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**<sup>90</sup>Sr ADSORPTION FROM THE AQUATIC ENVIRONMENT  
OF CHORNOBYL EXCLUSION ZONE BY CHEMICALLY ENHANCED TiO<sub>2</sub>**

This paper describes the testing of titanium dioxide, chemically modified by arsenate groups, as an adsorbent of <sup>90</sup>Sr from the component of aquatic ecosystems of the Chernobyl exclusion zone. It is shown, that the chemical composition of the aquatic environment impacts <sup>90</sup>Sr adsorption. The 4As-TiO<sub>2</sub> adsorbent reduces the activity of some samples by almost 100 %, which indicates selectivity and high adsorption capacity of the adsorbent in relation to <sup>90</sup>Sr. In some experiments, this value reached 100 %, and the activity was reduced to the level of the maximum permissible <sup>90</sup>Sr concentration.

*Keywords:* Chernobyl exclusion zone, fish, scale, specific activity, <sup>90</sup>Sr, TiO<sub>2</sub>.

**1. Introduction**

<sup>90</sup>Sr is an anthropogenic radionuclide, progeny of uranium nuclear fission. It may enter the environment as the result of a nuclear weapons test, with nuclear power plant emissions, or as a result of nuclear accidents. This radionuclide has a half-life of  $28.8 \pm 0.07$  years. If <sup>90</sup>Sr gets into the environment or a living organism, its harmful effect will continue for a long time. Therefore, monitoring <sup>90</sup>Sr content in the environment is extremely important.

Modification of TiO<sub>2</sub> surface by phosphate, carbonate, or arsenate groups for enhancing adsorption capacity toward strontium cations was described in our previous works [1 - 3]. Surface characteristics of adsorbents, their adsorption capacity, and determination of a mechanism of interaction of strontium cations with chemically modified TiO<sub>2</sub> samples were investigated in detail. The studies described in [1 - 3] were conducted with stable isotopes dissolved in distilled water, i.e. under conditions close to ideal. It is known that the chemical properties of all isotopes of the same element are identical, but high effects of radioactive decay and different chemical compositions of natural aquatic ecosystems can significantly affect the <sup>90</sup>Sr adsorption.

This paper describes the testing of titanium dioxide, chemically modified by arsenate groups, as an adsorbent of <sup>90</sup>Sr from the aquatic ecosystems of the Chernobyl NPP exclusion zone.

<sup>90</sup>Sr, once in an aquatic environment, gets involved in biogeochemical cycles and migrates through the food chain, effectively accumulating in the upper trophic levels, mostly occupied by fish. These processes are particularly relevant in the Chernobyl exclusion zone (ChEZ). Crucian carp

(*Carassius gibelio* Bloch) was selected as the object of the experiment. The characteristics of the crucian carp from the Verzhina Lake, including the content of <sup>90</sup>Sr in the crucian carp, are described in publications [4 - 6]. It should be noted that the crucian carp from Verzhina Lake is the most radioactive fish among all species in the reservoirs of the NPP Exclusion zone. Its radioactivity is 1960 - 4970 times above permissible levels of <sup>90</sup>Sr in fishery products in Ukraine (35 Bq·kg<sup>-1</sup>) [5 - 7]. For reference, the fish from the Chernobyl NPP cooling pond is only 6 - 41 times more radioactive than the permissible radiation level. <sup>90</sup>Sr is a chemical analog of calcium, so it accumulates mainly in scale and bones. Our experiments were conducted to investigate the possibility of adsorption of strontium (<sup>90</sup>Sr), extracted from organs and tissues of fish from highly contaminated reservoirs.

**2. Experimental technique****2.1. Characteristics of the adsorbent**

Anatase TiO<sub>2</sub> chemically modified by arsenate groups was selected as the adsorbent. In the publication by Mironyuk et al. [1], it has been shown that surface modification of anatase TiO<sub>2</sub> increases both its surface area and the volume of meso-pores (Table 1), which is important for the adsorption of cations with a large ionic radius. The surface modification also shifts the point of zero charges, which causes a greater adsorption activity of the material toward cations. In this case, the adsorbent with 4 wt % of arsenate groups in the structure (4As-TiO<sub>2</sub>) has the best adsorption properties for strontium cations. The high adsorption ability of 4As-TiO<sub>2</sub> is caused by the highest parameter of meso-pore area ( $S_{\text{meso}}, \text{m}^2\text{g}^{-1}$ ), meso-pore volume ( $V_{\text{meso}}, \text{cm}^3\text{g}^{-1}$ ), and maximum amount of  $\equiv \text{TiOH}^{\delta+}$  adsorption centers on the surface [1].

**Table 1. Textural characteristics, such as pore volume, pore area, and surface area of unmodified and modified TiO<sub>2</sub>, according to [1]**

Adsorbent	S <sub>BET</sub> , m <sup>2</sup> g <sup>-1</sup>	S <sub>micro</sub> , m <sup>2</sup> g <sup>-1</sup>	S <sub>meso</sub> , m <sup>2</sup> g <sup>-1</sup>	V <sub>p</sub> , cm <sup>3</sup> g <sup>-1</sup>	V <sub>micro</sub> , cm <sup>3</sup> g <sup>-1</sup>	V <sub>meso</sub> , cm <sup>3</sup> g <sup>-1</sup>
TiO <sub>2</sub>	349	129	230	0.191	0.061	0.130
2As-TiO <sub>2</sub>	395	232	163	0.223	0.106	0.115
4As-TiO <sub>2</sub>	<b>396</b>	<b>139</b>	<b>257</b>	<b>0.256</b>	<b>0.071</b>	<b>0.184</b>
8As-TiO <sub>2</sub>	405	221	183	0.236	0.101	0.133

Note. S<sub>BET</sub> – is surface area calculated using Brunauer - Emmet - Teller equation, m<sup>2</sup>g<sup>-1</sup>; V<sub>p</sub> – is pore volume measured by the method of the low temperature of nitrogen adsorption/desorption, cm<sup>3</sup>g<sup>-1</sup>.

## 2.2. <sup>90</sup>Sr adsorption study and analysis

Crucian carp (age 2+ – 4+, from Vershina Lake, for August 2019) was selected as the object of the experiment. <sup>90</sup>Sr contained in the crucian carp (*Carassius gibelio* Bloch), and separately in its scale, was extracted, by the oxalate technique, using hydrochloric acid. This procedure is described in detail in publications [7 - 9]. In brief, the concept of the technique is based on precipitation of rare earth elements and strontium oxalates at pH = 4 to separate <sup>90</sup>Sr from <sup>137</sup>Cs and <sup>40</sup>K; conversion of oxalates to dissolved state and subsequent separation of <sup>90</sup>Sr from <sup>90</sup>Y in an alkaline medium. The determination of <sup>90</sup>Sr content was performed by radiochemical methods involving the measurement of the daughter <sup>90</sup>Y using radiometer UMF-2000 (“Dose”) [9]. 0.1, 0.3, and 1.0 g of acid extracts from scale, and 0.5, 1.0, and 5.0 g of residue fish flesh (without scale) were taken for analysis. Adsorption studies were carried out in an alkaline medium (pH = 9) for 2 h in a batch mode. Activity concentrations of the samples (<sup>90</sup>Sr activity) were measured before and after the adsorption. The arithmetic average error was ~ 10 %.

Fish material for research was sampled within the framework of the radioecological monitoring of aquatic communities by the State Specialized Enterprise “Ecocentre” of the State Agency of Ukraine on Exclusion Zone Management.

Energy-dispersive (EDS) analysis of adsorbent samples with adsorbed strontium on the surface was performed. Test studies of adsorption of calcium cations by 4As-TiO<sub>2</sub> were conducted to determine the possibility of calcium adsorption by investigated samples. The magnitude of adsorption of strontium ions by 4As-TiO<sub>2</sub> at pH = 9 from distilled water, with solid-to-liquid phase ratio (L: S) = 200, was determined as the highest possible percentage of extraction of strontium cations in an alkaline medium. The high adsorption of strontium by the investigated adsorbent was confirmed by XRF analysis (Bruker AXS, Karlsruhe, Germany). The initial and residual concentrations of Ca<sup>2+</sup> and Sr<sup>2+</sup> were determined using direct

complexometric titration with Eriochrom Black T as an indicator, as described in publications [1 - 3, 10]. Adsorption value, as well as removed strontium percentage, which was removed (stable isotopes and <sup>90</sup>Sr radionuclides), were calculated according to [10, 11] using equations (1) - (4) as shown below:

$$q_e = \frac{[(C_o - C_e)V]}{m}, \quad (1)$$

$$\% \text{ removal} = \frac{(C_o - C_e) \cdot 100}{C_o}, \quad (2)$$

where  $q_e$  – is adsorption value, mg·g<sup>-1</sup>;  $C_o$  and  $C_e$  – are initial and residual concentrations of strontium in solution, respectively, mg·L<sup>-1</sup>;  $V$  – is solution volume, L;  $m$  – is dried mass of used adsorbent, g.

$$\% \text{ Sr} = \frac{A_i - A_f}{A_i} \cdot 100, \quad (3)$$

$$\text{Ads A} = A_i - A_f, \quad (4)$$

where  $A_i$  – is initial samples activity, Bq;  $A_f$  – is final sample activity, Bq;  $\text{Ads A}$  – is the activity, adsorbed by 4As-TiO<sub>2</sub>;  $\text{Res A} = A_f$ ; %Sr – is the percent of <sup>90</sup>Sr uptaken.

## 3. Results and discussion

### 3.1. Adsorption of strontium cations by TiO<sub>2</sub> in alkali medium

The results of determining strontium adsorption values from aqueous solutions (pH = 9) by 4As-TiO<sub>2</sub> are shown in Fig. 1 and Table 2. As the duration of the interaction of the 'SrCl<sub>2</sub> solution – 4As-TiO<sub>2</sub> surface' system exceeds 2 h almost 100 % strontium is adsorbed from distilled water. These results have been confirmed by XRF analysis (Fig. 2). The solubility of calcium phosphate phases in aqueous solution is an important property and mainly correlated with the calcium (Ca)/phosphorous (P) ratio.

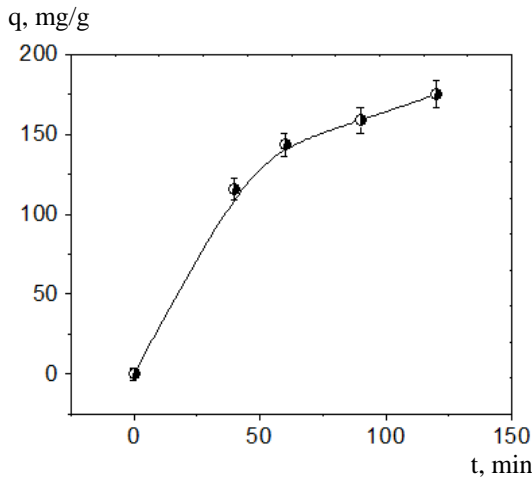


Fig. 1. Dependence of Sr<sup>2+</sup> adsorption by 4As-TiO<sub>2</sub> on agitation time in alkaline medium. Conditions of experiment: solution of SrCl<sub>2</sub> in distilled water, V<sub>Sr</sub> = 10 mL; N<sub>Sr</sub> 0.005 mol/L, L : S = 200.

Table 2. Sr<sup>2+</sup> adsorption by 4As-TiO<sub>2</sub> in alkaline medium

t, min	m Sr <sup>2+</sup> , mg	q, mg/g	q, mmol/g	Sr, %
0	0	0	0	10 – 99.73
40	5.785	115.7 ± 7	1.315	
60	7.179	143.59 ± 7.3	1.63	
90	7.93	158.6 ± 8.5	1.8	
120	8.75	175.5 ± 8.7	1.98	

Note. m Sr<sup>2+</sup> – mass of adsorbed strontium, mg; q – adsorption value, mg/g or mmol/g; Sr – percent of uptaken strontium, %.

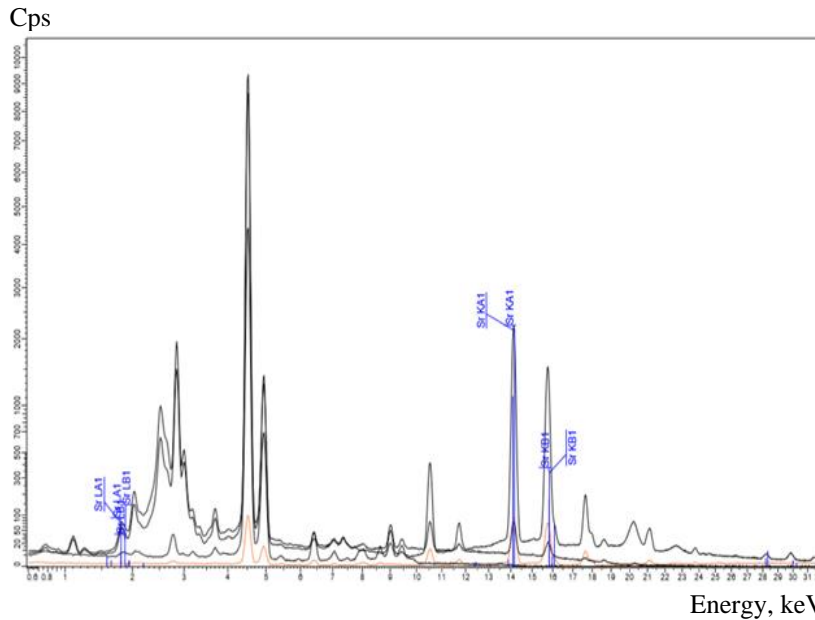


Fig. 2. XRF spectrum of 4As-TiO<sub>2</sub> with adsorbed strontium from the alkali medium.

EDS analysis of adsorbent samples with adsorbed strontium on the surface was performed. EDS spectrum and the outside of 4As-TiO<sub>2</sub> are shown in Fig. 3.

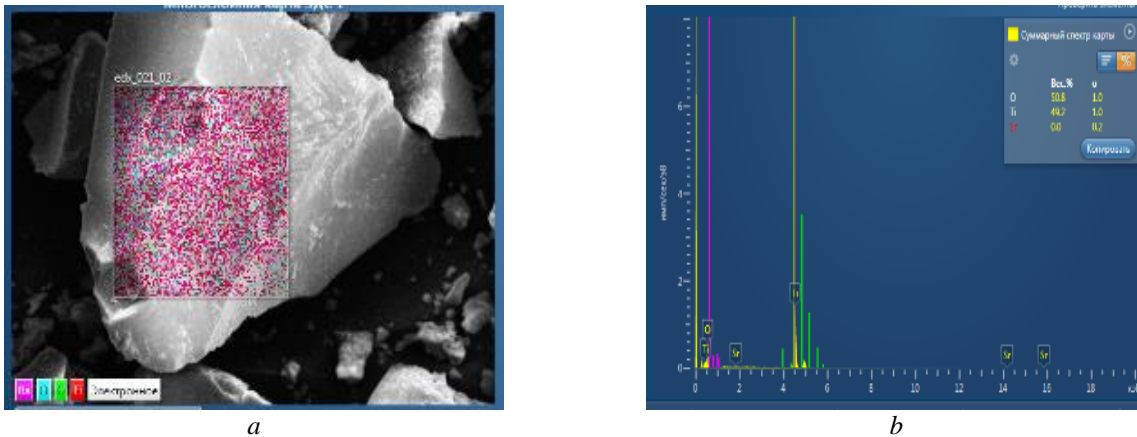


Fig. 3. Scanning electron microscope images of 4As-TiO<sub>2</sub> surface (a); EDS spectrum of 4As-TiO<sub>2</sub> with adsorbed strontium traces (b).

### 3.2. Adsorption of radioactive $^{90}\text{Sr}$ by $\text{TiO}_2$

$^{90}\text{Sr}$  accumulates in fish scale more than in other organs.  $^{137}\text{Cs}$ , on the contrary, accumulates mainly in the fish flesh [12]. The arithmetic mean of fish scale radioactivity is  $441 \cdot 10^3 \text{ Bq} \cdot \text{kg}^{-1}$ , which corresponds to  $0.87 \cdot 10^{-10} \text{ g}$  of  $^{90}\text{Sr}$  per 1 g of the investigated sample.

The arithmetic mean of residue fish (without scale) activity is  $68 \cdot 10^3 \text{ Bq} \cdot \text{kg}^{-1}$ , which corresponds to nearly  $0.134 \cdot 10^{-10} \text{ g}$  of  $^{90}\text{Sr}$  per 1 g of the investigated sample. Changes in the activity of  $^{90}\text{Sr}$  in the sample before adding the adsorbent and after the adsorption process are shown in Table 3.

Table 3. The activity of  $^{90}\text{Sr}$  in the sample before adding the adsorbent, and after the adsorption process

Acid extracts of bio-samples of different masses	m $\text{TiO}_2$ , g	$A_i$ $^{90}\text{Sr}$ , Bq	$A_f$ $^{90}\text{Sr}$ , Bq	Residual $^{90}\text{Sr}$ , %	Ads A, Bq	Uptaken $^{90}\text{Sr}$ , %
0.1 g of scale	5	44	1.7	4.5	42	95.5
0.3 g of scale	1	132	58	44	74	56.2
1.0 g of scale	1.5	441	222	51	219	49
0.5 g of residue fish without scale	5	34	0.38	1	34	98.9
1 g of residue fish without scale	5	68	1.9	3	66	97
5 g of residue fish without scale	1.5	339	148	43.7	191	56.2

The results in Table 3 indicate the effectiveness of  $4\text{As-TiO}_2$  adsorbent toward  $^{90}\text{Sr}$ . The activity of the solution after using  $4\text{As-TiO}_2$  decreased at least twice in all experiments. As mentioned above, the concentration of  $^{90}\text{Sr}$  in the scale was  $0.87 \cdot 10^{-7} \text{ mg} \cdot \text{g}^{-1}$  and in residual fish without scale  $0.134 \cdot 10^{-7} \text{ mg} \cdot \text{g}^{-1}$ . The probability of interaction of two elements (systems:

$^{90}\text{Sr}$  – adsorption center  $\equiv \text{TiON}^{\delta+}$ ) is proportionate to their concentrations. At low concentration ( $0.87 \cdot 10^{-7} \text{ mg} \cdot \text{g}^{-1}$ ), the probability of interaction  $^{90}\text{Sr}$  – adsorption center  $\equiv \text{TiOH}^{\delta+}$  [1] began to decrease. Therefore, the mass of adsorbent had to be increased [1 - 3, 11].

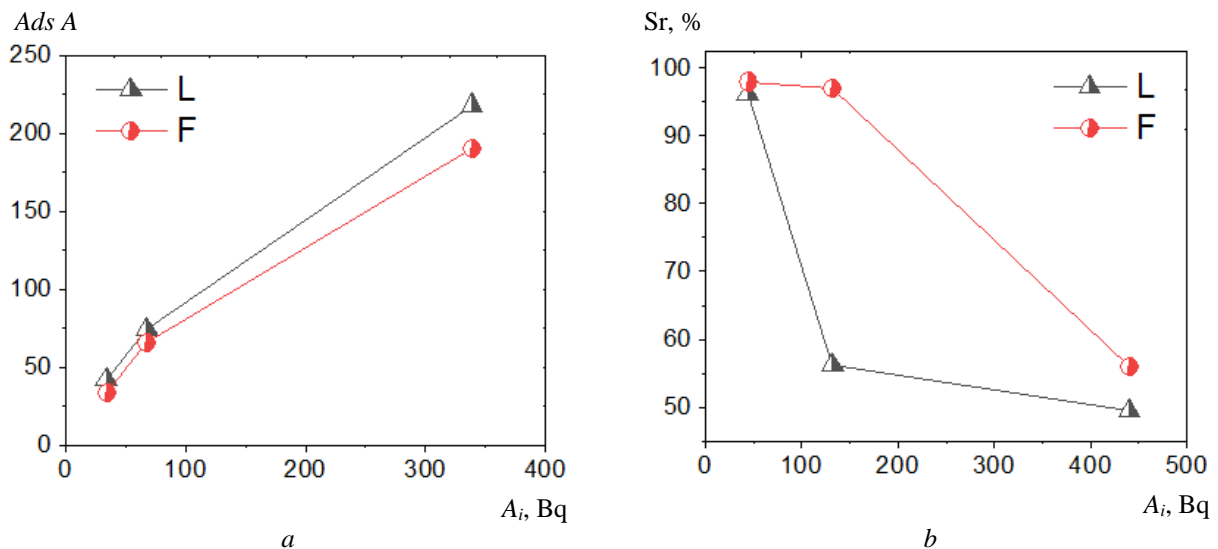


Fig. 4. Dependence of adsorbed activity  $Ads A$  (a) and percent of uptaken  $^{90}\text{Sr}$  (b) on initial activity  $A_i$  of  $^{90}\text{Sr}$  in the scale samples of *Carassius gibelio* (L) and flesh, of *Carassius gibelio* without a scale (F).

The results in Table 3 and Fig. 4 illustrate an increase of  $^{90}\text{Sr}$  adsorption, with the increase of initial activity of the samples. The percentage of  $^{90}\text{Sr}$  adsorbed is greater from the extracts of residue fish without a scale (see Fig. 3, b) than from the scale extract. In some experiments, this value reached 100 %, and the activity was reduced to the level of the maximum permissible  $^{90}\text{Sr}$  concentration.

### 3.3. Influence of chemical composition of scale on $^{90}\text{Sr}$ adsorption

Concentrations of the order of  $10^{-10} \text{ g} \cdot \text{L}^{-1}$  are micro-concentrations and any chemical components of the aqueous medium compared to  $^{90}\text{Sr}$  will be in excess. It is known that scale is modified bones [12, 13] and that their chemical composition partially depends on fish life conditions (climate, food, water). Bone

contains protein molecules, collagen, but the major mineral components of the scale are  $\text{Ca}^{2+}$ ,  $\text{PO}_4^{3-}$  or calcium salts with phosphate anions, for example, salts of hydro-oxy-apatite,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$  [14]. Calcium phosphates include various salts of tribasic phosphoric acid ( $\text{H}_3\text{PO}_4$ ).  $\text{H}_2\text{PO}_4^-$ ,  $\text{HPO}_4^{2-}$ , or  $\text{PO}_4^{3-}$  ions, which can all be formed under different pH va-

lues [15]. The main calcium phosphate compounds and their solubility according to literature data [15, 16] are provided in Table 4. This compound can assume a colloidal state when dissolved. For example, in milk, calcium salts with phosphate anions exist in the colloidal form and can bind  $\text{Zn}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Sr}^{2+}$ .

**Table 4. Chemical composition and abbreviation of calcium phosphate compounds with the different calcium (Ca)/phosphorous (P) ratio**

Name	Abbreviation	Formula	Ca/P ratio
Monocalcium phosphate monohydrate	MCPM	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$	0.5
Dicalcium phosphate anhydrate (monetite)	DCPA	$\text{CaHPO}_4$	1.0
Dicalcium phosphate dihydrate (brushite)	DCPD	$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$	1.0
Octacalcium phosphate	OCP	$\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$	1.33
$\beta$ -Tricalcium phosphate	$\beta$ -TCP	$\text{Ca}_3(\text{PO}_4)_2$	1.5
Amorphous calcium phosphate	ACP	$\text{Ca}_3(\text{PO}_4)_2 \cdot n\text{H}_2\text{O}$	1.5
$\alpha$ -Tricalcium phosphate	$\alpha$ -TCP	$\alpha\text{-Ca}_3(\text{PO}_4)_2$	1.5
Hydroxyapatite	HA	$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$	1.67
Tetracalcium phosphate	TetCP	$\text{Ca}_4(\text{PO}_4)_2\text{O}$	2.0

The oxalate technique widely used in the analysis of <sup>90</sup>Sr (also used in this work), is intended to separate <sup>90</sup>Sr from other  $\beta^-$ -emitters, such as <sup>137</sup>Cs and <sup>40</sup>K, from the daughter <sup>90</sup>Y (also the  $\beta^-$ -emitter) and heavy polyvalent radionuclides of lead, uranium, etc.

Stable calcium does not interfere with the determination of <sup>90</sup>Sr, and therefore it is not necessary to separate  $\text{Ca}^{2+}$  from <sup>90</sup>Sr. At the same time, the redox potential of calcium ions, the sum of the first two ionization potentials, and the ionic radius of calcium are very close to the corresponding characteristics of strontium ions [17]. This determines the similarity of

their chemical properties. For example, in the procedure for determining strontium in milk [9, 18], calcium oxalate is used as a carrier for <sup>90</sup>Sr. Therefore, bivalent calcium influences the <sup>90</sup>Sr adsorption by  $\text{TiO}_2$  from aquatic ecosystems. Adsorbents based on titanium dioxide can adsorb various divalent cations [10, 19]. Since the adsorption of calcium ions by this adsorbent has not been investigated yet, test studies of the adsorption of  $\text{Ca}^{2+}$  cations by 4As-TiO<sub>2</sub> (Table 5) were conducted. Shown, that 4As-TiO<sub>2</sub> also adsorbed calcium cations, but adsorption values of  $\text{Ca}^{2+}$  are lower than adsorption values of strontium in the same conditions.

**Table 5. Adsorption of strontium and calcium ions from their 0.01M chloride solutions by adsorbents based on titanium dioxide in neutral medium**

Element/adsorbent	2As-TiO <sub>2</sub>		4As-TiO <sub>2</sub>		8As-TiO <sub>2</sub>	
	q, mg/g	q, mmol/g	q, mg/g	q, mmol/g	q, mg/g	q, mmol/g
$\text{Sr}^{2+}$	53.8	0.6114	66.54	0.7562	47.97	0.5452
$\text{Ca}^{2+}$	20	0.50	23.2	0.58	23.2	0.58

On the other hand, salts of calcium with phosphate anions reduce the adsorption of <sup>90</sup>Sr and <sup>226</sup>Ra according to [20, 21]. Colloidal compounds of calcium with phosphate anions are capable of adsorption of strontium, uranium, and some other heavy and transition metals on their surface [21].

Adsorption of <sup>90</sup>Sr by 4As-TiO<sub>2</sub> occurs in the presence of excess calcium cations (extracted from scale) under the investigated conditions, simultaneously with the adsorption of <sup>90</sup>Sr by calcium phosphate micro-colloids. All the above notwithstanding, 4As-TiO<sub>2</sub> adsorbent reduces the activity of some samples by almost 100 % (Table 6), which indicates selectivity and high adsorption capacity of investigated adsorbent toward <sup>90</sup>Sr.

**Table 6. Dependence of the percentage of adsorbed <sup>90</sup>Sr on the mass of the adsorbent**

Adsorbent's mass, g	Bio-object	$A_i$ , Bq	$A_f$ , Bq	Ads A, Bq	<sup>90</sup> Sr, %
1	0.5 g of residue fish without scale	34	14	20	59
5		34	0.38	34	99

#### 4. Conclusions

The possibility of adsorption of <sup>90</sup>Sr extracted from *Carassius gibelio* by TiO<sub>2</sub> based adsorbent was investigated. The adsorption of <sup>90</sup>Sr onto TiO<sub>2</sub> reaches

99 % with activities of  $^{90}\text{Sr}$  in the range of 34 - 68 Bq per sample, from the extracts of residue fish without scale. By changing some of the external conditions of the experiment, such as increasing the duration of the interaction, we were able to achieve 100 % removal of  $^{90}\text{Sr}$  from acidic extracts of natural organic objects of the aquatic environment, and the  $^{90}\text{Sr}$  activity will reduce to the level of the maximum permissible  $^{90}\text{Sr}$

concentration in the fishery products [22].  $\text{TiO}_2$  is promising in developing new methods of concentrating long-lived radionuclides, one of which is  $^{90}\text{Sr}$ . Using the  $\text{TiO}_2$  with surface chemically modified by arsenate groups can increase the efficiency of methods of analysis of  $^{90}\text{Sr}$  in the aquatic environment.

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### АДСОРБЦІЯ <sup>90</sup>Sr З ОБ'ЄКТІВ ВОДНИХ ЕКОСИСТЕМ ЧОРНОБИЛЬСЬКОЇ ЗОНИ ВІДЧУЖЕННЯ ХІМІЧНО МОДИФІКОВАНИМ TiO<sub>2</sub>

Описано використання діоксиду титану з хімічно прищепленими арсенатними угрупованнями для адсорбції <sup>90</sup>Sr, вилученого з біооб'єктів водних екосистем чорнобильської зони відчуження. Показано, що на адсорбцію <sup>90</sup>Sr впливає хімічний склад біооб'єктів. Адсорбент 4As-TiO<sub>2</sub> зменшує активність деяких проб практично на 100 %. При цьому активність досліджених зразків зменшується до рівня гранично допустимої концентрації <sup>90</sup>Sr. Це свідчить про селективність дослідженого адсорбенту і високу адсорбційну здатність щодо <sup>90</sup>Sr.

*Ключові слова:* чорнобильська зона відчуження, риби, питома активність <sup>90</sup>Sr, TiO<sub>2</sub>.

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