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New Methods and New Types of Functionalised Nanocomposites Intended for the Ecological Depollution of Waters

Nicoleta Petrea, Petrișor Zamora Iordache,
Rodica Mihaela Lungu, Ioan Safta, Razvan Petre and Andrada Pretorian
*Scientific Research Center for CBRN Defense and Ecology
Romania*

1. Introduction

Nanotechnology as techniques for the removal of organic pollutants implies the use of certain types of nanostructures or nanostructured materials as a carrier host for the reticulation, encapsulation and degradation of pollutants. The main mechanisms through which nanocomposite structures separate or destroy organic pollutants are those of the catalytic oxidation, reduction, sorption or reticulation types (Lu, Zhao & Wan, 2010, Choi et al., 2009). Most materials and methods which are based on nanotechnologies have high selectivity regarding specific organic pollutants, and their applicability rely on the economic factor. The limited spectrum of separated or degraded pollutants, the decontamination time and the lack of control over degradation products are key factors which limit the widespread use of most nanostructured materials in organic decontamination processes.

Nanotechnologies offer promising solutions to the depollution field, thanks to their remarkable properties: depollution process control ability, structural and functional modelling flexibility, with a view to improving depollution parameters, high specific surface, etc. In addition, nanostructured materials offer solutions for the obtaining of new ecological depollution biodegradable materials. Microstructured and nanomembranary materials, as well as functionalized nanostructured materials have the most promising applicative potential. Functionalized nanomaterials are carriers of chemical functions capable of reticulating, incorporating and removing organic pollutants. Functionalized composite materials have depollution properties which are similar to those of functionalized materials. As compared to functionalized nanomaterials, composites materials have several possibilities of structural and functional modelling, bearing direct effect on the yield, efficiency and spectrum of reticulated pollutants. Ion exchangers or natural adsorbents (zeolites, synthetic resins, functionalized polymers, etc.) (Cheremisinoff, 2002; Evanghelou 1998) have organic depollution properties, which are similar to those of functionalized composites. Degradation residues of organic pollutants, resulting from depollution processes, as well as from secondary pollution processes caused by the latter are one of the decisive factors that limit current depollution technologies (Rosenfeld & Feng, 2011; Bayliss & Langley, 2003). Most depollution waste fractions have lower molecular weights, as compared to the initial pollutants, which can induce carcinogenic effects on humans or

changes on the structural and functional properties of the biodiversity and the environment. Membrane nanostructured materials and functionalized composites have pronounced limiting effects over the dispersion effects of secondary degradation pollutants by their retention, encapsulation and separation. As a consequence of the major social and economic implications (environmental protection, natural resource conservation, medicine, nanomedicine), organic depollution has imposed itself as a priority research field, as a source which generates solutions and implementable technologies. This chapter presents the experimental results acquired as a result of the obtaining and testing of new biodegradable functionalized composite materials, intended for the removal of a wide range of organic pollutants. The composites we obtained were tested on four categories of polluted water, coming from milk processing industry, medicine industry, the obtaining and processing of polymers and from the sewage waters in the city of Bucharest.

2. Functionality relations between environmental elements and pollutants

The environment can be defined as the manifestation of dependence and functionality relations established between physicochemical and biological structures characteristics of soil, water and air, as its fundamental structural macroentities (Tolgyessy, 1993). Overall, the environment represents a complex and dynamic structure, on the evolutionary process, due to the way and type of interaction established between its structural elements, and material and energy mediation vectors (Fig. 1). Ecosystems represent manifestations of the local distribution manner of basic structural entities of the environment. Ecosystems represent organized systems, well defined in relation to their physical, chemical and biological structure of their structural elements, among which are established structural and functional relations of their own (Socolow et al., 1994). Each set of functions is characteristic for a particular ecosystem, reflecting its biochemical structure, mediation and transport vectors, the dynamics of internal evolution, as well as the rate, role and functions of each structural component. Any changes to global environmental factors or local macrostructural distribution of an ecosystem may irreversibly affect the structural and functional evolution. From this perspective, ecosystems can be defined as stable phases of local equilibrium, established between the biochemical structure, compositional structure and functional structure of their macro- and microcomponents.

Transport and mediation vectors (TMV) influence local and global dynamics of transformation and transport, established between the local structural components of the environment and its ecosystems. The main TMV, which form the evolution and transformation of the environment and of its ecosystems are air, water and climatic factors. The distribution and local physical and chemical characteristics of TMV model the distribution and evolution of biodiversity models, as well as the distribution and evolution of the elements which form the composition of the soil, water, rocks, sediments and minerals. The modelling process leads to the establishment of relationships and dynamic biochemical balances, always subject to the evolutionary processes of transformation and structural and functional remodelling. The development and the establishment of equilibrium states within and between the local structures of the environment, reflect on going relationship of mass and energy transfer established at the level of microstructured elements, with TMV as mediating factor. The morphochemical and morphofunctional structuring manner of the soil influence pollutant absorption, modelling and conditioning applicative technologies and depollution resources (Reddy, 2010; Lal & Shukla, 2005).

Organic fractions resulting from degradation processes of biodiversity (humic and fulvic acids, organic derivatives and their macrocomplex, carbon, nitrogen, oxygen, water, etc.) constitute the foundation of chemical and biochemical transformation processes that model host ecosystems, and are factors which catalyse and support chemical and biochemical functionalities (Evangelou, 1998; Haider & Schäffer, 2009).

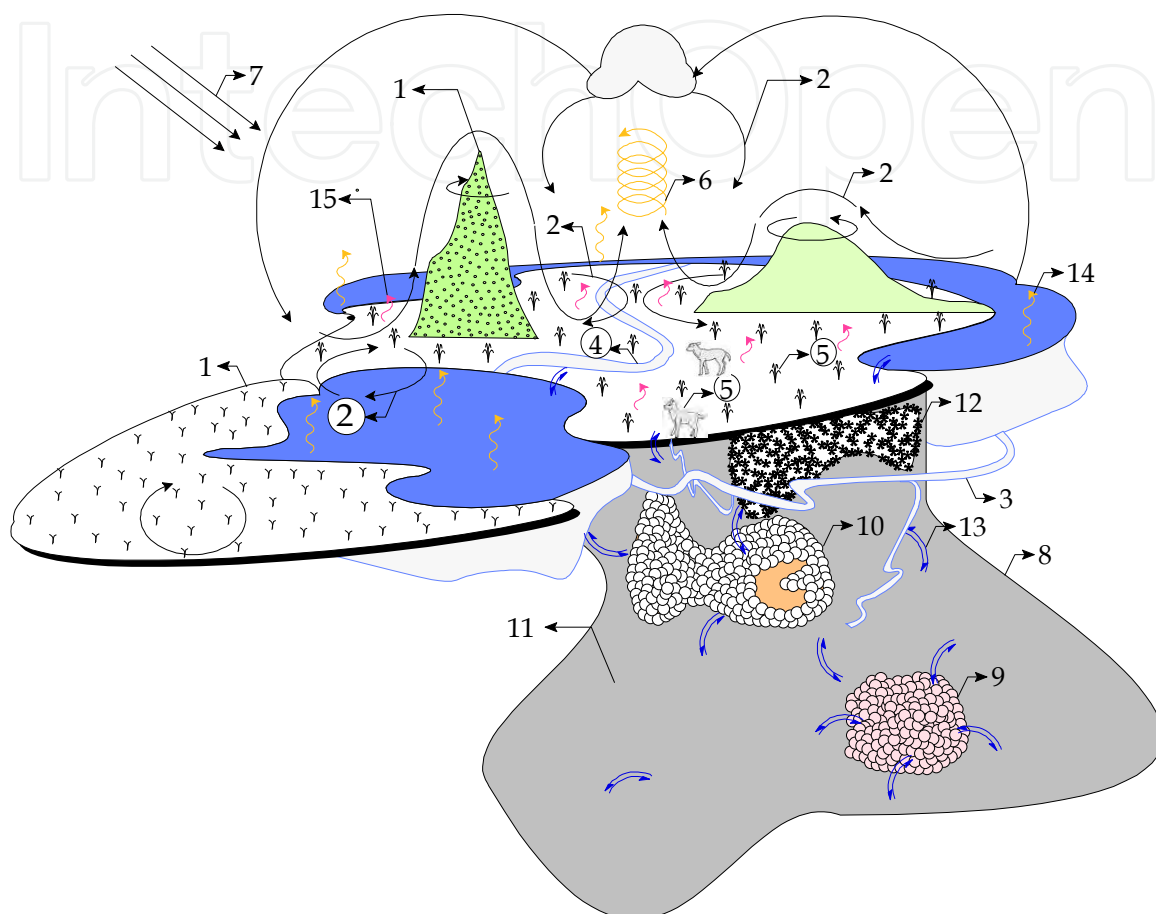


Fig. 1. Ecosystems and their structural and functional organization

(1 - surface form relief; 2 - flow of air and water vapour currents; 3 - underground water flows; 4 - surface water flows; 5 - biodiversity; 6 - energy fluxes; 7 - light; 8 - underground; 9, 10, 12 - underground mineral and rock distributions; 11 - other underground fine particulated matter; 13 - flow of matter and energy carried by fluids; 14 - sea water vapour; 15 - land water vapour)

Material and chemical structures resulting from natural processes of evolution are not a threat to biodiversity and the environment, as they support and model local exchange mechanisms and energy and material balance mechanisms. In terms of chemical functionality, the material structures resulting from natural processes of degradation can be considered inert structures, as they do not have the ability of modelling the morphochemical structure and the functions of flora, fauna and microorganisms of ecosystems. Pollutants can be defined as factors that induce sudden steps in the structural and functional evolution of an ecosystem, the biodiversity being the fastest element of the environment that responds and adapts to changes. The emergence, suppression or evolution of certain species or biological structures is conditioned by the relations established between the functionality

structure of the host ecosystem and the genetic profile specific to the targeted biostructures, as a structural conservation factor. Most dependence relations established between the functionality of ecosystems and the genetic profile of their biostructures are of the evolving type, in which the ecosystem, as a material and energy source, ensures the transition of available resources in higher biological forms of organization. The suppression of material and energy resources is equivalent to the suppression of the development and evolution of biodiversity. The factors that may suppress the dependency relations established between the functionality and the structure of the environment and the biodiversity hosted by it can be factors of organic and inorganic pollution, as well as natural equilibrium factors (air, light, temperature, humidity, etc.). These factors model and provide energetic and catalytic support, as well as the dynamics and complexity of structural and material organization forms of the soil, water and air. The dynamics and complexity of the structural and material organization forms of the soil, water and air constitute the structure that supports the entire evolution and the material and functional structuring of biodiversity. Most artificial chemical compounds are material structures with high functional and chemical reactivity, small molecular weight and high diffusion coefficients, which affect the morphochemical structure and the functionality of the environment biodiversity. For this reason, artificial chemical compounds are considered and act as poisons upon microorganisms or biochemical structures with nonexplicit biological functionality (amino acids, proteins etc.). The morphofunctional uniformity and the broad spectrum of natural, synthetic organic pollutants, or those resulting from industrial and domestic activities, contained in the surface waters are the main reasons that restrain the methods, materials and current organic depollution techniques. The constraints bearing upon functionalized nanostructured materials or nanostructured composites are due to the non-selective charging with chemical compounds, which are not toxic for the environment. The limitation of the type of functionality of nanostructured materials is the second major cause that limits the depollution processes, by separately constraining the range of pollutants. Considering the above mentioned statements, the most suitable retention methods of organic pollutants are the mixed ones, which use as active principles of pollutant separation: the mechanical filtration, chemical reticulation, reverse osmosis, degradation, encapsulation and controlled extraction of depollution products.

3. Urgent needs in the field of depollution and decontamination

Inorganic depollution involves relatively low complexity processes, related to organic depollution processes. In the field of inorganic depollution there are thoroughly-studied applicative depollution methods and technologies dedicated to each class of pollutants. Inorganic depollution can be considered as a controlled process, as pollutants may be eliminated in a controlled manner, directly in the stage of generation, limiting the dispersion and secondary contamination processes. In comparison with the organic pollution, the inorganic pollution has a much simpler complexity of the spectrum of pollutants contained in surface water, extending the possibilities of separation, storage, management, treatment and reassessment. Inorganic pollution represents a priority problem of depollution, which has a high complexity level, and whose resolution implies the utilization of some partial, limited applicative solutions. Both organic pollutants and the pollutants having a mixed morphochemical structure (of the organic - inorganic type) are compounds with high and average, slightly degradable reactivity. Most organometallic pollutants and of the

compounds having a mixed morphochemical structure (adsorbent inorganic matrices, host matrices, fibres, macro polymers, active suspensions, functionalized inorganic mixtures, etc.) come from food industry, medicine industry, polymer industry, wood processing industry, etc. The slightly degradable character of organic pollutants changes the organic pollution into a degenerative process that generates in time new types of degradation compounds. Most degradation compounds are toxic in relation to the environment, being able to influence, on long and short terms, the functionality and structure of the component elements of the environment. The accumulation and degradation processes are factors that support and amplify the secondary processes of pollution and biological contamination, by favouring the development and adaptation of some new biological structures, extraneous to the host ecosystems. Also, the biological pollution can influence, on long and short terms, the human health and chemical, biological and functional balance, established at the biodiversity level of the ecosystems, by favouring the uncontrollable evolution of the structure and functionality of the local and global environmental factors. By the nature of the sources, due to the complexity of industrial and domestic activities, as well as the needs and exigencies of the consumer society, huge quantities of residue result that require suitable management for collection, storage, reassessment and disposal. The identification of optimum solutions to residue management represents a priority field of depollution as a preventive and control factor. The main function of the waste management is that of controlling and limiting the pollution, by isolating and treating the resulted residue. Due to the exponential increment of the collected residue quantity, as well as to the financial and environmental implications, the depollution methods and technologies have been oriented in the direction of finding some alternative solutions of unitary depollution, disposal, reassessment and ecological reutilization of the residue (Asano et al., 2007). The aim of alternative solutions is that of minimizing the collected quantities of residue and reducing their impact upon the environment. Residue disposal technologies are limited by the implementation costs, the complexity of the type of contained contaminants, as well as the large quantity of collected residue (Cheremisinoff, 2003; Cheremisinoff & Cheremisinoff, 2005; Harrison & Hester, 2002). This type of technologies provides partial solutions, being dedicated especially to the disposal of certain classes of pollutants with high toxicity level or easily separable contaminants (paper, plastics, metals, wood, etc). Reassessment and reutilization of the collected residue have the highest applicative potential, favouring the preservation and regeneration of the natural resources and environmental factors (Baud, Post & Furedy, 2004). Research in the field of waste recovery follows more directions, among them: reassessment -reutilization (plastics, metals, precious metals, etc.), production of new biodegradable depollution materials, the recovery of wastes having organic substrates (building materials, semi-artificial fertilizers, etc.). Waste recovery has some direct active functions within depollution processes, having as direct objects: the simplification of management procedures, the reduction of pollution factors, minimizing the effects of residue and pollutants upon the environment, the preservation and regeneration of natural energy resources. Finding viable and applicable solutions of management and waste recovery, which might have a low impact upon the environment and which might be financially sustainable represents a top priority and a necessity of environment science.

The soil represents the fundamental structural element of the environment, highly exposed to pollutants, acting like an absorbent, due to its own morphological structure and due to

the morphochemical and compositional structure of its component elements. The soil presents in its structure an amorphous mineral component (95 – 99 %) (acid, basic and neutral rocks and particles containing: O 46.6%, Si 27.7%, Al 8.1%, Fe 5.0%, Ca 3.7%, Na 2.8%, K 2.6%, Mg 2.1%, etc.; mechanical, chemical, biogenical sediments or their mixtures) a fluid component (characterised by: osmotic pressure, pH, oxido-reduction potential, colloidal structure, buffering capacity, absorbing capacity), a gaseous component (CO₂ – up to 1%, O₂ – 10-20%, N₂, NH₃, water vapours, H₂S, H₂, CH₄, SO₂, etc), a living matter component (bacteria, fungi, algae, protozoa, insects, arachnoides, molluscs, earthworms, etc.) and non-living matter components (humic acids, fulminic acids, humins, simple saccharides, fatty acids, alcohols, esters, starch, proteins, complex proteins, pectins, hemicellulose, cellulose, lignin, wax, bitumen, etc) (Frank & Tolgyessy, 1993). The chemical and morphochemical structure of the soil shows the complexity of its morphofunctional structure. The morphofunctional complexity is expressed by the active chemical functionality variation, capable of absorbing, encapsulating and reticulating the pollutants. Excepting the persistent compounds, in most cases of diffusion of the inorganic pollutants in the soil, the local morphochemical composition is irreversibly modified (Yu & Wang, 1997).

Organic pollutants (especially the surfactants, fatty acids, gasoline, petroleum and its derivatives) diffuse in the surface and depth structure of the structural elements of the soil, deactivating, masking or isolating the structures having functions in establishing and adjusting the chemical and biological balance mechanisms (minerals, organic matter, micro-organisms, colloids, etc.) (Perk, 2007; Evanghelou, 1998). Organic pollution has a deep persistent character and in most situations of this kind, the morphochemical, functional and biochemical soil structure is irreversibly altered. Biotechnologies remain the only solutions that have potential for organic depollution and remediation of the soil. Soil depollution and remediation biotechnologies depend on the type of removed pollutants, they affect on short-term the local biochemical balance of the ecosystems, the action mechanisms taking place in time and being noninvasive (Evanghelou, 1998). The substantiation and development of new technologies of soil depollution and remediation is a fundamental priority of environmental science, with major implications to: human health, environmental factors, human habitat distribution, redistribution of natural resources, economy.

Radiological pollution differs from the other types of pollution sources through its generating mechanisms, methods and decontamination-depollution technologies and through the effects that it induces to biodiversity and environmental factors (Bayliss & Langley, 2003). The only known radiological depollution methods are the preventive measures, aiming at the complete isolation of radiological materials and compounds and at preventing their dispersion in the structure of environmental factors. Radiological pollution generators are made up of different radioactive chemicals that have the ability to emit radiation (charged radiation, γ , neutrinos, etc.), independent of the chemical structure that hosts the unstable nucleus. The carrier support of radioactive elements in the environment occurs through external factors (aerosols, encapsulating matrices, suspensions, oxides, halides, contaminated materials, etc.) called carrier vectors. In the case of nuclear accidents, the separation of carrier vectors is a delicate problem, due to the risk of staff contamination and due to their low chemical reactivity, which does not allow the separation and encapsulation processes. Considering the physical and chemical characteristics of the carrier vectors, the only solutions with applicative potential in the separation of radiological

contaminants are those based on the use of externally stimulable highly functionalized materials, in the form of sorbents or nanostructured suspensions with high specific surface, that will be able to reticulate, encapsulate, separate and store the contaminants safely, without exposing human staff (Ojovan & Lee, 2005). Radiological pollution and its effects model radically and on long term the structure and the overall functionality of the environment, human health and factors influencing macroeconomy. This type of pollution has an unpredictable nature, its implications on the environment and biodiversity, and the evolution orientation towards local and global equilibrium factors being difficult to estimate. Radiological depollution represents another priority research direction, due its deep implications on energy resources and on the needs of the consumer society (medicine, research, material science).

4. New types of functionalised materials and new applicative solutions concerning the ecological organic depollution of wastewaters

This paper presents the way a new class of functionalized nanocomposite materials, intended for the organic depollution of surface water from industrial and domestic activities, was obtained, investigated and tested. The means of obtaining the composite has already been presented in the previous chapter, "Nanocomposite materials with oriented functionalized structure - The modelling of structure and functionality of nanocomposites intended for ecological depollution". Considering the limitations of current methods and techniques for the separation of pollutants, research and structural and functional modelling investigations have been oriented with a view to obtaining a nanocomposite with an oriented functionality, able to: a. undifferentiatedly reticulate a large number of organic pollutants; b. encapsulate and degrade pollutants and their degradation products; c. separate and extract pollutants and depollution waste under controlled stimulability. Amorphous natural metal-oxide structures are the most suitable class of materials to be used in processes of chemical modelling and functionalization, intended for the removal of organic pollutants. Their functional, morphological and morphochemical structure, resulting from functionalizing processes, is compact and able to fully saturate its functionalized surface, with different pollutants which have been collected from the depolluted environment. Following the processes of chemical modelling and functionalization, the deep structure of the chemically modified metal-oxide components remains unchanged, giving the material a pronounced ecological and biodegradable character. In the first stage, the pollutants, reticulated on the surface and in the depth of the composite, are degraded and encapsulated, resulting in degradation products with modified functionality and toxicity. It is likely that the toxicity of the resulting degradation products might not be substantially modified, but it is very important that, following the reticulation process, the pollutants and the degradation products should remain reticulated on the surface and in the depth of the material, allowing a slow selective biodegradation of the pollutant layer collected on its surface. Equally, the stable reticulation of the pollutants and of the degradation products limits the effects of secondary pollution and contamination by dispersing the resulting degradation compounds. Such depollution materials have an increased application potential in relation to the applications of depollution under the dynamic conditions of polluted surface water flows (rivers, lakes, etc.), as they minimize the dispersion processes and favour local biodegradation processes by local sedimentation of

pollutant-charged functionalized suspensions in bottom water. Due to the macromolecular character and the micrometric structure of depollution suspensions, nanostructured materials cannot be assimilated at cell level by the biodiversity they encounter, as it has already been demonstrated in the previous chapter that the surface and the depth of the composite are able to host biological structures, without affecting their morphochemical structure and metabolism.

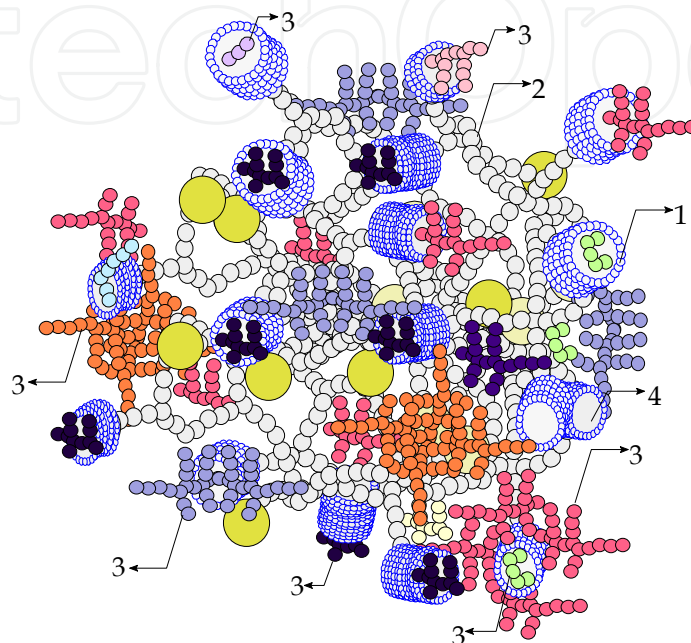


Fig. 2. (1 – mineral metal oxide; 2 – internal retaining metal-oxidic cage – mixed oxidic morphostructure; 3 – different types of organic and inorganic pollutants; 4 – external retaining metal-oxidic cage – specific and mixed oxidic component morphostructure)

The metal-oxide mixture (Su) used as a structural component of composing of the depollution material (M_{OF-DP}) has a natural origin and has some remarkable morphostructural (fig. 4), morphochemical and functional (fig.3) features. Using the basic segmentation method proposed by Iordache et al. (Iordache et al., 2010), the main morphostructural parameters of Su were determined and quantified (fig.4b): area (11.41 nm^2) compactness (1.29), perimeter (16.48 nm). Investigations carried out by X-rays dispersion show that Su contains, apart from oxidic fractions of the following elements Na, Mg, Al, Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Rb, Sr, Y, Zr, Ba, Pb, B, O (45.76%), oxidic fractions of other elements (Sn, Ge etc. - Fig.3), C ($\sim 4\%$), halogens (Cl ($\sim 0.019\%$), Br) and nitrogen. All metal-oxide structures identified in the Su structure form nanostructured elements having a compact amorphous structure (Fig.5), whose topological distribution is most likely of the type modelled in figure 2. This was confirmed by the numerical data obtained by elementary morphological segmentation (fig. 4b) for the compactness parameters. The compactness range of values (Round) has values in the interval $(1.29 \div 9)$, representing a measure of deviation from the sphericity of agglomeration domains and of the nanostructured elements which enter their structure (1 corresponds to the perfect spherical structures) (Jain, 1989). The perimeter (Round = $P^2/4\pi S$) has values on the domain ($\sim 10.3\text{-}107.12 \text{ nm}$), a measure of the size of nanoparticles and their agglomeration domains.

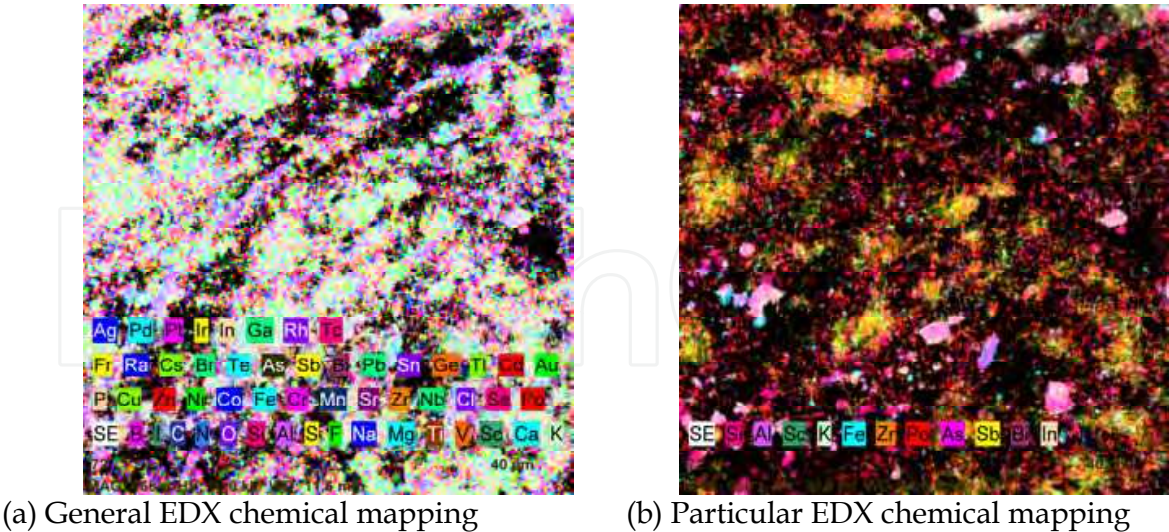


Fig. 3. Su EDX mapping (Su laid-down on ordinary paper)

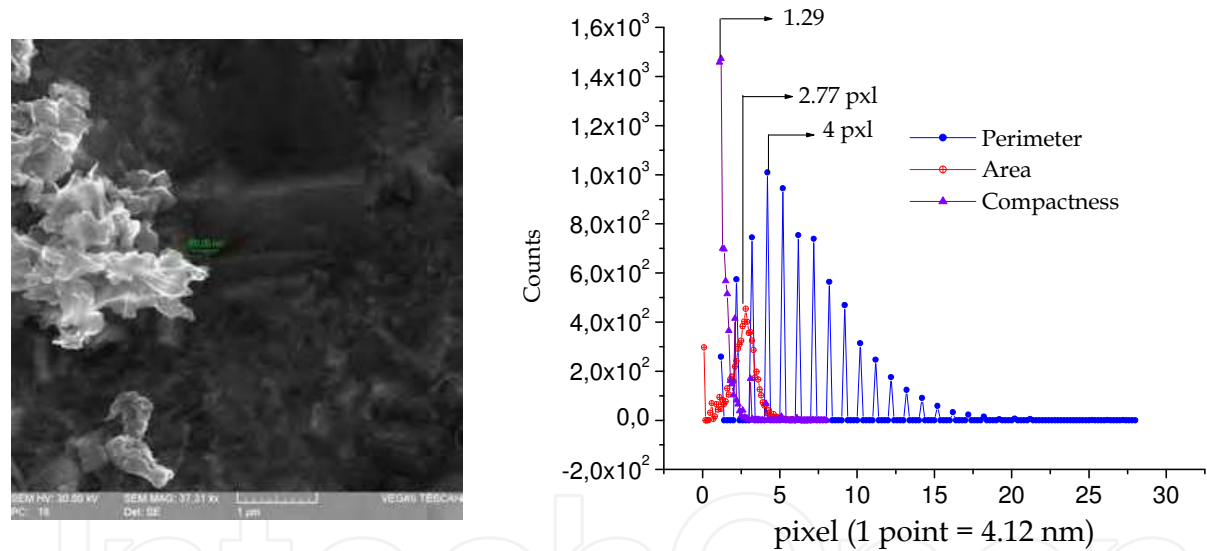


Fig. 4. SEM morphotopological investigation of Su (VEGA II LMU microscope)

The identification of chlorine and bromine in the Su structure implies that the metal-oxide mixture has a complex chemical functionality, which includes metal salts (MX, X - Cl, Br, etc.), halide oxides (MOX), metal-oxide-halogenated mixtures ((M_i-O-X)_n(M_j-O-X)_m; i, j - different types of metals; m, n - compositing ratio), H and -OH chemical reactive groups, electrical charged microsites, etc. M_{OF-DP} was obtained by the regeneration of the compositing mixture (functionalised magnetite, functionalised Su and solubilised cellulose) in distilled water. As an applicative and testing form, M_{OF-DP} was obtained as a powder by mild thermic dehydration at 80°C (fig.4A.a), and as a membrane (fig.4A.b) concentrated suspension.

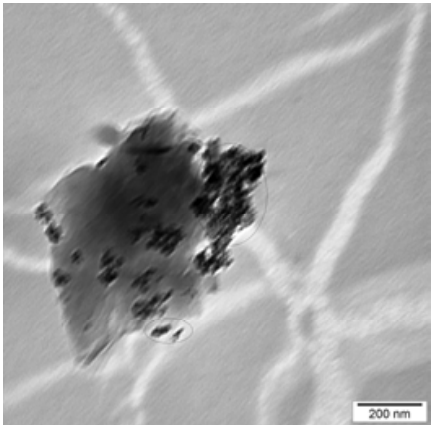
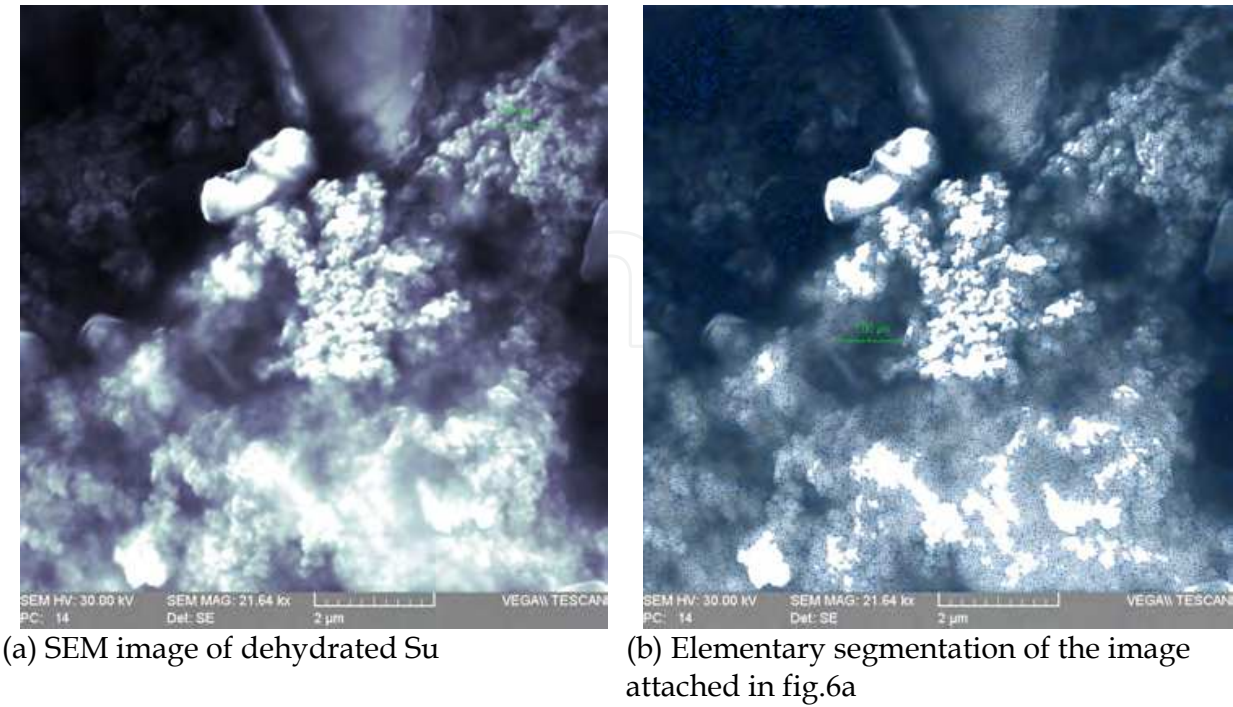


Fig. 5. TEM images of Su embedded in the structure of M_{OF-DP} -uncharged pollutants



Morphostructural investigations carried out by scanning electron microscopy (SEM) show that dehydrated M_{OF-DP} has a smaller and more irregular specific surface, as compared to the hydrated M_{OF-DP} . The distributions of the agglomeration domain surfaces of the dehydrated M_{OF-DP} has values between 194 nm^2 and $12\text{ }\mu\text{m}^2$ (fig.8). From the investigation of the distribution of the domain compactness of the agglomerated structures and of the nanoparticles within their structure, taking into account the analytical relationship $\text{Round} = P^2/4\pi S$, it follows that the dehydrated material suffered internal degradation processes that led to its severe discretization and to the increasing of agglomeration domains. It is likely that the increasing of the agglomeration domains may be due to secondary internal morphochemical modelling processes under the influence of temperature, as a result of which, uncontrolled reticulation processes might be established between the structural elements of the composite. It is very likely that, following the dehydration processes, M_{OF-DP} may lose and partially modify its functionality.

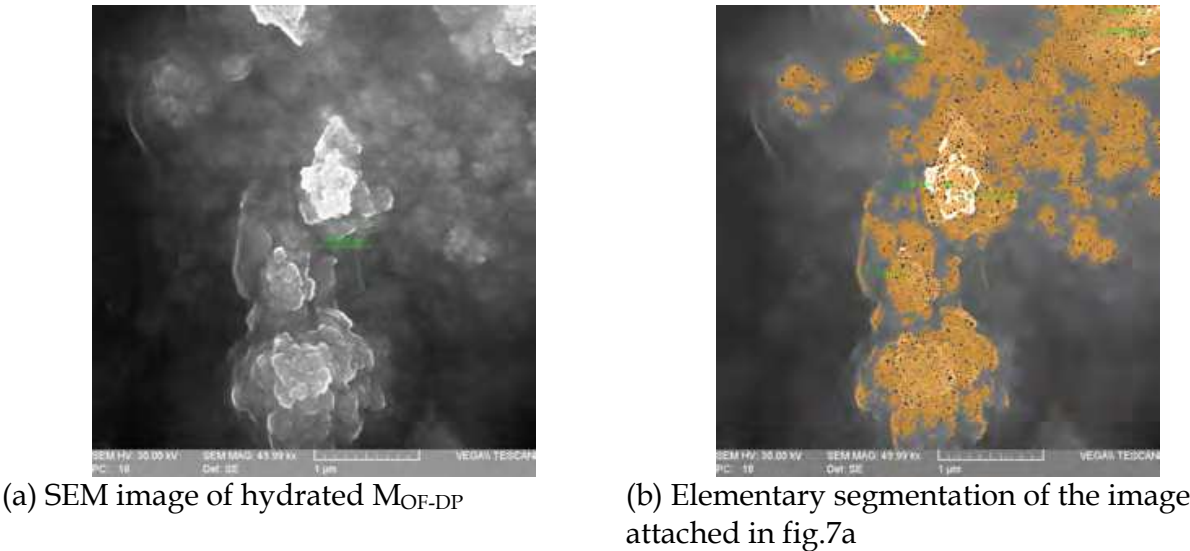


Fig. 7. SEM investigation of hydrated M_{OF-DP} (acquired with VEGA II LMU microscope)

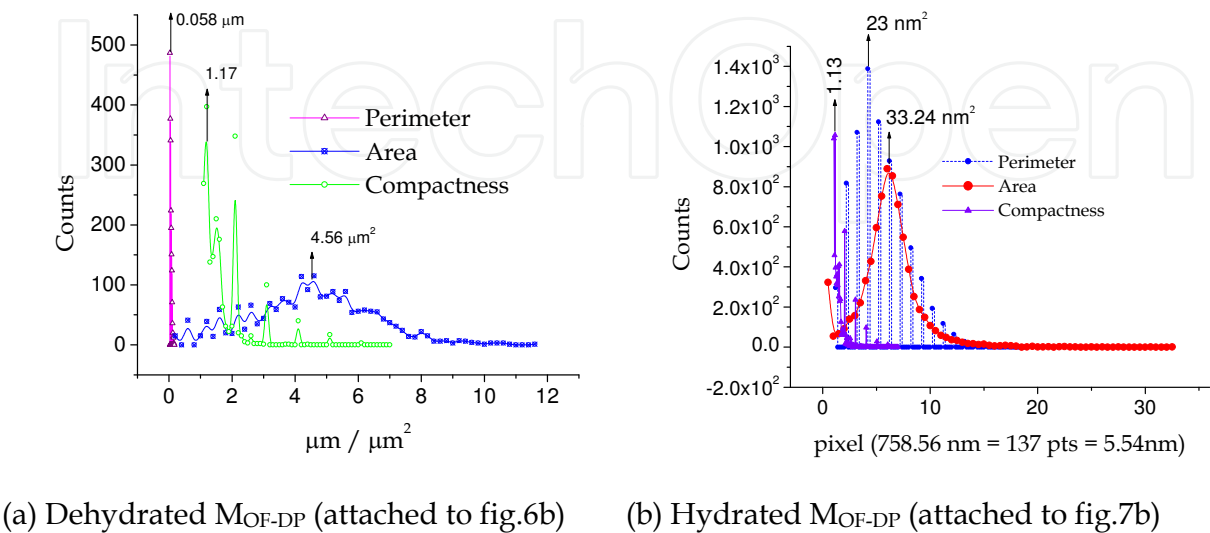


Fig. 8. Elementary morphostructural parameters of M_{OF-DP}

4.1 Depollution of sewage waters using functionalised M_{OF-DP}

This type of investigations had as main objective the determination of depollution parameters (yield and depollution efficiency) for the type of investigated water, as well as for the morphostructural and morphochemical stability of M_{OF-DP} . Investigations have been configured in accordance to the diagram in figure 9. Samples were investigated by gas chromatography coupled with mass spectrometry (GC-MS), using a chromatograph of the GC Focus type (AI300 autosampler, split / splitless injector, chromatograph column of the TR5MS - 30 m x type with $\Phi_{ext} = 0,25$ mm and 0.25 mm stationary film thickness) and a mass spectrometer of the DSQII type. Previously, samples were concentrated by solvent extraction and evaporated in nitrogen flow. To check the saturation point of M_{OF-DP} with pollutants, 50 ml of water charged with pollutants were passed through the filtering layer (fig. 9), in four steps, the (P1, P2, P3, P4) samples were properly prepared and investigated. To determine the analytical correspondence holding between the separated pollutant quantity and the real pollutant quantity in the contaminated water (R_p), the quantity of pollutants present in the non-filtered water was determined before starting the investigating processes. The separated pollutant quantity was estimated by determining the pollutant fractions found in P₁, P₂, P₃ and P₄, after filtering ($c\%$ (pollutants) + $c\%$ (separated) = 1). The investigated samples were taken from the sewerage system of Bucharest municipality. With a view to determining the morphochemical and morphostructural stability of M_{OF-DP} , the composite charged with pollutants was investigated by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX) and infrared spectroscopy (IR).

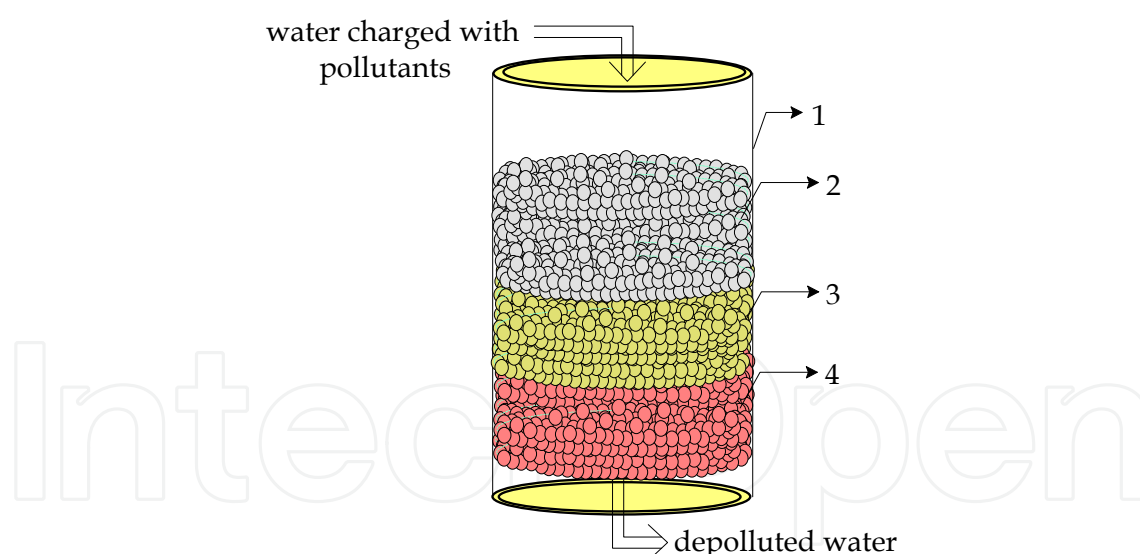


Fig. 9. Experiment configuring for the investigation of water depollution yield and efficiency of M_{OF-DP} (1 - fixing column; 2 - polluted water; 3 - M_{OF-DP} layer; 4 - fibre glass)

The results of the investigations carried out by GC-MS are presented in table 1 and they demonstrate the fact that the tested water had a high level of organic pollution, containing a large range of organic pollutants (n-tridecane, propanoic acid, 2-methyl-1-(2-hydroxy-1-methylethyl)-2,2-dimethylpropyl ester propanoic acid, 2-methyl-3-hydroxy-2,4,4-trimethylpentyl ester, diethyl benzene-1,2-dicarboxylate, phthalic acid, isobuthyl-2-pentylester, butyl octyl phthalate, dibutyl phthalate, ((Z)-9-octadecenitrile).

Pollutant/ undecelable peaks	A _r -R _P	A _r -P1	A _r -P3	A _r -P4
TS _P	2848678635	15047488950	10533922491	8526288052
8,33	5094541	0	2272703	7299050
PL1	977320	0	0	0
10,75	5268711	0	0	5547832
PL2	2465265	0	0	0
PL3	1405799	0	0	0
12,49	1740771	0	0	0
12,93	2828747	0	0	3710438
14,85	3347278	0	1841992	2755159
PL4	1680227	0	0	0
16,13	523552	0	0	0
16,51	1704027	0	0	0
17,23	1278456	0	0	0
17,98	901907	0	0	1129834
PL5	132278	1730996	0	0
18,27	267140	0	0	0
PL6	507889	0	0	0
PL7	10830818	8652426	0	0
PL8	1859244	0	0	0
24,00	5434244	2712903	4682044	2903562
26,56	11791893	8461410	6699962	4400335

Table 1. The relative abundance (A_r) of decelable and partially decelable pollutants in filtered water – case of sewage waters (where: PL1 - n-tridecane; PL2 - propanoic acid,2-methyl-,1-(2-hydroxy-1-methylethyl)-2,2-dimethylpropyl ester; PL3 - propanoic acid,2-methyl-, 3-hydroxy-2,4,4-trimethylpentyl ester; PL4 - diethyl benzene-1,2-dicarboxylate; PL5 - phthalic acid, isobuthyl-2-penthylester; PL6 - butyl octyl phthalate; PL7 - dibutyl phthalate; PL8 - (9Z)-9-octadecenitrile)

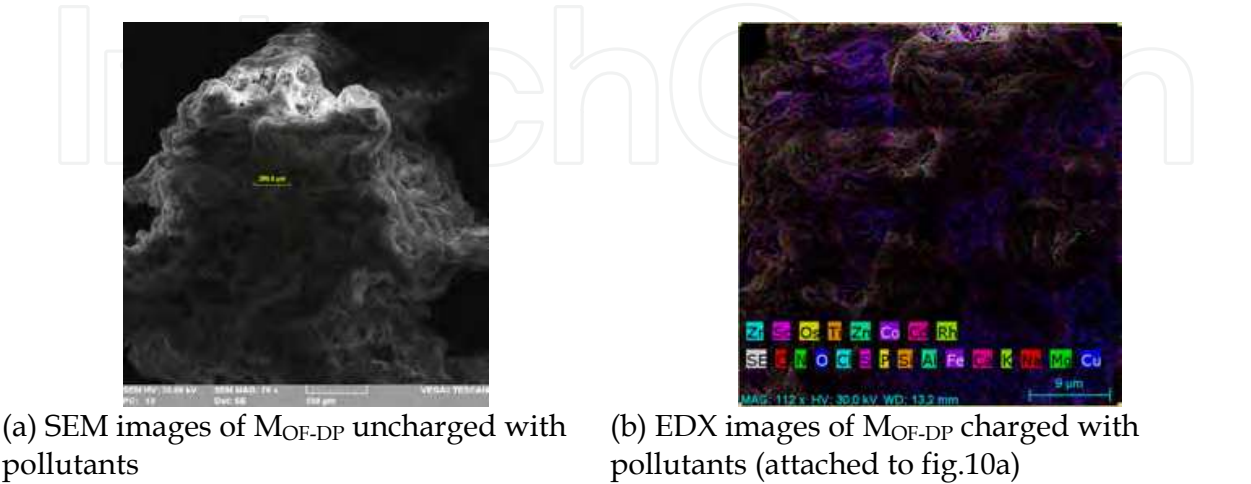


Fig. 10. SEM investigation of M_{OF-DP} charged and uncharged with pollutants – case of sewage waters (images acquired with VEGA II LMU SEM microscope)

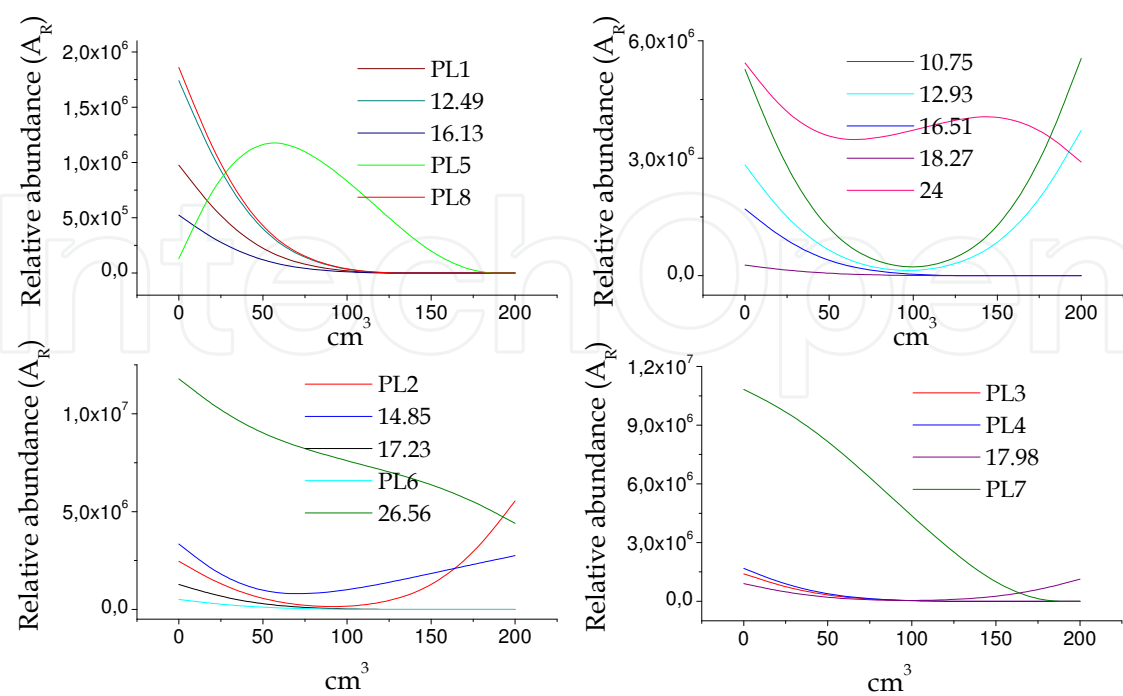


Fig. 11. Relative yield of decelable and partially decelable pollutants retained by 71.43 grams of M_{OF-DP} (according to tab.1)

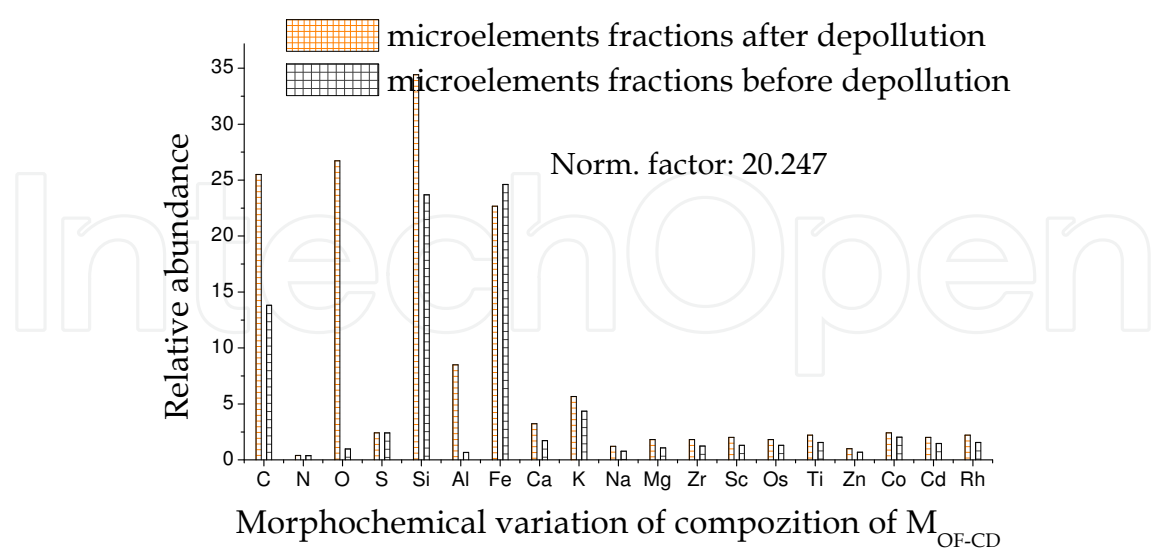
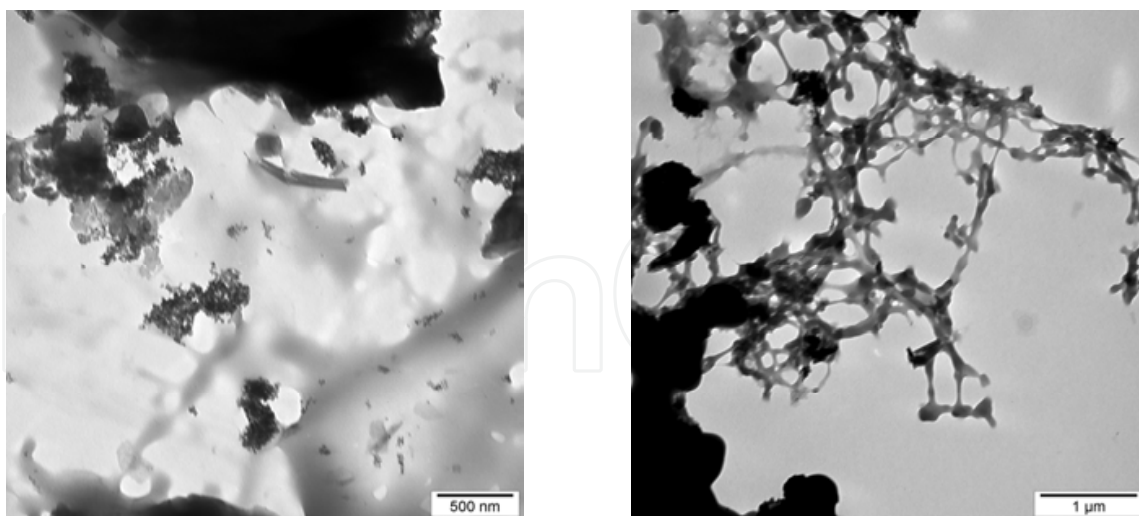


Fig. 12. Relative variation of microelement fractions in the depth and on the surface of M_{OF-DP} charged with pollutants



(a) TEM images of MOF-DP – uncharged pollutants

(b) TEM images of MOF-DP – charged pollutants

Fig. 13. TEM morphostructure investigation of MOF-DP charged with pollutants - case of sewage waters (images acquired with Philips S208 TEM microscope)

The GC-MS investigations revealed the fact that the filtered water (the water charged with pollutants) contains a wide range of organic pollutants, a significant fraction of them being partially decelable. Partially decelable pollutants have clear, distinct peaks, whose variation can be quantified depending on the quantity of filtered water (table 1). A great part of the chemical compounds present in the investigated water could not be sensitized in relation to the retention time and the specific mass fragments. The quantification of these types of pollutants was entirely determined, by quantifying the variation degree of the whole sensitization area. It has been noticed that the concentrations of decelable pollutants and of the partially decelable ones decrease rapidly with the increase of the filtered water quantity (fig.11). Each pollutant attains a certain value for which the membrane is saturated through specific reticulation. After attaining the saturation peak, the filtering layer loses its ability to separate the pollutants which saturated it, thus becoming permeable in relation to them.

The saturation processes are most likely due to the neutralization through reticulation of the functional groups with which the pollutants come into contact. This fact was pointed out indirectly by SEM (fig.10 a), TEM (fig.13) and EDX (fig.10b, fig.12) investigations. Thus, we observed the severe degeneration of the specific reticulation surface of MOF-DP (fig.13b). The degradation and reticulation processes have also been confirmed by EDX investigations, during which we noticed the fact that the microelement fractions (especially C and O) found in the depth and on the surface of MOF-DP increase significantly (fig.12). This fact can be explained by admitting the fact that organic pollutants reticulated on the surface or in the depth of MOF-DP form covering layers which lead to the masking of the detection of the structural elements of the composite.

The determinations in fig.12 have been carried out by supposing that the statistical quantity of silicon in the composite structure is approximately constant. Thus, to obtain the same quantity of silicon on the investigated microsurfaces, both before and after the depollution process, a normalization factor of 20.247 was added ($20.247 \times c\%\text{Si}(\text{after}) = c\%\text{Si}(\text{before})$), to minimize the errors of the statistical distribution of microelements. In

order to obtain results as accurately as possible, the normalization microelement must have variations of the concentration distributions only on one of the investigated microsurfaces. These errors may be due to the different distribution probabilities of morphochemical patterns on distinct microsurfaces, as well as to the different periods of time of data acquisition.

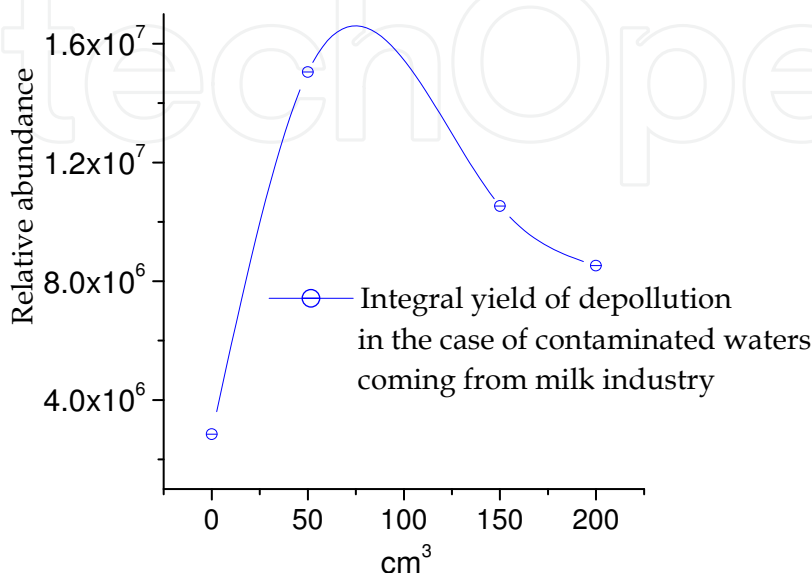


Fig. 14. Integral yield of sewage water depollution in the presence of 71.43 grams of M_{OF-DP}

The integral retention spectrum of the pollutants has been defined as the whole recorded sensitization area, minus the area of the background signal. This represents an indirect measure of the pollutant retention spectrum size, offering relative information regarding the variation of the quantity of organic compounds, which are generated or disappear from the collected depolluted water, before and after the depollution process. The integral retention curve shown in figure 14 presents the maximum variation at approximately the same value as that of the saturation point of M_{OF-D} with decelable and partially decelable pollutants. This suggests that during the depollution process, the reticulated pollutants are partially or completely degraded, at the level of functionalized micro surfaces of the structural components of M_{OF-D} . Degradation processes can influence the dynamics of the depollution processes, as well as the achievement of specific saturation points. This explains the analytical structure of the depollution curves of pollutants 12.93 (specific retention time), 24 and PL2 (fig.11). The same tendency is also shown by the decontamination curve of the entire spectrum of pollutants (fig.14).

4.2 Depollution of waters coming from milk industry using M_{OF-DP}

As in the previous case, this type of investigations had as objective the determination of depollution parameters of polluted water from milk industry, as well as the morphostructural and morphochemical stability of M_{OF-DP} . The investigated samples came from a milk processing plant. Complex investigations were carried out by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX).

Pollutants	A _r -R _P	A _r -P1	A _r -P2	A _r -P3	A _r -P4
Fenil carbatat	5786268	0	0	0	0
p cresol	28206310	0	3291097	2866582	4749335
5H-1 pyridine	9622808	0	0	0	0
NN dodecilamina	5710090	0	0	0	0
4 methylindolina	2179991	0	0	0	0
Heneicosane	589354	0	0	0	0
N[3[N-Azydiril]propilidena] 3-dymethylaminopropylamina	1378132	0	0	0	0
Octadecylacetate	6770653	0	0	0	0
Acidhexadecanoicisopropilester	5107004	0	0	0	0
Adogen	6786347	0	0	0	0
Acidoctadecanoictertbutilester	7061859	0	0	0	0
All-trans-squalene	20340307	0	0	0	0
Region with undecelable pollutants	198277663	0	0	0	0

Table 2. The relative abundance of decelable and partially decelable pollutants - case of waters coming from milk industry

Depolluted volume of water (cm ³)	Total relative abundance (A _r)
50	2494088578
100	1286801634
150	2164651861
200	5424524876

Table 3. Integral fraction of sensitized pollutants (58.6 grams of M_{OF-DP}) in the cases of P1, P2, P3 and P4 stages of depollution – case of waters coming from milk industry

To determine the relative degree of depollution, the total area of the contents of pollutants in the unfiltered water ($R_{P0} = 6227542517$) was measured. Also, the total area of the contents of pollutants contained in 50 cm³ of distilled water ($R_{PA} = 2371295106$) passed through the filtering layer was measured, to correct and determine the level of contaminants released by M_{OF-DP}.

C	13.83	S	2.42	C	2.83	S	1.11
Sc	1.32	Cd	1.48	Sc	0.16	Cd	0.15
Zr	1.25	Os	1.32	Zr	0.34	Os	0.22
Fe	24.62	Ca	1.72	Fe	1.29	Ca	0.64
K	4.37	Si	23.69	K	0.56	Si	2.72
Al	6.8	Mg	1.09	Al	1.05	Mg	0.22
Na	0.78	O	0.99	Na	0.36	O	1.01
N	0.39	Ti	1.56	N	0.1	Ti	0.14
Co	2.03	Zn	0.70	Co	0.13	Zn	0.06
Rh	1.56			Rh	0.15		
(a) before depollution				(a) after depollution			

Table 4. Fractions of microelements (arbitrary u.m) measured on surface and in the depth of M_{OF-DP} in the cases of contaminated water depollution coming from milk industry

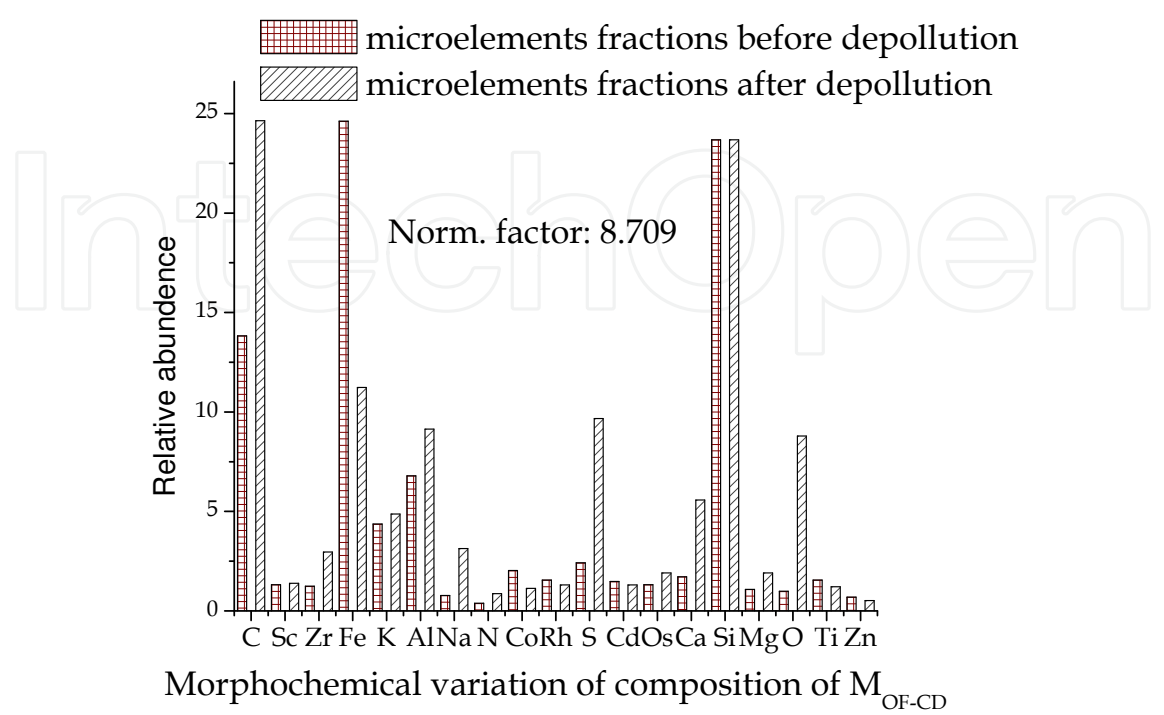


Fig. 15. Relative variation of chemical microelements fraction in the depth and on the surface of M_{OF-DP} (according to tab.4)

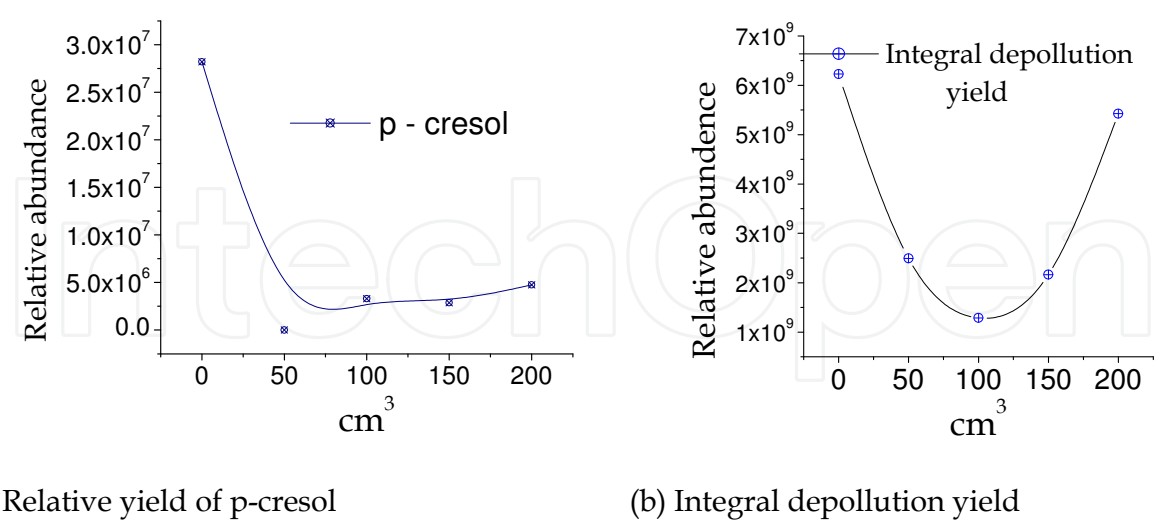
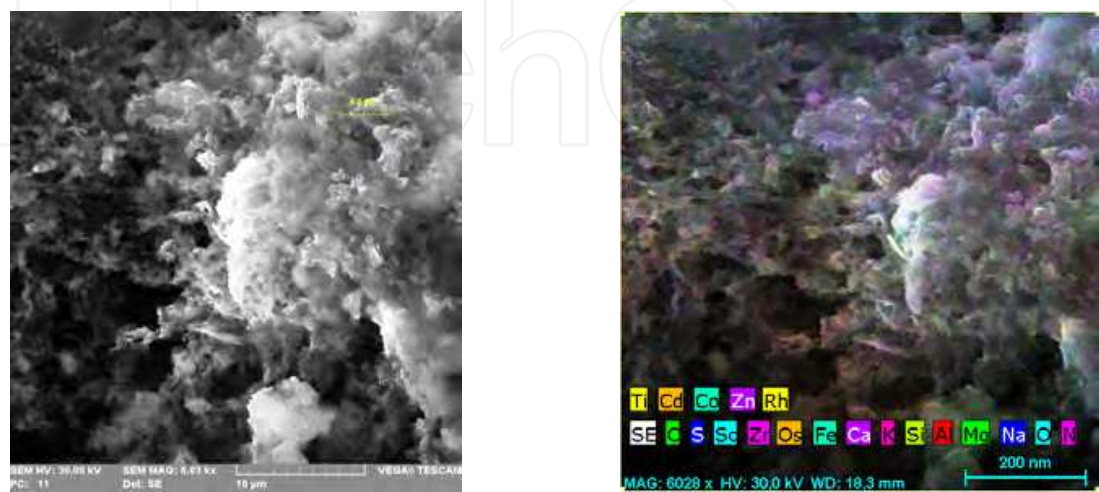


Fig. 16. Yield and efficiency of 58.6 grams of M_{OF-DP} – case of waters coming from milk industry

By determining R_{P0} și R_{PA} , we had in mind checking the morphochemical stability of M_{OF-DP} , as well as determining the contribution of the pollutants coming from the filtering layer to the total pollution spectrum of filtered water (fig.19). Also, the aim of determining R_{P0} and

R_{PA} was to the determine the background signal level, found in the analytical structure of the aquired GC-MS spectra. The results of the investigations for determining the depollution yield and efficiency carried out on waters charged with pollutants coming from the milk industry have shown that M_{OF-DP} has high depollution yields. In the case of decelable organic pollutants (table 2, fig.19), the individual retention yields of pollutants can reach the maximum value ($\sim 100\%$).



(a) SEM images of pollutant-charged M_{OF-DP} (b) EDX images of pollutant-charged M_{OF-DP}

Fig. 17. SEM investigation of pollutant-charged M_{OF-DP} - case of water depollution coming from milk industry (images acquired with VEGA II LMU SEM microscope)

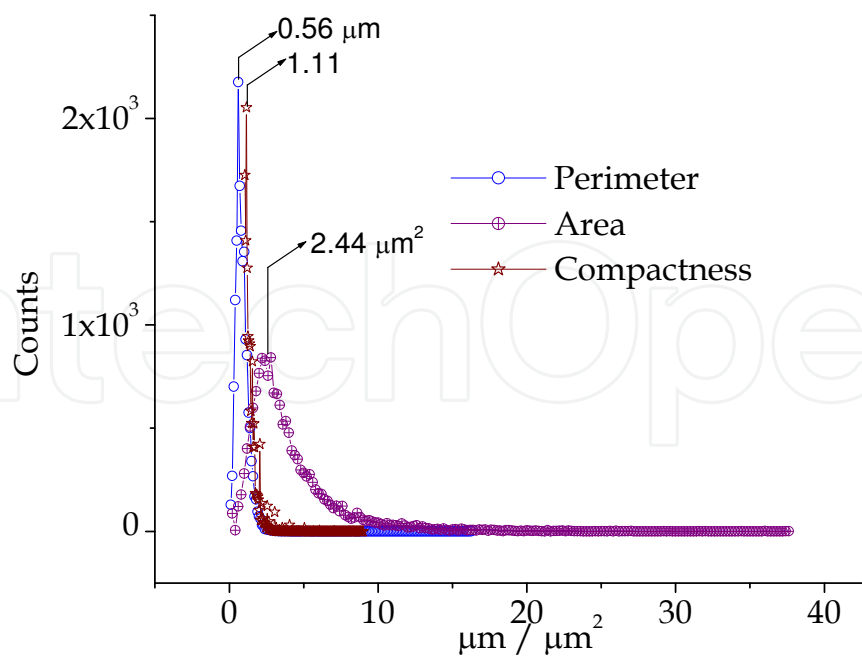


Fig. 18. Elementary morphostructural parameters of M_{OF-DP} in the case of depollution of water coming from milk industry (following the depollution process)

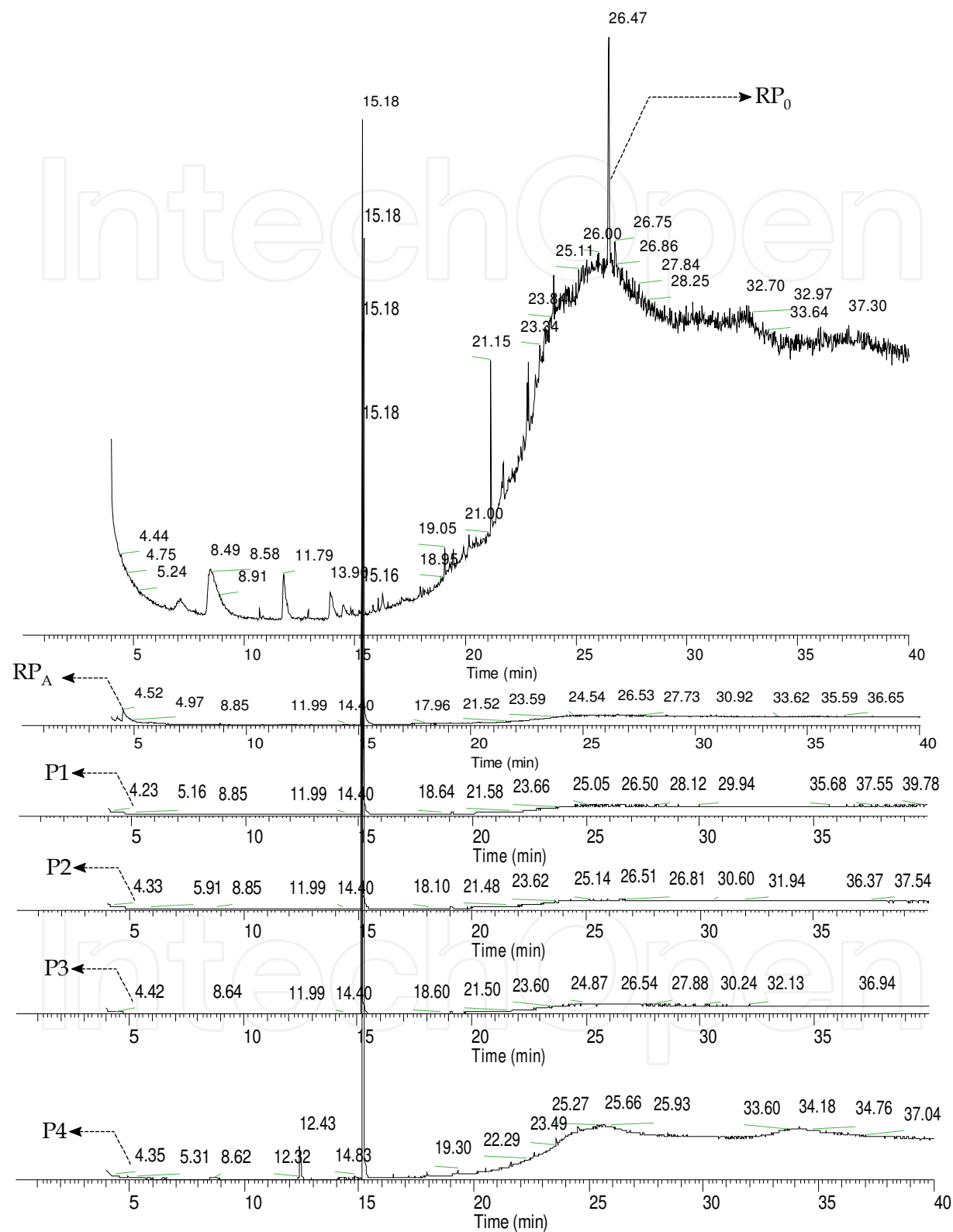


Fig. 19. GC-MS spectra of collected samples – evolution of integral depollution yield as a function of volume of filtered water ($\eta = f(V(\text{cm}^3), S(p_1, \dots, p_n))$; V – depolluted water volume; S – type of the contained p_1, \dots, p_n pollutants)

In figure 16.a one can observe that p-cresol has a higher saturation capacity (fig.16), most likely, due to the high content of this pollutant, either due to the lack of chemical affinity of the specific functional groups of M_{OF-DP} in relation to p-creso. The obtained results demonstrate that the mechanisms of reticulation and separation processes of the pollutants are uniform in relation to the functionality type. These findings also remain valid in the case of the investigation of waters coming from milk industry.

In the case of the contaminated waters coming from milk industry, several features have emerged, due, most likely, to the type of pollutants and to their concentrations in contaminated water. It is noted that the integral depollution spectrum (fig.16b) presents a more predictable analytical behaviour, as compared to the integral depollution spectrum of sewage waters. Pollutants contained by water generate specific morphostructural reticulation and agglomeration effects (fig.17, fig.18). We have observed that the characteristic agglomeration domains have the following values: a. average surface: $\sim 2.44 \mu m^2$ (distributed on an agglomeration domain that ranges from tens of nanometers to $35 \mu m^2$); b. maximum perimeter: $13 \mu m$; c. compactness value distributed on the 1 - 10 range (fig.18). The determined values for compactness show that M_{OF-DP} charged with pollutants has a compact structure, which has been severely degraded from a morphostructural point of view, as a result of the reticulation and internal morphochemical modification processes. The obtained numerical results in the case of polluted waters coming from milk industry demonstrate the high capacity of M_{OF-DP} to separate, degrade and encapsulate pollutants, on the surface and in the depth of its composite structure (tab.4, fig.15).

GC-MS investigations have demonstrated the ability of the obtained composite to reticulate a large number of pollutants (table 2), having a complex and varied functionality, as follows: saturated compounds with large linear chain (heneicosane), organic compounds with functionality and nitrogen content (adogen, 5H-1 pyridine, NN dodecilamina, N [3 [N-Azydiril] propilidena] dymethylaminopropylamina 3, 4 methylindolina), organic acids, esters, derivatives of benzene, carbamates, cyclic organic compounds, etc.

4.3 Depollution of waters coming from medicine industry using M_{OF-DP}

The investigated samples came from an antibiotics production company. The results of the investigations carried out by GC-MS are presented in table 5, demonstrating that they are heavily contaminated and degraded, containing a wide range of organic pollutants, varied from a morphochemical, functional and morphostructural viewpoint.

As it can be seen in the analytical representations in figures 20 and 22, the obtained material has a high potential of separation of organic pollutants, being able to reduce their concentrations below the limit of analytical technique detection (at ppm scale), given that they are found in appreciable fractions in the contaminated water.

It is to be noted that the investigations revealed the undifferentiated action of M_{OF-DP} on the the entire spectrum of partially decelable, decelable and undecelable pollutants found in the investigated polluted waters. This feature of the functionalized nanocomposite material is reflected by the total decontamination yield, which has a decreasing analytical variation, with the increasing of the water passed through the retention layer. It is also to be noted that the relative depollution yields, specific to each pollutant type, has different analytical behaviours, in accordance to the specific chemical affinity in relation to the functionalized filtering support (fig.20, fig.22).

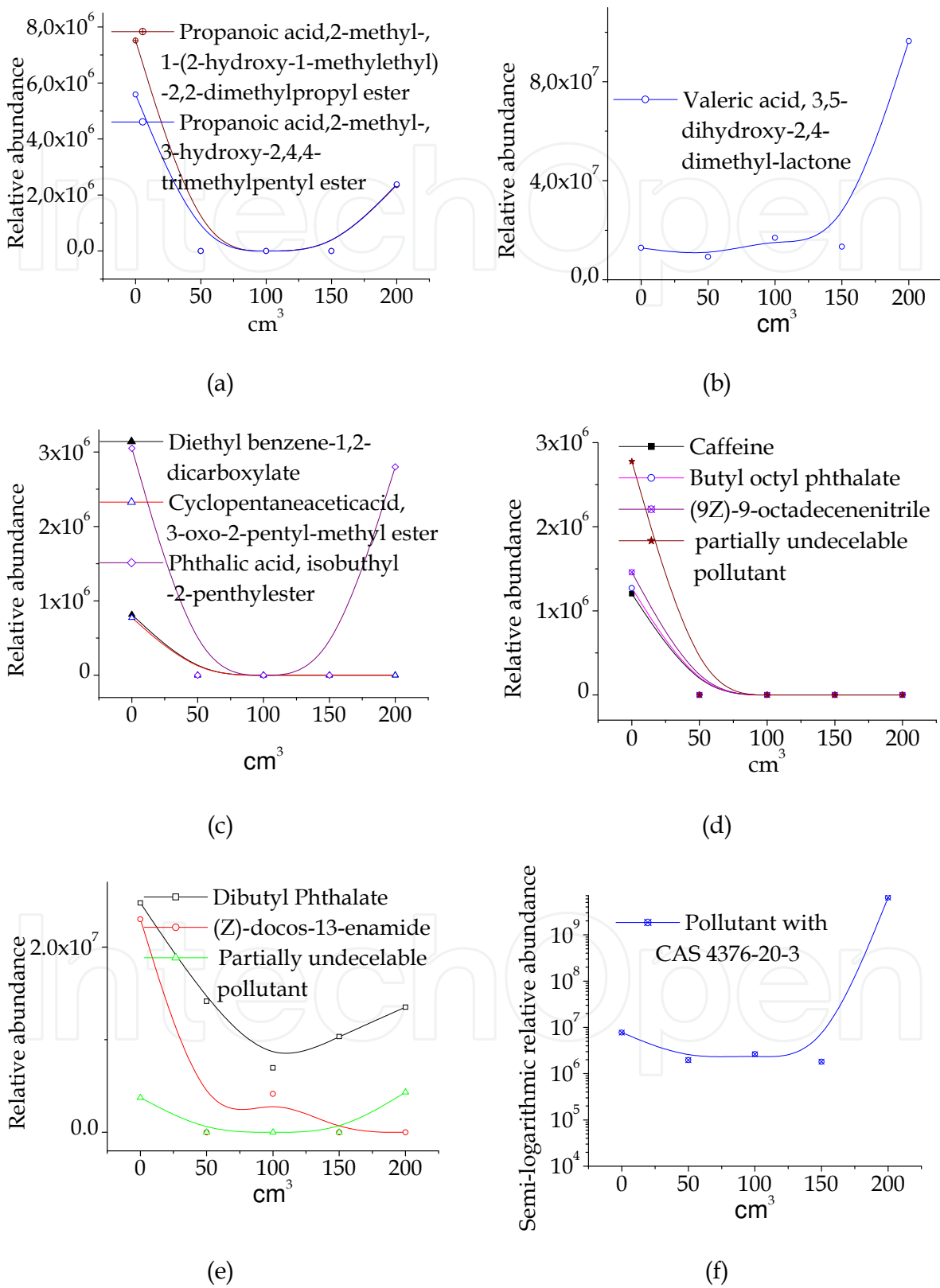


Fig. 20. Relative yield of decelable and partially decelable pollutants retained by 67.4 grams of MOF-DP (according to tab.5)

Pollutants	A _r -R _P	A _r -P1	A _r -P2	A _r -P3	A _r -P4
Integral pollutants abundance	39093281641	12969701158	11684530024	9414486816	13743895083
Acetonyl dimtehylcarbinol	268513312	295489231	736052142	570281431	503499232
Propanoic acid,2-methyl-, 1-(2-hydroxy-1-methylethyl)-2,2-dimethylpropyl ester	7515830	0	0	0	2353024
Propanoic acid,2-methyl-, 3-hydroxy-2,4,4-trimethylpentyl ester	5585726	0	0	0	2379634
Valeric acid, 3,5-dihydroxy-2,4-dimethyl-lactone	12891275	9221762	16911174	13363342	96422370
Partially undecelable	3741563	0	0	0	4326558
Diethyl benzene-1,2-dicarboxylate	815869	0	0	0	0
Cyclopentaneaceticacid, 3-oxo-2-pentyl-methyl ester	772606	0	0	0	0
Phthalic acid, isobuthyl-2-penthylester	3049818	0	0	0	2799809
Caffeine	1202278	0	0	0	0
Butyl octyl phthalate	1270036	0	0	0	0
Dibutyl Phthalate	24757815	14138926	6956899	10335640	13505478
(9Z)-9-octadecenitrile	1458619	0	0	0	0
Partially undecelable pollutant	2776681	0	0	0	0
CAS 4376-20-3	7729544	1965075	2632588	1815752	6402313990
(Z)-docos-13-enamide	22996078	0	4152090	0	0

Table 5. The relative abundance of the decelable and partially decelable pollutants - case of polluted waters coming from medicine industry

C	13.83	S	2.42	C	2.31	S	0.23
Sc	1.32	Cd	1.48	Sc	0.16	Cd	0.14
Zr	1.25	Os	1.32	Zr	0.19	Os	0.16
Fe	24.62	Ca	1.72	Fe	1.23	Ca	0.49
K	4.37	Si	23.69	K	0.4	Si	2.48
Al	6.8	Mg	1.09	Al	0.62	Mg	0.14
Na	0.78	O	0.99	Na	0.11	O	1.76
N	0.39	Ti	1.56	N	0.08	Ti	0.15
Co	2.03	Zn	0.70	Co	0.14	Zn	0.07
Rh	1.56			Rh	0.32		
(a) before depollution				(a) after depollution			

Table 6. Fractions of microelements (arbitrary measure units) measured on surface and in the depth of M_{OF-DP} in the cases of depollution of contaminated waters coming from milk industry

As expected, due to its polyvalent functionality, M_{OF-DP} saturates discretizedly, each pollutant separately saturating the composite, without masking or cancelling its

functionality and the integral depollution potential. This implies the existence of specific differentiated reticular mechanisms, which take place in the composite nanostructures, leading to the selective separation of pollutants, depending on the specific chemical reactivity and on the achieved density of functional groups on MOF-DP microspheres.

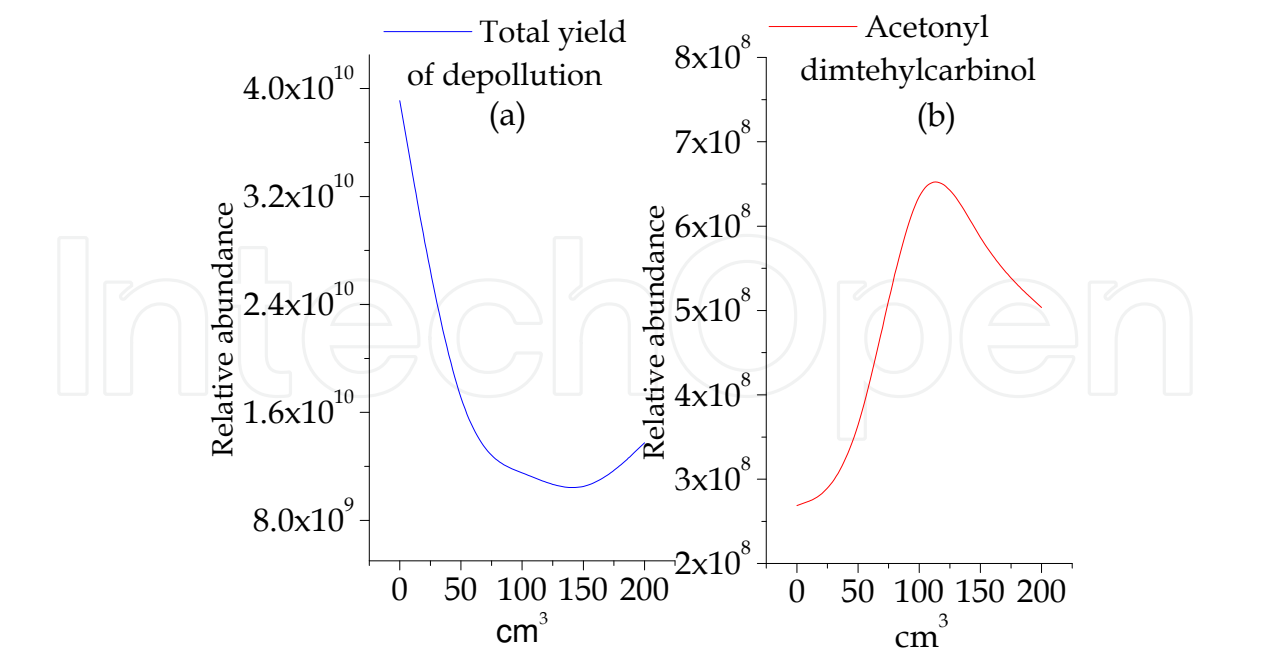
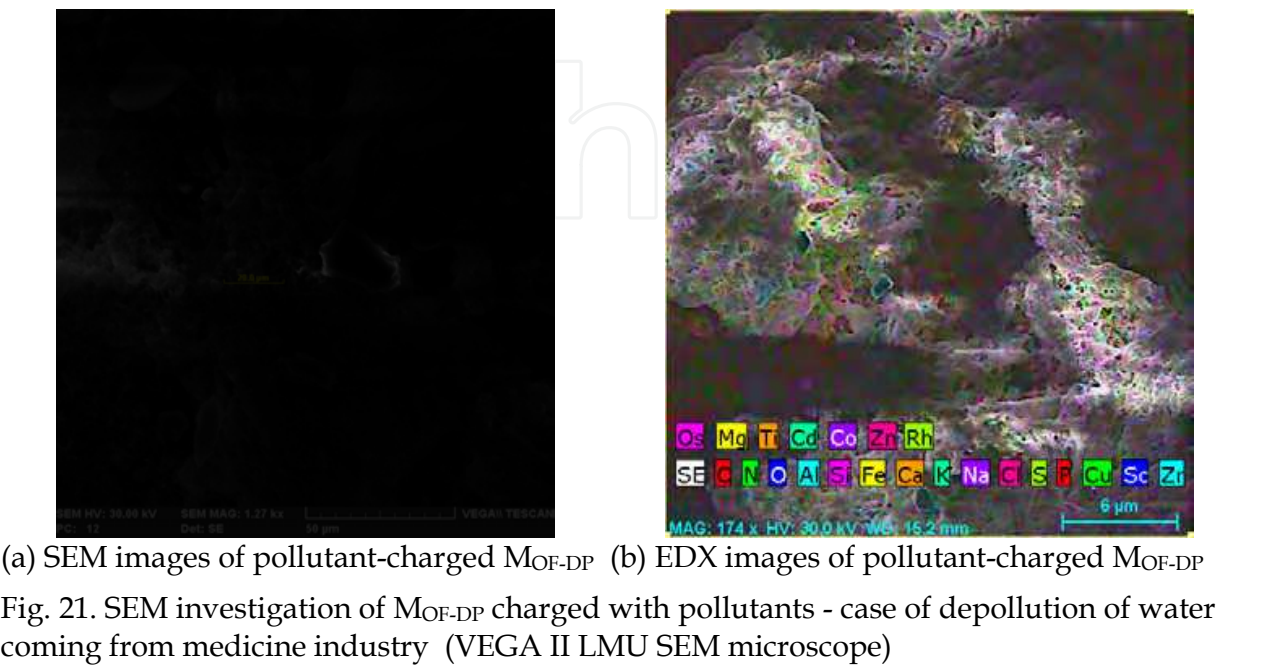


Fig. 22. Integral yield and efficiency of the depollution of water coming from medicine industry in the presence of 67.4 grams of MOF-DP ((a) - integral yield of depollution; (b) - relative yield of acetonyl dimethylcarbinol)

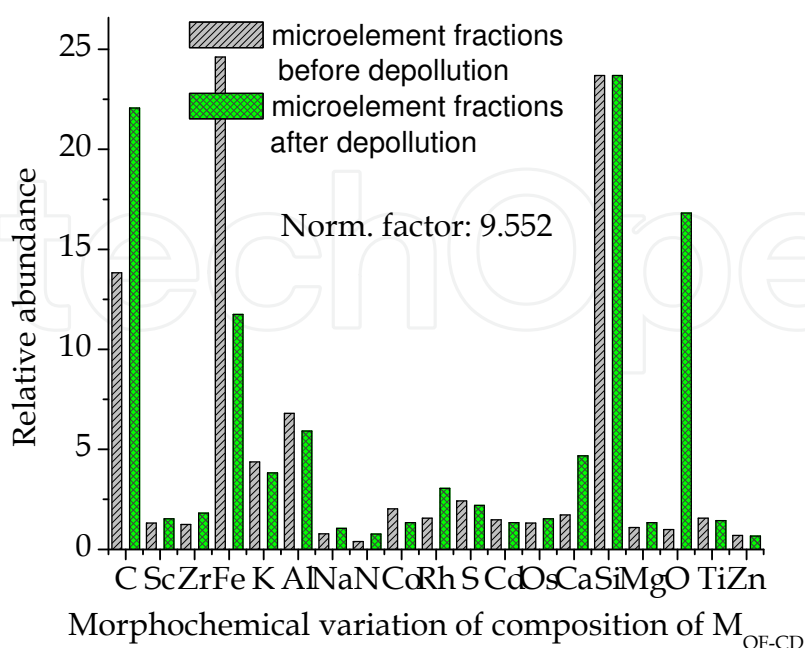


Fig. 23. Relative variation of microelements in the depth and on the surface of M_{OF-DP} (according to tab.6)

The separation investigations of the pollutants found in waters coming from medicine industry shows the manifestation of the dynamic degradation phenomenon of the pollutants. This is demonstrated by the analytical behaviour of the depollution curves of the valeric acid, 3,5-dihydroxy-2,4-dimethyl-lactones (fig.20b), the partially decelable pollutant CAS 4376-20-3 (fig.20f) and acetonyl dimethylcarbinol (fig.22b), which, after reaching the saturation point of the functionality of M_{OF-DP} , have growth rates that exceed by far the originally detected values. This indicates the presence of a pollutant in the contaminated water, which, following the reticulation processes, degrades and generates chemical structures that cannot be reticulated, or that are partially reticulated by the composite. All pollutants entering the structure of the integral spectrum, sensitized by pollution, of the investigated waters, have lower or higher separation rates. Thus, the lack of the reticulation potential, in relation to the degenerated pollutants listed above, is due, most likely, to: a. the saturation of M_{OF-DP} with the previously existing fractions of pollutants; b. large quantities of pollutants present in depolluted water, which quickly saturate the functionality of M_{OF-DP} ; the degeneration of the separated pollutants in the depth of M_{OF-DP} . Moreover, the morphostructural (agglomeration, compactness) and morphochemical (functionality saturation processes, masking, chemical degradation processes of reticulated pollutants) degeneration processes were emphasized in all investigated depollution cases. The effects of pollutant charging are also showed in the case of polluted waters coming from medicine industry. In this sense, the processed data presented in figure 15 (tab.6) indicate a significant increase in the quantity of chemical microelements, specific to the structures and organic pollutants (C, N). This is indirectly confirmed by morphostructural investigations (fig.21, fig.21b), indicating the increase of the agglomeration and compactness degree of the composite, as well as its degeneration under the action of separated organic pollutants.

5. Conclusions

In this chapter we have presented and investigated the specific mechanisms underlying the reticulation, separation and encapsulation processes of organic pollutants. Specific depollution mechanisms, as well as the main depollution parameters (yield, efficiency), have been investigated by testing a polyfunctionalized nanocomposite material (M_{OF-DP}) on three types of water charged with organic pollutants. The polluted waters on which the depollution features of M_{OF-DP} have been investigated came from various pollution sources: milk processing industry, medicine industry, sewage waters. The investigations' secondary aim was to determine the effects of organic pollutants on the morphological and morphochemical structure of the obtained M_{OF-DP}, as well as to determine how they influence the depollution parameters. It was intended, in particular, to check the range of pollutants that M_{OF-DP} is able to separate, in order to determine the type of chemical functionality of pollutants capable to establish reticulation relations with the functional structure of the composite. The reticulated functionalities are a direct measure of the depollution efficiency of the obtained material, by defining the range of separated pollutants and the extent to which the depollution was carried out. The investigation processes included various analytical techniques, in order to confirm, through complementarity (SEM, TEM, EDX, FTIR, GC-MS), the depollution parameters and the morphostructural characteristics of M_{OF-DP}, both before the testing and after its completion. The investigation results confirm the reticulation, encapsulation and degradation mechanisms of the pollutants that the authors proposed in chapters „New methods and new types of functionalised nanocomposites intended for the ecological depollution of waters” and „Nanocomposite materials with oriented functionalized structure”. The acquired experimental data have shown that the obtained M_{OF-DP} presents high pollutant separation yield and efficiency. The concentrations of most decelated pollutants and of those partially decelated in depolluted water were below the detection limit, showing the applicative potential of this type of materials. The aim of the investigations of the morphological and morphochemical structure which we have carried out was to identify and quantify the critical parameters that influence the processes of encapsulation and degradation of pollutants. The acquired experimental data indicate that M_{OF-DP} is able to reduce the level of organic pollution in surface waters by up to two orders of magnitude (the observed typical values have a separation factor of $20 \div 50 \times$). The oxide materials with a functionalised mixed structure represent a viable solution for the removal of organic pollutants in surface waters, having the most favourable applicative potential in terms of economy, depollution yield and efficiency.

6. Acknowledgements

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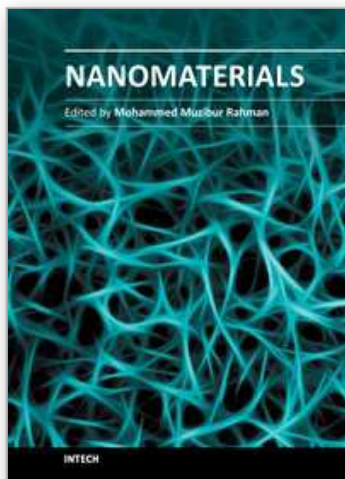
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The book "Nanomaterials" includes all aspects of metal-oxide nano-structures, nano-composites, and polymer materials instigating with materials survey and preparations, growth and characterizations, processing and fabrications, developments and potential applications. These topics have utilized innovative methods of preparation, improvement, and continuous changes in multidimensional ways. The innovative frontiers are branching out from time to time to advanced nanotechnology. It is an important booklet for scientific organizations, governmental research-centers, academic libraries, and the overall research and development of nano-materials in general. It has been created for widespread audience with diverse backgrounds and education.

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