

CONTRIBUTIONS TO THE DESIGN OF ADSORBENT MATERIALS USED FOR THE REMOVAL OF ARSENIC AND SELENIUM FROM AQUEOUS SOLUTIONS

Doctoral thesis – Abstract

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The doctoral thesis is structured in two parts, 8 chapters, respectively 182 pages.

In **the first part of the doctoral thesis, Chapters 1-3**, the literature study is presented, which refers to the identification of the current state of research in the proposed topic.

Given that water is essential for life and natural processes, our existence and our economic activities are entirely dependent on this precious resource. Moreover, globally, water is a limited resource, despite the numerous efforts and financial resources invested in finding solutions, there is a risk that millions of people around the world consume water polluted with arsenic or selenium every day.

In **Part II of the doctoral thesis**, the original contributions are presented.

The general objective of the doctoral thesis was to design, characterize and evaluate the performance of new materials based on SiO_2 , Fe_2O_3 , Al_2O_3 or ZrO_2 in order to develop an efficient and sustainable solution for the removal of arsenic and selenium ions from aqueous solutions by adsorption.

At the same time, the aim was to develop an integrated concept for the decontamination of waters contaminated with arsenic and selenium, using their adsorption on a waste in the form of sludge, originating from the groundwater treatment process, followed by the immobilization of the resulting waste in vitreous matrices.

The specific objectives of the doctoral thesis were:

- (i) synthesis of materials based on SiO_2 , Fe_2O_3 , Al_2O_3 or ZrO_2 by the coprecipitation method;
- (ii) characterization by physicochemical methods and testing of the obtained materials, in order to eliminate arsenic and selenium ions by adsorption;
- (iii) establishment of specific adsorption parameters in static regime;
- (iv) establishment of mechanisms of processes of elimination by adsorption of arsenic and selenium ions;
- (v) adsorption - desorption studies, establishing the number of adsorption-desorption cycles;

(vi) proposal of an integrated concept regarding the decontamination of waters containing arsenic and selenium, by adsorption on a waste, in the form of sludge, resulting from the process of treating deep waters for drinking purposes, which has a high iron content, followed by the immobilization of the waste in vitreous matrices.

The activities carried out to achieve the objectives of the doctoral thesis were:

- establishing the optimal method for the synthesis of materials;
- synthesis of materials based on SiO_2 , Fe_2O_3 , Al_2O_3 or ZrO_2 by the sol-gel method;
- physico-chemical characterization of the synthesized materials and of the sludge resulting from the treatment of deep water for drinking purposes, to highlight their adsorption properties, using various investigation methods, such as: X-ray diffraction, XRD, Fourier transform infrared spectroscopy, FT-IR, Raman spectroscopy, atomic force microscopy, AFM, scanning electron microscopy, SEM and energy dispersive X-ray spectroscopy, EDX, also determining the zero charge point, pH_{pZc} and structural parameters (specific surface area, pore size distribution, pore volume) from nitrogen adsorption-desorption isotherms.
- establishing the specific parameters of the adsorption process in order to eliminate arsenic and selenium ions by adsorption, in static mode, namely: solid:liquid ratio (S:L), pH, contact time, temperature, initial concentration of metal ions;
- establishing the mechanisms of the processes of elimination by adsorption of arsenic and selenium ions through kinetic, thermodynamic and equilibrium studies;
- establishing the efficiency of materials through adsorption-desorption studies, also establishing the number of cycles;
- proposing an adsorption mechanism
- proposing an integrated concept regarding the depollution of waters containing arsenic and selenium, by adsorption on a waste, in the form of sludge, which has a high iron content, by immobilization in vitreous matrices.

In **Chapter 4**, regarding the **$\text{SiO}_2/\text{Fe}_2\text{O}_3$ material** used for the removal of arsenic and selenium ions by adsorption in static mode, the following can be stated:

- ✓ The synthesis of the material was carried out by the sol-gel method.
- ✓ Subsequently, the synthesized material was physicochemically characterized by specific methods (DRX, SEM, EDX, FT-IR, AFM, Raman spectroscopy, BET, pH_{pzc} , magnetic measurements), to investigate the chemical and mechanical stability, structure, porosity, specific surface area and adsorption performances.
- ✓ Analysis of data obtained by X-ray diffraction (XRD) demonstrates the predominant presence of the specific phase - maghemite.
- ✓ Characterization by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDX) was performed to highlight the morphology of the material and demonstrate the formation of the compound. It was observed that the obtained adsorbent material presents a relatively homogeneous structure. From the EDX spectrum, the presence of Fe, O and Si atoms was observed, which confirms the formation of the $\text{SiO}_2/\text{Fe}_2\text{O}_3$ matrix.
- ✓ Characterization by Fourier transform infrared spectroscopy, FT-IR, allowed us to observe the presence of vibrations that can be attributed to metal-oxygen bonds, Si-O, but also to O-H, C=C, C-C and O-H bonds.
- ✓ Based on the images obtained by atomic force microscopy (AFM), the values for the average roughness (S_a), the root mean square roughness (S_q), the maximum peak height (S_p), the maximum valley depth (S_v), the maximum peak-to-valley height (S_y) and the average height were calculated.

- ✓ Raman spectroscopy characterization was performed to provide information about the structural fingerprint through which the material molecules can be identified, largely confirming the bonds presented by infrared spectroscopy.
- ✓ Structural parameters and pH_{pzc} were determined. Thus, by the BET method, the specific surface area was determined to be $276 \text{ m}^2/\text{g}$, the pore size has an average value of $5,564 \text{ nm}$ and the pore volume is $3,940 \times 10^{-1} \text{ cc/g}$ for pores smaller than $130,1 \text{ nm}$. The pH_{pzc} is $6,3$.
- ✓ The magnetic behavior of the $\text{SiO}_2/\text{Fe}_2\text{O}_3$ material was determined, showing superparamagnetic behavior.
- ✓ Further, studies were conducted on the removal of arsenic and selenium by adsorption in static mode. In this regard, specific parameters for the adsorption process in static mode were established, namely: the S:L ratio, pH, contact time, temperature, as well as the initial concentration of arsenic and selenium ions, respectively. The established adsorption conditions were:
 - solid:liquid ratio: $0,1 \text{ g}:25 \text{ mL}$ for both cases;
 - pH: $6-8$;
 - contact time: 60 minutes ;
 - temperature: 298 K ;
 - at a concentration of 3 mg As(V)/L , the $\text{SiO}_2/\text{Fe}_2\text{O}_3$ material reaches the maximum adsorption capacity, $q_{\max} \sim 0,575 \text{ mg/g}$, and in the case of selenium, for a concentration of 30 mg Se(IV)/L , the $\text{SiO}_2/\text{Fe}_2\text{O}_3$ material reaches the maximum adsorption capacity, $q_{\max} \sim 6,180 \text{ mg/g}$;
- ✓ The mechanism of the process of removing arsenic and selenium ions by adsorption in static mode was established by performing kinetic, thermodynamic and equilibrium studies. Thus:
 - the experimental data are very well modeled according to the pseudo-second-order kinetic model, both for As(V) and Se(IV) adsorption.
 - from the obtained thermodynamic data it is observed that in the case of both adsorption processes, ΔH^0 has a positive value, so the adsorption process is endothermic. ΔG^0 has negative values for both adsorption processes and increases in absolute value with increasing temperature, which indicates that the adsorption process is spontaneous and influenced by temperature. A positive value of ΔS^0 indicates the favorability of the adsorption process, which operates at the interface of the $\text{SiO}_2/\text{Fe}_2\text{O}_3$ material / solution with As(V) , respectively with Se(IV) .
 - the activation energy for the adsorption process of As(V) is $4,13 \text{ kJ/mol}$. In the case of the adsorption process of Se(IV) , the activation energy is $8,3 \text{ kJ/mol}$. In both adsorption cases studied, $E_a < 40 \text{ kJ/mol}$, which means that the adsorption process is physical in nature.
 - from the equilibrium data obtained, it is observed that the model that best describes the adsorption process, for both cases, is the Sips model
 - the maximum adsorption capacity of As(V) on the adsorbent material was $q_{\text{exp}} = 574,9 \text{ } \mu\text{g/g}$, close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 591,9 \text{ } \mu\text{g/g}$. In the case of Se(IV) adsorption $q_{\text{exp}} = 6180 \text{ } \mu\text{g/g}$, it is also close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 6718 \text{ } \mu\text{g/g}$.
- ✓ Subsequently, studies were carried out on the regeneration capacity of the $\text{SiO}_2/\text{Fe}_2\text{O}_3$ material, establishing the number of adsorption-desorption cycles. From the data obtained, it is observed that the adsorbent material can be reused with good efficiency for 7 adsorption/desorption cycles for As(V) removal and 8 adsorption/desorption cycles for Se(IV)

removal. Desorption studies showed that 91% of the adsorbed As(III) and 95% of the adsorbed Se(IV) can be desorbed from the material, using 5% HCl solution.

In **Chapter 5**, regarding the **SiO₂/α-Al₂O₃ material** used for the removal of arsenic and selenium ions by adsorption, in static mode, the following can be stated:

- ✓ The synthesis of the material was carried out by the sol-gel method.
- ✓ Subsequently, the synthesized material was physicochemically characterized by specific methods (DRX, SEM, EDX, FT-IR, BET, pH_{pzc}), to investigate the chemical and mechanical stability, structure, porosity, specific surface area and adsorption performance.
- ✓ Analysis of the data obtained by X-ray diffraction, XRD, demonstrates the presence of two different phases: the Al(OH)₃ phase, specific to the monoclinic crystal system with the space group: P21/n, and the SiO₂ phase, also indicating specific to the monoclinic crystal system with the space group C2.
- ✓ Characterization by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDX) was performed to highlight the morphology of the material and demonstrate the formation of the compound.
- ✓ Characterization by Fourier transform infrared spectroscopy (FT-IR), showed vibrations that were attributed to metal-oxygen bonds.
- ✓ Structural parameters and pH_{pzc} were determined;
- ✓ Specific parameters for the adsorption process in static mode were established, namely: S:L ratio, pH, contact time, temperature and initial concentration of arsenic and selenium ions;
- ✓ Analyzing the results, the adsorption conditions were established:
 - solid:liquid ratio: 0,1 g:25 mL for both cases;
 - pH:6–10;
 - contact time: 90 minutes;
 - temperature: 298 K;
 - at a concentration of 5 mg As(V)/L, the SiO₂/α-Al₂O₃ material reaches the maximum adsorption capacity, q_{max}~0,575 mg/g, and in the case of selenium, for a concentration of 7 mg Se(IV)/L, the SiO₂/α-Al₂O₃ material reaches the maximum adsorption capacity, q_{max}~1,053mg/g;
- ✓ The mechanism of the process of removing arsenic and selenium ions by adsorption in static mode was established by performing kinetic, thermodynamic and equilibrium studies. Thus:
 - the experimental data are very well modeled according to the pseudo-second-order kinetic model, both for As(V) and Se(IV) adsorption.
 - from the obtained thermodynamic data it is observed that in the case of both adsorption processes, ΔH⁰ has a positive value, so the adsorption process is endothermic. ΔG⁰ has negative values for both adsorption processes and increases in absolute value with increasing temperature, which indicates that the adsorption process is spontaneous and influenced by temperature. A positive value of ΔS⁰ indicates the favorability of the adsorption process, which operates at the interface of the SiO₂/Al₂O₃ material / solution with As(V), respectively with Se(IV).
 - the activation energy for the adsorption process of As(V) is 4,52 kJ/mol. In the case of the adsorption process of Se(IV), the activation energy is 5,22 kJ/mol. In both cases of adsorption E_a < 40 kJ/mol, which means that the adsorption process is physical in nature.
 - from the equilibrium data obtained it is observed that the model that best describes the adsorption process, for both cases, is the Sips model

- The maximum adsorption capacity of As(V) on the adsorbent material was $q_{\text{exp}} = 576,5 \mu\text{g/g}$, close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 584,9 \mu\text{g/g}$. In the case of Se(IV) adsorption, $q_{\text{exp}} = 1053,4 \mu\text{g/g}$ is also close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 1046,8 \mu\text{g/g}$.

- ✓ Subsequently, studies were carried out on the regeneration capacity of the $\text{SiO}_2/\alpha\text{-Al}_2\text{O}_3$ material, establishing the number of adsorption-desorption cycles. From the data obtained, it is observed that the adsorbent material can be reused with good efficiency for 6 adsorption/desorption cycles for As(V) removal and 6 adsorption/desorption cycles for Se(IV) removal.

In **Chapter 6**, regarding the $\text{SiO}_2/\text{ZrO}_2$ material used for the removal of arsenic and selenium ions by adsorption in static mode, the following can be stated:

- ✓ The synthesis of the material was carried out by the sol-gel method.
- ✓ Subsequently, the synthesized material was physicochemically characterized by specific methods (DRX, SEM, EDX, FT-IR, BET, pH_{pZc}), to investigate the chemical and mechanical stability, porosity structure, specific surface area and adsorption performance.
- ✓ Analysis of the data obtained by X-ray diffraction, DRX, according to the JCPDS sheet, demonstrates the presence of two different phases: the ZrO_2 phase, indicates that the material presents a monoclinic crystal system with the space group: P21/c and the SiO_2 phase, and the material presents a tetragonal crystal system with the space group P42/mnm. The crystallite size was calculated with the Scherrer equation having a value of 19,5 nm.
- ✓ Characterization by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDX) was performed to highlight the morphology of the material and demonstrate the formation of the compound.
- ✓ Characterization by Fourier transform infrared spectroscopy, FT-IR, allows the following conclusions: (i) deformation vibrations $\square$ (Si-O-Si) at wave number $\sim 470 \text{ cm}^{-1}$; (ii) symmetrical stretching vibrations $\square$ (Si-O-Si) at wave number $\sim 791 \text{ cm}^{-1}$; (iii) stretching vibrations $\nu(\text{Si-OH})$ or $\nu(\text{Si-O-M})$ at wave number 967 cm^{-1} ; (iv) asymmetrical stretching vibrations $\nu_{\text{as}}(\text{Si-O-Si})$ at wave number 1082 cm^{-1} .
- ✓ Structural parameters and pH_{pZc} were determined, so that by the BET method the specific surface area was determined to be $52,9 \text{ m}^2/\text{g}$, the pore distribution determined by the BJH method is 17,8 nm, and the pore volume is $2,832 \text{ e}^{-1} \text{ cc/g}$ for pores smaller than 168,3 nm. The pH_{pZc} is 6,35.
- ✓ Specific parameters for the adsorption process in static mode were established, namely: S:L ratio, pH, contact time, temperature and initial concentration of arsenic and selenium ions;
- ✓ Analyzing the results, the following adsorption conditions were established:
 - solid:liquid ratio: 0.1 g:25 mL for both cases;
 - pH: 6–10;
 - contact time: 90 minutes;
 - temperature: 298 K;
 - at a concentration of 6 mg As(V)/L, the $\text{SiO}_2/\text{ZrO}_2$ material reaches the maximum adsorption capacity, $q_{\text{max}} \sim 0,688 \text{ mg/g}$, and in the case of selenium, for a concentration of 6 mg Se(IV)/L, the $\text{SiO}_2/\text{ZrO}_2$ material reaches the maximum adsorption capacity, $q_{\text{max}} \sim 1,007 \text{ mg/g}$;
- ✓ The mechanism of the process of removing arsenic and selenium ions by adsorption in static mode was established by performing kinetic, thermodynamic and equilibrium studies. Thus:

- the experimental data are very well modeled according to the pseudo-second-order kinetic model, both for As(V) and Se(IV) adsorption.

- from the obtained thermodynamic data it is observed that in the case of both adsorption processes, ΔH^0 has a positive value, the adsorption process being endothermic. ΔG^0 has negative values for both adsorption processes and increases in absolute value with increasing temperature, which indicates that the adsorption process is spontaneous and influenced by temperature. The positive value of ΔS^0 indicates the favorability of the adsorption process, which operates at the interface of the SiO₂/ZrO₂ material / solution with As(V), respectively with Se(IV).

- the activation energy for the adsorption process of As(V) is 5,10 kJ/mol. In the case of the adsorption process of Se(IV), the activation energy is 1,88 kJ/mol. In both cases of adsorption $E_a < 40$ kJ/mol, which means that the adsorption process is physical in nature.

- from the equilibrium data obtained it is observed that the model that best describes the adsorption process, for both cases, is the Sips model

- the maximum adsorption capacity of As(V) on the adsorbent material was $q_{\text{exp}} = 688,7$ $\mu\text{g/g}$, close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 689,9$ $\mu\text{g/g}$. In the case of Se(IV) adsorption, $q_{\text{exp}} = 1.007,5$ $\mu\text{g/g}$, close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 1.110,28$ $\mu\text{g/g}$.

- ✓ Subsequently, studies were carried out on the regeneration capacity of the SiO₂/ZrO₂ material, establishing the number of adsorption-desorption cycles. From the data obtained, it is observed that the adsorbent material can be reused with good efficiency for 5 adsorption/desorption cycles for As(V) removal and 6 adsorption/desorption cycles for Se(IV) removal.

In **Chapter 7**, the removal of arsenic and selenium from water was investigated using static adsorption techniques using as **adsorbent material a waste, sludge resulting from the treatment of groundwater for drinking purposes, having a substantial iron composition**.

The following can be stated:

- ✓ The composition of the sludge was established, observing a high iron content (43,2%), thus being able to state that the respective waste is a possible adsorbent material for the removal of arsenic and selenium from aqueous solutions.
- ✓ The unconventional material was physicochemically characterized by specific methods (SEM, EDX, FT-IR, BET, pH_{pzc}), to investigate the chemical and mechanical stability, structure, porosity, specific surface area and adsorption performance.
- ✓ Characterization by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDX), shows peaks corresponding to the chemical elements that make up the composition of the material, namely iron (Fe), manganese (Mn) and calcium (Ca), in significant proportions.
- ✓ by Fourier transform infrared spectroscopy (FT-IR) showed the following: two vibrations associated with the –OH group were observed at wave numbers 3.387 cm^{-1} and 1.634 cm^{-1} . In addition, the appearance of a specific peak at wave number 478 cm^{-1} was correlated with the stretching vibrations corresponding to the Fe-O bond, indicating the presence of iron oxides and hydroxides.
- ✓ Structural parameters and pH_{pZc} were determined, so that by the BET method the specific surface area was determined to be $117\text{ m}^2/\text{g}$, the pore distribution determined by the BJH method is $3,974\text{ nm}$ and the pore volume is $,99\text{e}^{-1}\text{ cc/g}$ for pores smaller than $145,9\text{ nm}$. The pH_{pZc} is 7,65.

- ✓ Specific parameters for the adsorption process in static mode were established, namely: S:L ratio, pH, contact time, temperature and initial concentration of arsenic and selenium ions;
 - ✓ Analyzing the results, the adsorption conditions are:
 - solid:liquid ratio: 0.1 g:25 mL for both cases;
 - pH: 6–10;
 - contact time: 90 minutes;
 - temperature: 298 K;
 - at a concentration of 350 mg As(V)/L, the material reaches maximum adsorption capacity, $q_{\max} \sim 70$ mg/g, and in the case of selenium, for a concentration of 600 mg Se(IV)/L, the material reaches maximum adsorption capacity, $q_{\max} \sim 100$ mg/g;
- ✓ The mechanism of the process of removing arsenic and selenium ions by adsorption in static mode was established by performing kinetic, thermodynamic and equilibrium studies. Thus:
 - the experimental data are very well modeled according to the pseudo-second-order kinetic model, both for As(V) and Se(IV) adsorption.
 - from the obtained thermodynamic data it is observed that in the case of both adsorption processes, ΔH^0 has a positive value, so the adsorption process is endothermic. ΔG^0 has negative values for both adsorption processes and increases in absolute value with increasing temperature, which indicates that the adsorption process is spontaneous and influenced by temperature. Positive value of ΔS^0 indicates the favorability of the adsorption process, which operates at the material - solution interface with As(V), respectively with Se(IV).
 - the activation energy for the adsorption process of As(V) is 8,15 kJ/mol. In the case of the adsorption process of Se(IV), the activation energy is 6,48 kJ/mol. In both adsorption cases, $E_a < 40$ kJ/mol, which means that the adsorption process is physical in nature.
 - from the equilibrium data obtained it is observed that the model that best describes the adsorption process, for both cases, is the Sips model
 - the maximum adsorption capacity of As(V) on the adsorbent material was $q_{\exp} = 70,2$ mg/g close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 69,8$ mg/g. In the case of Se(IV) adsorption, $q_{\exp} = 100,2$ mg/g close to the value obtained by modeling the data with the Sips model, $q_{\text{calc}} = 99,82$ mg/g.
- ✓ Subsequently, studies were carried out on the regeneration capacity of the material, establishing the number of adsorption-desorption cycles. From the data obtained, it is observed that the adsorbent material can be reused with good efficiency for 5 adsorption/desorption cycles for As(V) removal and 5 adsorption/desorption cycles for Se(IV) removal.

The waste resulting from the removal of arsenic or selenium from water was utilized by immobilization in glass matrices to obtain decorative crystallized glazes. The unconventional material used was a sludge/waste with a high iron content, originating from the treatment of groundwater for drinking purposes.

- ✓ Subsequently, it was found that the spent sludge, resulting from the adsorption process, contains a quantity of ~ 350 mg/L As(V), respectively 600 mg/L Se(IV). This was utilized by immobilization in glass materials in order to obtain decorative glazes. To obtain the glass mass, 5 mixtures of spent sludge: glass were made.
- ✓ In order to study the stability of As(V), respectively Se(IV) in the obtained vitreous mass, leaching tests were performed at different pHs (5,5; 7 and 8,5), the pH being adjusted with acetic acid or ammonia solution. No hazardous species were observed in the solutions

resulting from the leaching, so it can be concluded that the spent adsorbent containing arsenic and selenium was successfully immobilized in the vitreous matrix. The obtained frit was further processed in order to obtain decorative glazes.

The process of inertization of hazardous waste by vitrification (encapsulation in glass matrices) is recognized as one of the safest and most efficient methods of final management of industrial waste.

Vitrification ensures the chemical immobilization of toxic contaminants (such as heavy metals or metalloids such as arsenic or selenium) by integrating them into the amorphous network of glass. The glass matrix offers superior chemical stability to any other solidified material, minimizing the rate of release of pollutants into the environment in the long term. The final glass product is dense, non-porous and mechanically resistant, being optimal for final disposal.

Although the main purpose is inertization, the glass mass obtained can, in certain cases, be used as a secondary raw material in the construction materials industry or as aggregates, thus supporting the objectives of the circular economy.

In conclusion, inertization in glass matrices represents the cutting-edge technical solution for transforming hazardous waste into a stable material, directly contributing to the protection of the environment and human health.

The experimental data obtained for the removal of arsenic and selenium by adsorption on various forms of materials with adsorbent properties are presented in Table 1.

Table 1. Experimental data obtained for the removal of arsenic and selenium by adsorption on various forms of materials with adsorbent properties

Adsorbent material	Initial concentration, mg/L		Adsorption capacity, mg/g	
	As(V)	Se(IV)	As(V)	Se(IV)
SiO ₂ /Fe ₂ O ₃	3	30	0,575	6,18
SiO ₂ /α-Al ₂ O ₃	5	7	0,574	1,053
SiO ₂ /ZrO ₂	6	8	0,688	1,007
Unconventional material/ Sludge	350	600	70	100

It can be stated, based on the resulting data, that the unconventional material presents the best adsorption capacity for the removal of arsenic and selenium by adsorption from aqueous media. At the same time, the solution found for the immobilization of spent sludge in vitreous matrices represents a viable solution that falls within the direction of closed-cycle technologies.

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The original research presented in this doctoral thesis resulted in the publication of 3 papers in journals indexed in Web of Science (Materials (Q2)-Impact Factor 3,2; Gels(Q1)-Impact Factor 5,3 and Toxics(Q1)-Impact Factor 4,1).

The H-index is 3 and the cumulative factor (FIC) is 12,6.

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