start with this original endeavour in 2008 hoping that it will be appreciated by editorial houses, authors of books and especially by their potential readers.

PREHISTORY OF CLAY MINERALOGY – FROM ANCIENT TIMES TO AGRICOLA

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This contribution gives a succinct overview on clays and argillaceous raw materials in the texts of ancient authors (Theophrastus, Vitruvius, Strabo, Columella, Pliny, Dioscorides, Galen), including an early medieval one (Isidore of Seville), and summarizes the reception of this tradition in the Renaissance by the “Father of Mineralogy“, Georgius Agricola, in particular his first mineralogical work, “Bermannus“ (1530), and his main work concerning mineralogy and mineral classification, “De natura fossilium“ (1546). We give a brief summary of the second book of A.’s work “De natura fossilium“, which is the first attempt towards a rational and systematic classification of clays and argillaceous raw materials (i.e. earths, “terrae“). Based on our work on the first Czech translation of this text, A.’s main authorities and literature sources are recalled and separated from new information added by A. himself (either directly as a result of his own experience or talking to men of practice in Jáchymov/Joachimsthal and the Krušné hory Mts. region or indirectly as a result of personal communication with travelling savants and from traditional late-medieval miners‘ knowledge). It is shown that Isidore must in certain aspects be considered as a predecessor of A. (although the latter never mentions him), that A.‘s emphasis on properties was largely determined by Aristotelian tradition and that the main novelty of A.‘s mineral classification was his “combinatorical” approach. Further it is shown that A. knew “nacrite” (“Steinmark”) and was well aware of the refractory qualities of kaolin (“argilla”), but that he still did not distinguish between clay and chalk. Thanks to A., the Greek word “smektis” (denoting the cleaning ability of very fine – and therefore highly adsorptive – clay, i.e. fuller’s earth), has given its name to the clay mineral group of “smectites”. Interestingly, among the places where fuller’s earth is found he mentions Kadaň/Kaaden in Bohemia (as if he already knew the Rokle bentonite deposit). Following Vitruvius, Pliny and others, A. does not forget to mention (in the tenth book of “De natura fossilium“) the “powdered rock from Pozzuolo”, which has to be considered as the first hydraulic cement (geopolymer). Cum grano salis it may therefore be said that A., being acknowledged as the “Father of Mineralogy” in general, is also the “Father of Clay Mineralogy” in particular. His treatment of clays and argillaceous raw materials is remarkable mainly because it is completely aside from the mainstream interests of that time, which concerned ore mining, refining and metallurgy. A.’s work “De natura fossilium“ provided the fundamental compilation of ancient mineralogy, and – much more than his famous posthumous magnum opus “De re metallica” (1556) – broke with medieval tradition and provided a basis on which further generations could build. For more than 250 years, this was the last word on clays and argillaceous raw materials, until at the beginning of the 19th century F. F. Reuss performed his electrophoretic experiments (1809). Modern clay science, of course, had to wait another 100 years, until the discovery of X-ray diffraction by M. v. Laue (1912) gave the starting point for the structural analysis of clay minerals.

Theme C

Clay Materials in Traditional and Modern Technologies

AGEING OF HISTORICAL CERAMIC

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Clay raw materials are the basic component for the ceramic production from prehistoric age till the present times. Mixtures for historical ceramics production were created only from soils where the main component was the fine dispersal plastic clay accompanied by various raff grained non-plastic components such as quartz, feldspar, micas and others. One of the basic process in ceramic technology, besides forming and drying, is firing. Right firing process influences significantly properties of originated ceramic body related with given raw material composition.

Considerable process which is running in the stage of heating in temperature range from 450 to 750 ^{o}C is the dehydroxilation of clay minerals. This endothermic reaction is accompanied by the evaluation of water steam and shrinking of ceramic body and originates marked changes in the structure of clay minerals. The instable phases, practically non-crystalline, originate from the stabile crystalline phases of clay minerals. But, non-crystalline phases are insufficiently protected against long-time attack of humidity. They react also with the atmospheric humidity condensate in open pores and their simultaneous volume increasing proceeds. This process is irreversible and with long-term duration. Water is bonded in the structure of dehydroxilated clay minerals by variously strong chemisorptive bonds and it could be removed only by annealing of ceramic body and it is releasing in temperature range from 200 to 650 °C. The volume increasing of ceramic body causes often unwanted cracking of surface layers, e.g., glazes. The described phenomenon is called the irreversible moisture expansion of porous ceramic. This phenomenon is also called the ageing of ceramic. The moisture expansion is utilized not only in field of historical ceramics, but it is necessary to take attention in contemporary porous ceramic production, e.g., wall tiles, masonry units (bricks and blocks) and various structure elements from porous ceramic. The mentioned long-term process could be accelerated by special curing method, e.g., boiling in water under atmosphere pressure or high pressure steaming in autoclave. The value of irreversible moisture expansion is determined from length differences of samples measured before and after accelerated ageing test.

The ageing of historical ceramic was studied on the sufficiently large set of roof tiles. The dependences of moisture expansion on chemical and mineralogical composition and basic properties of ceramic shard, i.e., water absorption, bulk density, apparent porosity, apparent density and pore size distribution have been studied. Obtained results contribute to better understanding of substance of irreversible moisture expansion of porous ceramic made from mixtures containing clay minerals.

ALTERATION PROCESSES IN BENTONITES

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The main use of bentonites has traditionally been in drilling muds, for bonding in foundries and for pelletizing iron ores. These materials have also found many uses in cosmetic formulations and the clarification of edible oils, fats and beverages. In addition, bentonites have an increasing potential in civil engineering and environmental applications. In the Czech Republic, Czech Geological Survey performs research on suitable montmorillonite-rich clays and bentonites for environmental applications. The evaluation of local deposits is in progress and the selected physico-chemical properties of montmorillonite-rich materials and bentonites are being determined.

In the recent times, one of the most important uses of bentonite is a buffer material for barriers in geological disposal facilities for radioactive waste. Search for suitable buffer clays for isolating canisters with a high-level radioactive waste goes on in many countries. This poster describes two types of experiments: long-term (3 years and 9 months) and short-term (2 months). The influence of heat (and saline) treatment on structural changes in bentonites and their selected physico-chemical properties was examined.

Long-term experiment: The Mock-Up-CZ experiment simulated vertical placement of a container with the radioactive waste. The experiment has been constructed at the Centre of Experimental Geotechnics, Czech Technical University in May 2000. The model consists of bentonite barriers (buffer - bentonite blocks, backfill) and a heater. The buffer material used in the experiment was a mixture of 85 % of ground non-activated Ca/Mg bentonite from Rokle deposit, 10 % of quartz sand and 5 % of graphite. The thermally induced bentonite samples were studied using powder X-ray diffraction (XRD). Two transformation processes were identified; illitization and beidellitization. These processes affected nine samples only. On the bentonite envelope of the container (backfill) newly formed gypsum with illitic aureol was observed. The transformation processes affected only minor part of the original montmorillonite (< 1 %) and the extent of transformation is not sufficient to influence significantly physico-chemical properties of the bentonite barrier.

Short-term experiment: Two types of bentonites were used in the experiment − the international standard of Na-bentonite (Wyoming MX 80) and Ca/Mg bentonite from the Rokle deposit. The aim of this study was to determine effects of the heat (40 °C – 120 °C) and saline (30 wt. % NaCl) treatment on selected physico-chemical properties (cation exchange capacity, swell index) and to identify the transformation processes occurring in the bentonite and compare obtained results to the MX 80 standard. A significant decrease in CEC and SI values was observed after a heat treatment. Samples in high saline environment showed more rapid decrease of CEC and SI (even at the lowest temperature, 40 °C). Structural changes in the bentonite were identified using powder XRD. Three main transformation processes (illitization, beidellitization and kaolinization) were identified. Initial values of CEC and SI are higher in MX 80 bentonite than in the Rokle bentonite. However, the decreasing trends recorded for the Ca/Mg bentonite after heat/saline treatment are very similar to the Na-bentonite standard.

CLAY MINERALS AS MATRIX FOR FUNCTIONAL NANOSTRUCTURES

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Remarkable physical and chemical properties of clay minerals still attract the attention of technologists as matrix for anchoring of various guest species. Nowadays mineralogy provides us with a lot of valuable information about the structure, physical and chemical properties of cationic and anionic clays allowing us their structural modification. This structural modification can be based on various techniques: (1) ion/exchange intercalation reaction, (2) intercalation based on ion-dipole interaction of polar neutral molecules with interlayer cations, (3) surface modification of clay particles by anchoring of ions and/or polar organic molecules on the clay particle surfaces, (4) anchoring of nanoparticles of metals, metal oxides and sulfides with and without various dopants on the clay surface. Nanostructures created by these techniques can find a wide scale of practical applications like sorbents, catalysts, photocatalysts, antibacterial nanocomposites, hybrid organo-inorganic pigments, nanofillers for composites, various photofunctional units (optical switches, laser dyes), etc.

The functionalization of these nanostructures in Nanotechnology Centre VŠB-TU Ostrava is focused on the following fields: catalysis, photocatalysis, antibacterial agents, nanofillers for composites with polymers, light emitting and photosensitive devices, laser dyes, etc. The key problem we have to solve is the choice of suitable combination of host matrix and guest species in order to reach the desirable properties. To solve this problem we have to characterize the clay host matrices as to the composition, ion exchange capacity, structural parameters and clay particle size distribution. All these characteristics affect the structure and function of final nanocomposites hence the special attention is paid to the investigation of these relationships by combination of X-ray fluorescence, atomic emission and IR spectroscopy, X-ray diffraction, electron and AFM microscopy, Mössbauer spectroscopy, and particle size analyzer.

The important task in design of new functional nanostructures based on modified clays is the search for the suitable host-guest combination in order to optimize the desirable properties. Solving this problem, we use molecular modelling as the useful complementary tool to analytical methods. The use of molecular modelling enables the understanding of structure-properties relationship and the prediction of structure and properties and reduces the experimental time-consuming search for the optimum combination of host matrix and guest species.

The characterization of final nanostructures – intercalates and nanocomposites as to their structure, stability, composition and properties carried out in our laboratories uses the combination of experimental methods mentioned above with the molecular modelling, completed with the testing of catalytic, photocatalytic and antibacterial activity. The device for the on-line monitoring of catalytic and photocatalytic reactions constructed in our laboratory enables to analyze the reaction kinetics and reaction products.

Fe/Mn MODIFICATION OF ALUMINOSILICATES: MECHANISM AND KINETICS OF SURFACE PROCESSES

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Adsorption of As, Se and Sb oxyanions on natural clay materials (often inferior for another use) has got into increasing concern for economical end ecological reasons. However, aluminosilicates are not selective sorbents for anionic contaminants thanks to a low pH(ZPC). A simple Fe/Mn pre-treatment of initial material can significantly improve their sorption affinity to oxyanionic contaminants, including arsenites and arsenates, selenites and selenates and antimonites and antimonates, respectively (Gupta et al., 2005, Doušová et al. 2006). The pre-treating methods included Fe^{II} (Bonnin, 2000), Fe^{III} (Izumi et al., 2005, modified), and Mn^{II} (Herzogová, 2007) modification. Mineralogically pure clays were used for surface changes study – two kaolins from West Bohemia with the high content of kaolinite (>80 %), bentonite (Riedel de Haën, Germany) containing mostly montmorillonite and standard Ca-rich montmorillonite SAz-2 (Arizona, USA).

The adsorption efficiency of modified sorbents increased significantly for all investigated systems (to >90 %) by the initial As/Se/Sb concentration of 10-40 mg L^{-1}, and the solid-liquid ratio ranged from 0.5 to 8 g L^{-1}. The mechanism and kinetics of surface processes depend on the structure of raw material (steric properties, chemical and mineralogical composition, specific surface area) and applied treating method associated with Fe/Mn chemistry and corresponding phase equilibrium. The quality of initial solution (pH, As/Se/Sb concentration) was also significant. The characteristic changes on the sorbent surface during the pre-treating processes were checked up with infrared spectroscopy and voltametry of microparticles.

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FRACTAL DIMENSIONS OF BARRIER MATERIAL

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Bentonite is one of the most important components of the majority of engineered barriers and seal designs. The problem of a natural material application for a barrier construction may be relatively divided into two smaller problems: 1) a study of radionuclide sorption by clay particles with regard to structural features and physical-chemical properties of clays and clayey materials and 2) a study of pore space among the barrier material particles. The study of fractal properties of materials including those used as geochemical barriers allows an increase of their effectiveness for a repository construction.

A clay particle surface fractal dimension may be calculated by a sorption at molecules of different sizes or by various fraction specific surface measurements separated at a narrow size range.

Using the results of calculated fractal dimensions of the particle aggregate, the mixture of Wyoming bentonite MX-80 with $D = 1.72 \pm 0.14$ at $R = 0.97$ and $F_k = 369$ in size range from 70 μm to 1000 μm and the gravel substance with $D = 2.20 \pm 0.06$, $R = 0.99$ and $F_k = 767$ have the following values when bentonite/gravel ratio is 10 % and 90 % respectively, $D = 2.23 \pm 0.1$, $R = 0.96$ and $F_k = 281$, when the ratio is 30 % of bentonite and 70 % of gravel, then $D = 2.42 \pm 0.13$, $R = 0.88$ and $F_k = 98$. In this case the decrease of dispersion fraction R to 0.88 may be explained by rising of the multifractal structure. It is significant that the values were received for the experimental distribution for dry particles. After the particle saturation and swelling, and consequently the increase of their size, a new particle aggregate with dimensions $D = 2.67 \pm 0.07$, $R = 0.90$ and $F_k = 119$ was formed mixing bentonite and gravel in the ratio 10 % : 90 %. After changing the bentonite/gravel ratio to 30 % and 70 % respectively, $D = 2.83 \pm 0.04$, $R= 0.87$ and $F_k = 85$. The mixture with these fractal characteristics was considered to be very promising as a low-porous barrier material. The most complicated question arising at the final stage of balancing the barrier material components (qualitative and quantitative) is an agreement of the main characteristics of the components in the mixture in the way allowing an achievement the best characteristics of the barrier material in the whole. The difficulty lies not only in selection of the optimum bentonite and ballast sand or gravel fractions, their weight ratio, etc. but the mixing procedure itself (in dry or wet state), considering mechanical damages of bentonite particles, prediction of swelling features of the mixture and others.

INFLUENCE OF PARTICLE SHAPE ON THE VISCOSITY OF KAOLIN SUSPENSIONS

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It is well known that particle shape has a decisive influence on the rheology of kaolin suspensions. The degree of anisometry of a kaolin (characterized e.g. via an average aspect ratio) is reflected in an increase of the effective viscosity over and above that for a sphere suspension. Thus, one may predict certain features of the concentration dependence of viscosity (such as the intrinsic viscosity or the maximum volume fraction) when the particle shape is known. On the other hand, also the viscosity increase may be indicative of particle shape anisometry. In this contribution we recall first a simple method to determine an average aspect ratio of kaolins by comparing laser diffraction (or microscopic image analysis) and sedimentation data. In particular, when the median equivalent diameter has been determined by both methods (D_{50}^{L} and D_{50}^{S}, respectively), an average aspect ratio R is given by the formula $R = C \cdot \pi \cdot \left(D_{50}^{L} / D_{50}^{S} \right)^2$, where the proportionality constant C ranges from 0.5 to 1.5, depending on the model shape adopted (flat cylinder or oblate spheroid) and the particle orientation in the optical measurement (preferentially oriented or random). Second, we summarize the most important theoretical formulae and numerical results for the prediction of the intrinsic viscosity (Einstein-Jeffery coefficient) $[\eta]$ in dependence of the aspect ratio, with or without Brownian motion being taken into account (Jeffery, Peterlin, Simha, Kuhn-Kuhn, Scheraga, Brenner). When the effective viscosity η of dilute suspensions (i.e. suspensions with a solids volume fraction ϕ approaching zero, $\phi \rightarrow 0$) is given by a linear approximation of the form $\eta = \eta_0 \cdot (1 + [\eta]\phi)$, where η_0 is the viscosity of the liquid medium, it is shown that the intrinsic viscosity can in the absence of Brownian motion (weak Brownian motion case) approximated by a linear relation of the form $[\eta] = a + b \cdot R$, where a = 1.4 – 1.5 and b = 0.671 – 0.673. Finally, we present results of experimental measurements for different types of Czech kaolin products (floated kaolins). Kaolin suspensions are characterized by rotational viscometry. After optimizing the state of deflocculation, flow curves are measured (shear stress versus shear strain), relative viscosities η/η_0 are plotted in dependence of the kaolin volume fraction ϕ and fitted using the Krieger relation, $\eta/\eta_0 = (1 - \phi/\phi_C)^{-[\eta]\cdot\phi_C}$ (with the maximum volume fraction ϕ_C) to obtain the intrinsic viscosity $[\eta]$ for each kaolin type. Possible error sources are identified and the expected precision of this approach is indicated.

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Poster

ADSORPTION OF As AND Se OXYANIONS ON MODIFIED CLAYS

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The surfaces of clay minerals tend to attract cations due to their low pH_{ZPC}. Their modification with metal ions (using Fe^{2+}, Al^{3+}, Fe^{3+}/Al^{3+} and Mn^{2+} salts) increases the pH_{ZPC} and ability to catch anions (Bonnin, 2000; Wen-Hui Kuan et al., 1998; Ramesh et al., 2007; Herzogová, 2007). Thereby, the low-cost and easily available adsorbents for the sorptions of toxic As and Se oxyanions (AsO_3^{3-}, AsO_4^{3-}, SeO_3^{2-}, SeO_4^{2-}) can be prepared. For the experiments different pure adsorbents, two kaolins from West Bohemia (Horní Bříza, Sedlec Ia) containing predominantly kaolinite, muscovite and quartz, bentonite (Riedel de Haën, Germany) with the majority of montmorillonite and the Ca-rich montmorillonite (Arizona, USA), were used. The increase of the sorption capacities of modified clays was observed during the adsorption of AsO_3^{3-}, AsO_4^{3-}, SeO_3^{2-}, SeO_4^{2-} from model aqueous solutions. The initial concentration of As and Se was about 40 mg/L. The sorption processes ran from 24 to 72 h for reaching the equilibrium state. The separation of arsenates (AsO_4^{3-}) and selenites (SeO_3^{2-}) ions on the clay minerals modified with Fe^{3+}/Al^{3+} ions proceeded effectively (more than 90 % removal). The efficiency of As and Se sorption on the samples treated with Fe^{2+} ions varied in the range from 90 – 99 %. In the case of arsenites (AsO_3^{3-}) and selenates (SeO_4^{2-}) ions the treatment with Al^{3+} ions has been estimated as the most effective method because 50 - 99 % of sorption effectivity was achieved. Leaching tests demonstrated a possible release of adsorbed anions in the range from 0.3 to 10 %.

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LAYERED DOUBLE HYDROXIDES INTERCALATED WITH ORGANIC ANIONS AND THEIR APPLICATION IN PREPARATION OF LDH/POLYMER NANOCOMPOSITES

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The intercalation of 4-vinylbenzoic acid, acrylic acid and its derivatives, namely methacrylic acid, 2-acrylamido-2-methyl-1-propansulfonic acid (AMPS), and 4,4'-azobis(4-cyanopentanoic) acid into Mg-Al and Zn-Al layered double hydroxides (LDHs) was studied. The coprecipitated and hydrothermally treated Mg-Al-NO_3 and Zn-Al-NO_3 hydrotalcite-like compounds with M^{II}/Al molar ratio of 2 were used as precursors. The anion exchange and rehydration of calcined precursors were applied for the intercalation. The anion exchange appeared to be the most effective method; all examined organic anions were intercalated succesfully by this way both in Mg-Al and Zn-Al host structures. The intercalated LDHs were used as comonomer and initiator for preparation of LDH/polymethacrylate nanocomposites. Polybutyl methacrylate-modified LDH hybrid latexes were prepared by *in situ* emulsion polymerization. The LDHs intercalated with organic anions were dispersed in aqueous phase and then the emulsion polymerization was performed in the presence of inorganic (nano)particles. Nanostructured hybrid materials containing a low amount (1 wt.%) of inorganic nanofiller were obtained; particles with size of 65 – 80 nm were detected by light scattering analysis. Furthermore, a hydrophobization of LDH nanofillers was tested in order to facilitate their dispersion in the polymer matrix. After addition of the Mg-Al LDH intercalated with dodecylsulfate, nanocomposite with butylmethacrylate was prepared by *in situ* emulsion polymerization. A minor part of AMPS anions was incorporated into solid during anion exchange in the AMPS-containig solution, when LDH-dodecylsulfate intercalate was used as the precursor; a slight decrase of LDH basal spacing was observed in powder XRD patterns. Using LDH dodecylsulfate intercalate modified with AMPS, the nanocomposites were obtained by solution polymerization in 1-methyl-2-pyrrolidone and by suspension polymerization in ethanol. It was found that hydrotalcite-like compounds modified with organic acids are promising materials for preparation of LDH/polymethacrylate nanocomposites and the hydrophobization of intercalated LDHs improves their dispersion in polymethacrylate matrix.

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FORMATION OF MIXED OXIDES DURING THERMAL TREATMENT OF Co-M^{III} LAYERED DOUBLE HYDROXIDES (M^{III} = Al AND/OR Fe)

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Layered double hydroxides (LDHs) are often used as precursors for preparation of mixed oxide catalysts and therefore it is useful to know in detail their thermal behavior. The Co-Al, Co-Fe-Al, and Co-Fe LDHs with Co:Fe:Al molar ratio of 4:0:2, 4:1:1, and 4:2:0, respectively, were prepared by coprecipitation of corresponding nitrate solutions. Thermal decomposition of prepared LDHs and phase transformations of related mixed oxides were studied using thermal analysis (TG/DTA/EGA), powder X-ray diffraction including high-temperature measurements and Raman spectroscopy. The prepared LDHs were decomposed at about 200 – 260 °C; the thermal stability decreased with increasing Fe content. The thermal decomposition of LDHs resulted in a formation of rather amorphous spinel-like mixed oxides; their gradual crystallization with increasing calcination temperature was observed. Raman spectroscopy indicated a segregation of Co-rich spinel, i.e., Co_3O_4 or slightly Al-doped one, in Co-Al samples obtained at lower temperatures (300 – 600 °C). A recrystallization of primary Co-rich spinel accompanied by incorporation of Al into the spinel lattice at higher temperatures can be considered. In the Co-Fe samples, a segregation of two spinel-like phases, Co_3O_4 and $CoFe_2O_4$, was observed by XRD at temperatures higher than 600 °C. The Co-Fe-Al calcination products contained a Co-Fe-Al spinel only, whose lattice parameter gradually increased with increasing calcination temperature. A new band at 696 cm^{-1} was found in the Raman spectra of the samples obtained at 500 – 1000 °C; this band was ascribed to the presence of Al^{3+} cations in the spinel lattice. It can be concluded that a Co-rich spinel of Co_3O_4 type is likely formed as a primary crystalline phase after Co-Fe-Al LDH thermal decomposition; Fe and Al cations from the amorphous components are gradually incorporated into the spinel lattice at higher calcination temperatures. The crystallization of Co-Fe-Al spinel takes place up to 1000 °C. At high calcination temperatures (1100 °C), a partial decomposition of the Co-Fe-Al spinel to form Co_3O_4 and CoO was detected by XRD. A formation of CoO to the detriment of spinels was observed at high temperatures in all examined samples.

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Poster

LAYERED DOUBLE HYDROXIDES INTERCALATED WITH ORGANIC ANIONS: MOLECULAR MODELLING COMBINED WITH EXPERIMENTS

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Various types of layered double hydroxides (Mg_2-Al, Mg_3-Al and Zn_2-Al LDH) intercalated with organic anions (benzoate, p-methylbenzoate, p-bromobenzoate anions and (5,10,15,20-tetrakis(4-sulfonatophenyl)porphyrin anions – TPPS anions) were prepared and experimentally characterized by X-ray diffraction, thermogravimetry measurements and chemical analysis. Molecular modelling (molecular mechanics and classical molecular dynamics) combined with X-ray diffraction was used to calculate the interlayer arrangements of LDHs. The calculations were done in *Cerius*^{2} modelling environment. Various arrangements of the guest anions and water molecules in the interlayer space were calculated and a relationship between the experimental and the calculated X-ray diffraction was investigated. In the case of LDH intercalated with benzoate and its derivatives a high correlation between the changes of the interlamellar arrangements and the X-ray diffraction was found. Slight changes in the interlayer arrangements (especially the location of interlayer water) lead to significant changes of the calculated X-ray diffraction pattern. It leads to a clear conclusion about the position of the guest anions (nearly perpendicular to LDH layers), their disorder and location of interlayer water (in-plane arrangement near the LDH layers). In the case of LDH intercalated with TPPS anions where the guest anions adopt a tilted orientation of 70° with respect to the LDH layers we did not observed any significant structure changes between the Mg_2-Al and Mg_3-Al/TPPS intercalates. We calculated models with various concentrations of TPPS anions in the interlayer space and we observed that interlayer distance does not nearly depend on the concentration of TPPS anions in the interlayer space and the interlayer distance keeps the value of (22.1±0.4) Å.

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NANOCOMPOSITES POLYMER–LAYERED SILICATE

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In recent years, considerable attention has been paid to nanocomposites (NC) based on the system polymer-layered silicate. These NC proved to be exceptionally promising lightweight and easily processable engineering materials with high stiffness, strength and heat resistance, as well as good flame retardance and gas/liquid barrier properties.

Our research activity is focused on the preparation of polyesteramides (PEA) nanocomposites. PEAs based on ε-caprolactam (CLA) and ε-caprolactone (CLO) belong to the biodegradable polymers. With increasing content of the ester units in the copolymer, its impact strength and drawability increase but the content of crystalline phase, melting temperature, and modulus decrease. The PEA modified to NC are expected to increase their modulus and hardness.

This contribution deals with the preparation of PEA NC by the exfoliation adsorption (preparation of NC from solution) and *in situ* polymerization techniques.

EXFOLIATION ADSORPTION: A series of polyesteramides prepared by an anionic copolymerization of CLA and CLO was used as a polymer matrix. Cloisite 30B and Cloisite 25A were used as fillers. Exfoliation/intercalation of the filler within the polymer matrix depends on mutual interactions of the components, i.e., polymer, solvent and organophilized montmorillonite. The interactions silicate-solvent and polymer-solvent were studied by measuring a swelling factor, an interlayer distance, and a limiting viscosity number. Only a few of the large number of PEA-solvent-montmorillonite combinations lead to the NC having fully or partially exfoliated structure.

IN SITU POLYMERIZATION: Two procedures of PEA NC preparation have been used: (*i*) anionic polymerization of CLA in the presence of poly (ε-caprolactone) NC with high content of exfoliated filler and (*ii*) a similar procedure to the preparation of NC by the anionic polymerization of CLA in the presence of sodium montmorillonite preswelled in water. Dispersion of MMT in CLA was obtained after removing of water and part of CLA. At the final stage polymerization feed was obtained after introduction of CLO and polymerization initiator.

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Poster

Cd^{2+}-VERMICULITE AS A PRECURSOR FOR CdS PARTICLES SYNTHESIS

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Cadmium sulfide (CdS) in a form of nanoparticles belongs to a family of semiconductors with applicability in different fields of industry. Light-emitting diodes, solar cells, lasers, thin films transistors and other optoelectronic devices are probably the most intensively studied applications where CdS is utilized. The band gap of CdS - 2.41 eV, ensure that photoactivity of CdS is generated after the irradiation in visible light region, in comparison with probably the most studied photocatalyst – TiO_2, which broader band gap (anatase form – 3.2 eV) requires the irradiation in UV region to produce excitons. This significant feature also predetermines CdS as a strong candidate e.g. for hydrogen production via water photosplitting in solar cells.

The CdS nanoparticles can be produced in their pure form or in the form of thin layers deposited on the suitable substrate. The synthesis routes include different techniques like sonochemical route to fabricate one-dimensional CdS or preparation of CdS particles in self-reproducing reverse micelles or preparation of CdS thin films by the chemical bath deposition.

Vermiculite group belongs to 2:1 planar hydrous phyllosilicates with hydrated exchangeable cations in the interlayer. An isomorphous substitution of metal cations with a similar size and a lower valence, such as Si^{4+} - Al^{3+}, Al^{3+} - Mg^{2+} creates a net negative charge of 2:1 layers. The positive charge deficiency is compensated by exchangeable cations K^+, Na^+, Ca^{2+}, Mg^{2+} located in the interlayer space between the parallel 2:1 layers. Vermiculites show layer charge $0.6<x<0.9$ eq/ $(Si, Al)_4\,O_{10}$, arising mostly from the tetrahedral substitution of Al^{3+} for Si^{4+}.

In our work we present preparation of Cd^{2+}-vermiculite precursor from natural Mg-vermiculite. Such prepared precursor was subsequently used for synthesis of the CdS/vermiculite composite. Scanning electron microscopy and X-ray powder diffraction analysis were used for characterization of the prepared samples.

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Poster

USAGE OF CLAY MINERALS IN THE TECHNOLOGY FOR THE REFINING OF DRINKING WATER

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In the conditions of accelerating development in various fields of industry, a reasonable and complex usage of water resources and an introduction of methods for water purification, especially for drinking purposes, present a very serious task.

For this task to be fulfilled, it is reasonable to use natural clay minerals, in particular, glauconite which has low cost price, high adsorptive capacities, and is easily, regeneratable, modificable, and recyclable.

The research on purification of drinking water from heavy metals and radionuclides by glauconite has been conducted. Glauconite efficiently purifies drinking water from copper, zinc, lead and cadmium. It has been proved that glauconite has adsorptive capacities as well as selectivity. The author also found a mechanism of adsorptive processes. Glauconite adsorbs Cs^{137} and Sr^{90} and increases ecological safety of drinking water.

The results of the research have provided the possibility to determine optimal technological parameters for the purification of drinking water from heavy metals, radionuclides and to introduce these parameters in the industrial production of drinking water.

Determination of heavy metals and radionuclides in purified drinking water has been performed in accordance with state standard methodologies.

The used glauconite has been further utilized as a filler for building materials.

Poster

MOLECULAR MODELLING OF THE CATIONIC SURFACTANTS ON MONTMORILLONITE

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An arrangement of cetylpyridinium (CP) cations and cetyltrimethylammonium (CTA) cations on Na-montmorillonite (MMT) surface was studied. The molecular modelling was calculated in Cerius^2 and Material Studio modelling environment. The set of initial models were built above 2*a* x 2*b* surface unit with different amount of the CP cations (two CP cations and 1 Na cation compensate negative layer charge, in some cases, higher amount of CP cations was compensated by Cl^- anions), 55 water molecules and 17 ethanol molecules in different mutual positions and orientations to simulate all possible state space of the investigated models. The same molecular modelling methods were done for the CTA cations in 55 water and 17 ethanol molecules. The resultant models with minimum energy are usually characterized with the cation head groups adjacent to the MMT surface. The arrangement of the CP and CTA cations above the MMT surface is quite different but it relates to experimental results obtained from the adsorption (Praus, Pospíšil, 2008). The CP cations are immersed in water-ethanol bulk because of the sublimation energy values for whole system is lower and the CP cations are surrounded by bulk. In opposite of this, the CTA cations are not immersed in the water-ethanol bulk. The sublimation energy values are lower for systems where the CTA cations are more closely packed to the MMT surface than CP cations. This leads to a conclusion that interactions between CP and MMT are weaker than between CTA and MMT.

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SORPTION OF ANIONIC AND NONIONIC SURFACTANTS FROM AQUEOUS SOLUTIONS ON ORGANOSUBSTITUTED LAYER SILICATES WITH RIGID STRUCTURAL CELL

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Layer silicates possessing rigid structural cell (hydromica from Cherkasy deposit and kaolinite from Gluchov deposit, Ukraine) with the surface modified by cationic surfactant, cetyl pyridinium bromide (CPB), were studied to elucidate the influence of the modification on their sorption capacity. In particular, the adsorption isotherms of anionic surfactant, sodium dodecyl sulphate (SDDS) and nonionic surfactant, neonol APh_9-10, from aqueous solutions on the studied sorbents were measured. It was shown that the modification results in a drastic increase of both the adsorption and the extraction degree of the sorbates in a low sorbates concentration range as compared with the values characteristic for natural minerals, and also to the significant increase in the limiting adsorbed amount of APh_9-10, and, to the lower extent, to the increase in the limiting adsorbed amount of SDDS.

To explain the adsorption mechanisms, the interaction of these surfactants in the binary mixtures was studied. In particular, the Du Noüy method was used to measure the surface tension isotherms of individual solutions of surfactants and binary mixtures APh_9-10/CPB and SDDS/CPB; the results were processed by Rubing-Rosen approach. The composition of mixed micelles and adsorption layers at the solution/air interface, the activity coefficients and the surfactants interaction parameter β were calculated. In the APh_9-10/CPB system some excessive interaction between the components, the enrichment of mixed adsorbed layer, and (to the lower extent) the enrichment of micelles by the non-ionic surfactant were observed. For the SDDS/CPB system, the formation of a new compound (catanionic surfactant) was shown to take place, resulting in high negative β values. From the comparative analysis of sorption isotherms and calculated characteristics of binary solutions of the surfactants, the conclusions about the interaction of these surfactants with solid surface during the adsorption were drawn. In particular, taking into account the parameter β values calculated for binary mixtures, the differences were explained in the mechanism and intensity of the SDDS interaction with various active centres of the modified clay surface. It was shown that for the interaction of APh_9-10 with CPB in the concentration range below CMC, the molar fraction of APh_9-10 in mixed adsorption layers at the interface for the APh_9-10/CPB binary mixture (0.73) is quite equal to the fraction (0.75) at the interface for the system in which the clay mineral modified by CPB is brought into contact with the APh_9-10 solution.

The results of the study indicate that the proposed modified sorbents based on clay minerals can be used for the extraction of organic pollutants, in particular surfactants, from waste waters.

Poster

CLAY MINERALS AFTER TREATMENT IN SILVER NITRATE SOLUTION

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The 2:1 clay minerals have four basic important characteristics: (1) their layers, may be called the substrate, bear a negative charge due to the ionic substitution in the structure; (2) the exchangeable cations between the layers (Brønsted acid sites) compensate the negative charge of the layer and influence surface acidity; (3) some cations such as Al^{3+}, Fe^{3+}, normally occupying octahedra, remain on the crystal edges (Lewis acid sites), where they coordinate water molecules; (4) neutral inorganic molecules including water, which occur between the layers are associated with the interlayer cation and the layer surface. The clay minerals in aqueous media offer possibility to prepare particles of a few nanometers size on their surfaces and within lamellae and interlayer space. Physical and mechanical properties of nanosize particles differ from those of macroscopic material. The negative charge x on the clay silicate layers, in case of surface utilization as matrix, can serve for the reduction and growth of e.g. silver nanoparticles.

The testing of montmorillonite (M), x = 0.48 eq/ $(Si,Al)_4 O_{10}$ and vermiculite (V), x = 0.73 eq/ $(Si,Al)_4 O_{10}$ with distinct potential, as carriers for functional silver, is performed. The metal precursor was aqueous solutions of silver nitrate (0.01 and 0.1 mol dm^{-3} $AgNO_3$). The M and V were stirred in nitrate solutions for 24 h and repeated twice. The suspension was centrifuged for 30 min at 3000 rpm and gently washed with demineralized water to remove free water-soluble silver nitrate and dried subsequently.

Based on the X-ray diffraction patterns we can see that montmorillonite after both nitrate treatments becomes slightly exfoliated. Vermiculite after the second nitrate treatment, according to the multiple value of the basal reflection d_{001}, rebuilds layered interstratified structure. As a consequence of the nitrate solution treatment a more intensive cation leaching from montmorillonite than from vermiculite was determined. The results from elementary analysis and electron microscopy showed differently reduced Ag content and distribution on the M and V layer surface. Antibacterial activity of the Ag-M and Ag-V was tested on Escherichia coli and Enterococcus faecium.

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Poster

MOLECULAR MODELLING OF NATURAL AND INTERCALATED MONTMORILLONITES ADSORBING PHENOLE AND ANILINE

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Montmorillonites (MMT) due to their enormous surface area and expansible interlayer space can be used as effective adsorbents for various organic substances. The properties of intercalated forms of MMT could be widely modified (originally hydrophilic surface turns into hydrophobic after sorption of organic compounds). The adsorption of phenol and aniline, which belong to hazardous pollutants occurring in effluents, was studied. Three intercalated samples were prepared and together with the sample of pure MMT were used as adsorbents. Modified montmorillonites were prepared by the intercalation and the sorption of three quaternary ammonium salts (cetyltrimethylammonium bromide, tetramethylammonium chloride and tetrabutylammonium iodide) into the interlayer space and on the surface of montmorillonite.

The experimental results (adsorption curves, X-ray diffraction) revealed that intercalated montmorillonites adsorbed phenol much more readily than the pure sample (even though phenol was apparently more adsorbed into the interlayer space in the case of pure MMT). On the contrary, aniline was adsorbed much more by pure montmorillonite. This behaviour can have following explanation: aniline is adsorbed by the ion-exchange interaction as the charge compensating anilinium cation in place of the original alkaline ions. After a saturation of the anilinium cations in the interlayer and on the surface other neutral aniline molecules can be adsorbed. The surface models of these structures were studied in detail by techniques of molecular simulations (molecular mechanics and classical molecular dynamics) in Cerius^2/Material Studio modelling environment.

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Poster

THE INVESTIGATION OF ADSORPTION OF ORGANIC CHEMICALS BY CLAY MINERALS

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An understanding of the environmental transport, fate, and effect of organic chemicals became necessary from a health standpoint due to the toxicity of many of these chemicals. The transport and fate of these contaminants in ground water systems are found to be dependent upon the chemical or physical reactions including binding rate, capacity and strength of pollutants to soil particulates. The binding of contaminants may be through ion exchange reactions or adsorption onto soil particles. The sorption characteristics of contaminants are governed by material properties as well as environmental factors. The sorption of organic chemicals to solid phases is a key reaction in determining the environmental transport. Knowledge of the sorption coefficients for nonpolar contaminants on minerals and how they relate to both sorbate and sorbent physical and chemical properties is a requirement for predicting the contaminant movement.

The purpose of this study was to examine the possibility of predicting the sorption on minerals based on knowledge of clay mineral chemistry and sorption coefficient measurements on mineral sorbents. The experiments were conducted to determine the magnitude of the sorption coefficients on clay minerals that are components of aquifer materials. The results of batch measurements of the equilibrium sorption coefficients (Kd) will be used to predict the mobility of a volatile organic compound in clay materials (kaolinite; illite, bentonite from Ukraine).

Three chlorine-organic sorbates were used as surface probes to observe their sorption trends on the model mineral surfaces. Experiments were conducted to determine the magnitude of the sorption coefficients on clay minerals that are components of aquifer materials. Time and pH were varied over possible environmental ranges to observe effects on the sorption. Variation in the observed Kd for the clays with pH may be attributed to a number of factors. Surface area may be casually associated with sorption, but it is not the primary factor in determining Kd values.

An understanding of pollutant transport by groundwater is necessary to select a cleanup strategy at sites of high contaminant concentration and to predict the long-term distribution and fate of the contaminants that cannot be recovered by the cleanup procedure.

Theme D

Utilization of Metakaolin in Geopolymers and Building Materials

STRUCTURE OF VARIOUS GEOPOLYMER SYSTEMS AS STUDIED BY SOLID-STATE NMR SPECTROSCOPY

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Geopolymers that belong to the family of aluminosilicate ceramics synthesized at room temperature have attracted commercial and scientific attention for their superior mechanical properties and relatively low production cost for a long time. Current trends in geopolymer chemistry are to develop new types of geopolymer systems at low energy requirements but still possessing good mechanical properties. Consequently, these geopolymer systems must be considered as very complicated nanocomposites with variable structure, composition and morphology that are difficult to define. Ideally geopolymers should have non-crystalline structure, which keeps molecular water inside 3D framework. However, during the time, these materials can undergo gradual transformation to zeolite structures at some circumstances. These transformations are accompanied by loss of water and mechanical properties. The understanding and disclosure of the fine relations between the structure, processing and post-processing of various types of geopolymer requires an application of carefully designed and optimized advanced solid-state characterization techniques.

In general solid-state NMR spectroscopy is an excellent method providing information about the microstructure of these systems without requirement of long-range ordering. In our contribution we would like to present a large and representative set of NMR spectra of geopolymer systems differing in the composition of the starting reaction mixtures, processing and post-processing procedures (hydrothermal treatment at variable temperature, pressure, humidity and time). It will be demonstrated that for each type of geopolymer systems and the processing and post-processing procedures another type of NMR spectra has optimum information capability. Further it will be demonstrated that even fine changes in the local structure of geopolymers can be nicely probed by the advanced two-dimensional solid-sate NMR experiments. *i*) Localization of water molecules and OH groups can be probed by carefully optimized cross-polarization experiments between ^{1}H and ^{29}Si, ^{27}Al and ^{23}Na nuclei. *ii*) Various $Al^{-}O_4^{5-}$ and Na^+ sites can be distinguished by triple-quantum ^{27}Al and ^{23}Na MAS NMR experiments combined with the polarization transfer from protons. *iii*) And finally, the connectivity of Al and Si units and the additional resolution of diverse structural motifs in the geopolymer systems can be achieved by double- and triple-resonance experiments based on the polarization transfer from ^{27}Al to ^{29}Si nuclei.

FIXATION OF ARSENIC AND SELENIUM IN GEOPOLYMERS AFTER THEIR SORPTION ON MODIFIED KAOLIN

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In recent years geopolymerization has represented the possibility of effective stabilization and immobilization of toxic materials. The present work studies the fixation of As(V), As(III) and Se(IV) in geopolymeric matrix (GP). For preparation of GP two types of different calcined kaolins (metakaolins) from West Bohemia, water glass and NaOH solution were used. Kaolins were calcinated at 550 °C or 750 °C for 6 hours. As/Se was added in the solution (0.5 – 22 g/L ≈ 0.04 – 1.3 mg/g GP), or in the solid phase (1 – 1.2 mg/g GP). For the preparation of GP with As/Se in solid phase, As/Se oxyanions were adsorbed on Fe, Al or Mn-modified metakaolin. The mixture of these ingredients was aged for 7 - 133 days at ambient conditions. Afterwards, some of these samples were heated in the air at 140 °C for 24 h. Various experimental and analytical techniques were used in this investigation, i.e. compressive strength test, electron microscopy, XRF, XRD and IR spectroscopy. The results showed that As/Se stability increased with the heating process and aging time. It seems that As added in the solid phase proved the higher stability in GP matrix in comparison with As in solution. The both possibilities of Se addition were comparable for its stabilization. The best results were achieved for GP prepared from Fe-modified metakaolin after heating process, i.e. 108 MPa and 4 % (5.2 mg/L) As in leach. IR spectra of prepared GP demonstrated the presence of As with absorption peaks varying from 816 cm^{-1} to 930 cm^{-1}. For heating processes the reduction of Si-OH and Al-OH bands was found.

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COMPRESSIVE STRENGTHS OF AMBIENT AGED AND HYDROTHERMALLY TREATED GEOPOLYMERS PREPARED FROM METAKAOLINITE – (Na,K) WATER GLASS - NaOH (KOH) – WATER SYSTEM

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Geopolymers based on calcined Sedlec Ia kaolin (550 °C/6 h) were prepared from the (0.5- 1.1) Na_2O (K_2O) – $4SiO_2$ – Al_2O_3 – 18.5 H_2O system. Truncated pyramidal shaped samples of geopolymers with the size of 25x25 mm and 20x20 mm were prepared at ambient ageing for 7 days and 90 days respectively and were subjected to the measuring of the compressive strength. Some of 7-days geopolymer samples were hydrothermally treated for 24 hours at 180 °C. The compressive strengths of all samples after 90 days of ambient ageing were improved in comparison with 7-days samples. The hydrothermal treatment of 7-days K-geopolymers had only a negligible effect on the compressive strengths compared to 90-day samples and Na-geopolymers. The values of the compressive strengths after the hydrothermal treatment have fallen considerably in the geopolymers with the higher Na/Al ratio (1 and 1.1). The instability of geopolymers is caused by the formation of zeolite phases in matrix. K-system was very stable in the whole range of monitored composition, while Na-system was stable only in the range of Na/Al = 0.5-0.9 ratio. Except the mentioned ratio, the geopolymeric bodies did not attain the sufficient compressive strengths due to the presence of many open interconnected voids, which were confirmed by the water absorption exceeding 25 %. On the other hand it was found that some impurities can affect the growth of the compressive strengths of ambient aged geopolymers. Frost and wet-dry resistance, as well as water absorption was improved by addition of impurities (Fe_2O_3, TiO_2, etc.), which are commonly occurring in soils and rocks. The compressive strength of K-specimens increased by 5 % after 25 cycles of freeze-thawing and by 20 % after 25 cycles of wet-dry resistance tests. The behaviour of Na-specimens was similar only in wet-dry test; the compressive strength rose by 6.5 %. The chemical resistance to the thawing and aggressive environment, however, did not improve.

CHARACTERIZATION AND REPRODUCTION OF ARCHAEOLOGICAL HYDRAULIC MORTARS BASED ON LIME AND METAKAOLIN

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In the past (before the half of the 18th century), because of low calcination temperatures and the hydration technique which slaked all the fired material, hydraulic mortars could be obtained only by using lime and natural or artificial aggregate with pozzolana properties. Generally the ancient hydraulic mortars were prepared with lime and “cocciopesto” (bricks reduced to fragments and dust), or/and lime and pozzolana. Rarely historical hydraulic mortars composed of lime, metakaolin and sand can be identified. During the setting phase the lime can react with the metakaolin, obtained by the firing of the kaolin at about 600 °C, giving rise to hydrated calcium aluminium silicates, responsible for the hydraulic properties of these mortars. These unusual and sophisticated mortars are present in localized areas and in precise periods, and often show high mechanical properties.

Last year our research group characterized, by means of different analytical methodologies (OM, XRD, DTA-TGA and SEM-EDS), mortars and plasters, with a high cohesion degree and an excellent conservation state, coming from the production sector of the Roman archaeological site of Pietratonda in Southern Tuscany (Italy). All the samples, composed of lime and siliceous sand, revealed hydraulic properties. Neither pozzolana granules nor “cocciopesto” were identified. In addition, SEM-EDS analyses highlighted the presence of neo-formation crystals of hydrated calcium aluminium silicates (HCAS) in different point of the binder. These crystals are responsible for the hydraulic properties of the mortars. Very probably these neo-formed crystals are due to an addition of metakaolin in the mortars and to its reaction, during the setting phase, with the lime. In fact, several outcrops of kaolin are present in the area of Pietratonda, and two kilns were discovered in the archaeological site.

In order to reproduce these mortars, which can be used in restoration interventions or in the field of the bio-building (natural components, negligible concentrations of soluble salts, good compatibility with the original materials, high mechanical properties and excellent durability in humid environment), different metakaolin mortars were prepared with lime and metakaolin obtained by the firing of kaolin coming from outcrops in Pietratonda area. We obtained hydraulic mortars in which we determined HCAS crystals with morphology and composition similar to those observed in the historical mortars of Pietratonda.

Poster

INFLUENCE OF CHEMICAL AND MINERALOGICAL COMPOSITION OF METAKAOLIN ON MORTAR CHARACTERISTICS

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Three different metakaolins processed by a Portuguese factory were studied aiming to assess the influence of chemical and mineralogical composition of metakaolin on mortar characteristics. Mineralogical analysis of both the fine (<63 μm, random-oriented powder samples) and clay (< 2 μm, random-oriented powder samples and oriented-aggregates) fractions was carried out using a Philips X'Pert Pro X-Ray Diffractometer. Chemical composition (major elements) was analyzed using a Philips PW 1400 X-Ray Fluorescence Spectrometer. Crystallochemical analysis was performed by Scanning Electron Microscope (SEM). Mineralogical composition reveals some significant differences between the studied samples, in both analyzed fractions; concerning fine fractions, metakaolin 1 is the one richer in quartz whereas 2 is richer in kaolinitic minerals; concerning clay fractions, metakaolin 1 shows clear evidences of halloysite and disordered kaolinite whereas 3 shows a more ordered kaolinite as well as some evidences of dickite, metakaolin 2 showing an intermediate composition. From a chemical point of view, siliceous content is always high (around 60 %) as well as Al_2O_3 content (around 30 %), but once again the three samples show some clear differences; metakaolin 2 is the one richer in Al_2O_3 (showing also a sligthly higher Loss on Ignition value) and poorer in sodium and potassium. Metakaolin from the three different batches was incorporated in lime mortars in order to produce a pozzolanic reaction and hence enable them to harden in high relative humidity conditions or when access to CO_2 is limited, as in the case of mortars supporting glazed tiles. The addition of pozzolanic materials increases the mechanical characteristics of these mortars and contributes towards a higher durability. Mortars with the same volumetric ratio were tested in terms of flexural and compressive strength at ages of 28 and 90 days, and the Modulus of Elasticity was determined. Although all metakaolin additions improved mechanical strength of the lime mortars, a clear difference in mortar strength was detected with the use of metakaolin 3 in relation to the other metakaolin mortars. A relation between the mechanical strength achieved by lime mortars with the addition of metakaolin from different batches and metakaolin mineralogic and chemical composition was assessed. The knowledge of the influence of the metakaolin composition on these mortars's strength is an important step to improve their performance and enlarge their application field.

Conclusion

Some History in One Poster

50 YEARS OF CLAY CONFERENCES IN CZECHOSLOVAKIA AND CZECH REPUBLIC

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Czech National Clay Group (CNCG, former Czechoslovak NCG) has a remarkable history among the related Groups in the part of Europe once being behind the Iron Curtain. The activity of CNCG was always inspiring to better research production, positive criticism, interdisciplinary co-operation, popularization of our science and international collaboration. In last 50 years argillologists in Czechoslovakia or Czech Republic organized 17 Clay Conferences (from 1958 to 2004) and edited 17 volumes of the Clay Conference Proceedings plus one Euroclay volume (1983) and 38 numbers of CNCG Newsletter *Informátor* starting in 1991. They realized also a large number of seminars taking place twice a year.

The actual research infrastructure of argillology in Czech Republic can be deduced from addresses of the authors publishing in the Book of Abstracts of the 18th Clay Conference. The major factors contributing to the argillological research activity in this country are as follows: 1) a goalseeking concentration on the maintaining the position of the argillology developed in Czech Lands in the second half of the 20th century; 2) a friendly support of the co-operation among the existing research institutions; 3) a continuation of the organizing Clay Conferences open to experts from other countries; 4) edition of Book of Abstracts and Conference Proceedings; 5) edition of Newsletter *Informátor* with the following sections: a) Editor´s Word; b) summaries of the spring or autumn seminar lectures; c) transmission of the most important foreign research activities; d) seminars and excursions in related sciences; e) papers *in memoriam*; f) new CNCG members; g) new books; h) other scientific conferences and possible-co-operation; i) actualities. Argillology in Czech Republic inherited productive and creative research environment, probably best shown in the essay “A foolhardy and perhaps enlighting reflection on my bibliography” published in Czech by Professor Jiří Konta to his 85th birthday anniversary (*Informátor* 2007, No. 35).

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