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In situ synthesis of copper ferrocyanide by solid-phase reactions for the extraction of cesium ions from low-level radioactive waters

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Abstract

The article presents the results of in situ synthesis of copper ferrocyanide by the powder method – solid-phase synthesis. The obtained sorbent is characterised by a cesium ion removal coefficient of up to 81.4% with a sediment activity of 146 Bq after 3 days of exposure. The storage capacity of sorbent sample 1 is manifested in the sorption of 137Cs1+ ions on the surface of copper ferrocyanide particles due to hydroxyl groups with spherical formations of bright beet-coloured compounds, such as $Cs_2[Fe(CN)_6]\cdot 7H_2O$. It has been shown that the addition of 10% Portland cement to the sorbent reduces the removal of caesium ions from water (to 70.3%) with sediment activity after 3 days of exposure to 125 Bq. The addition of Portland cement promotes rapid precipitation and chemical binding of cesium ions into compounds of the type $Cs_2O\cdot 2CaO\cdot 2SiO_2\cdot 4H_2O$. The prospects for the application of the developed sorbent are evident in the simplicity of synthesis, low cost due to the use of inexpensive commercially available reagents, adsorption rate, high adsorption capacity and high selectivity of cesium ion removal from low-level radioactive water.

Key words: activity, cesium ions, copper ferrocyanide, extraction, in situ, low-level radioactive waters, sediment, solid-phase reactions, sorbent

Introduction

Ukraine has a powerful nuclear industry that includes all components of the nuclear fuel cycle (NFC) – from uranium mining, primary enrichment at mining and processing plants to nuclear energy production at 15 nuclear power plant units, as well as an underdeveloped reprocessing system and a low-efficiency radioactive waste disposal system from NFC facilities.

Every year, Ukraine generates tens of millions of tonnes of Class 1 hazardous waste, namely waste from uranium mining and enrichment and waste from the operation of nuclear power plants. A single nuclear power plant generates about 28 tonnes of various types of radioactive waste, of which about 20 tonnes is spent nuclear fuel (SNF), which is a valuable secondary raw material. SNF contains up to 97% of unused nuclear materials, which are effectively used in next-generation reactors. Today, there are already problems with the storage of SNF (cubic residue of about 9,000 thousand m3, salt solution of about 3,000 thousand tonnes) and long-lived radioactive waste (up to 80 thousand m3).

The issues of safe and cost-effective spent fuel management and the creation of a geological repository for long-lived high-level waste remain unresolved. In addition, due to military and terrorist provocations in the eastern regions by the Russian Federation, as well as cases of terrorist sabotage in other regions of Ukraine, the problems of radiation hazards have become even more acute. Therefore, based on the threats and risks, there is an urgent need to develop new innovative and effective complex sorbents with high selectivity to caesium ions for the treatment of low- and intermediate-level liquid radioactive waste.

Literary review and problem statement

According to data presented in works,[1–3] transition metal ferrocyanides (Ni^{2+}, Co^{2+} and Cu^{2+}) are common materials used for radionuclide sorption in the nuclear industry. Firstly, they are characterised by high selectivity to caesium ions. They can sorb rubidium, strontium, thallium and other elements. Their main characteristics include high sorption capacity, specific surface area and porosity, good radiation stability, low solubility and ease of synthesis. Copper cation ferrocyanides demonstrate higher sorption capacity for caesium-137 compared to other types of metal cations, which is extremely important in the field of environmental remediation and nuclear waste management. The successful combination of copper ferrocyanide (CuFC) with natural mineral sorbents is important in the treatment of liquid radioactive waste (LRW) with high Cs ion mineralisation, but the presence of high concentrations of complexing organic substances in LRW contributes to a reduction in the sorption capacity of CuFC sorbents.[4]

During the accident at the Fukushima-1 nuclear power plant, a huge amount of 134Cs and 137Cs was removed by combining a mixture of ferrocyanides with clay sorbents, which significantly improved the environmental situation around the nuclear power plant.[5] The authors noted insignificant sorption of CuFC thorium oxide from aqueous solutions and showed that increased temperature and excess alkali content lead to degradation and rapid ageing of such ion-exchange materials.[6]

In,[7] a positive effect was noted from adding copper nitrate to a potassium ferricyanide solution at 80 °C, which contributed to the stabilisation of CuFC in acidic and saline solutions. The change in Kd values revealed a high affinity of CuFC for heavy monovalent cations as a function of nitric acid and ammonium nitrate concentration. This made it possible to determine the probable exchange mechanism and develop elution conditions for the binary separation of 137Cs from a number of other ions.

Classically, nanoscale CuFC is obtained by in situ synthesis using inexpensive chemical co-precipitation from solutions ($CuSO_4\cdot 5H_2O$) and $K_4[Fe(CN)_6]\cdot 3H_2O$. According to [8], $Cu_{13}[Fe(CN)_6]_{14}\cdot(2K)\cdot 10H_2O$

has a cubic structure (space group F-43) in the range from 10 to 30 nm and a surface area of 462.42 m^2/g, determined by the Brunauer-Emmett-Teller (BET) algorithm. The adsorption of Cs$^+$ and Sr^{2+} ions directly depends on pH. The maximum adsorption capacity (qmax) for CuFC was recorded at pH 6, which corresponds to the Langmuir model. According to this model, in nuclear reactor water, the maximum adsorption capacity qmax is 190.52 mg/g and 72.43 mg/g for Cs$^+$ and Sr^{2+}, respectively.[8]

The kinetics of CuFC adsorption is described using pseudo-first-order and pseudo-second-order intra-particle diffusion models at temperatures of 288, 298, and 308 K, respectively. The authors found that the pseudo-second-order kinetic model is characterised by a correlation coefficient (R^2) of 1.000 with an adsorption rate constant that increases with temperature and best describes the Cs+ adsorption process on CuFC. This result indicates that chemisorption occurs during the adsorption process. The adsorption equilibrium data are in good agreement with the Langmuir, Freundlich and Dubinin-Radushkevich (D-R) isothermal models. The average adsorption energy (E) between 11 and 13 kJ/mol at different temperatures indicates that ion exchange is the main mechanism during the adsorption process. The values of the standard Gibbs free energy change (ΔG_2o) and standard enthalpy change (ΔH_2O) indicate that adsorption is spontaneous with an endothermic process. At the same time, the distribution coefficient (Kd) was more than 2.94×10^6 ml/g when the pH of the solution was between 2.6 and 10.9 and the initial concentration of Cs$^+$ was 100 μg/l.[8]

Works[9, 10] show that the presence of K+ and Na+ ions in solutions with concentrations of 20 and 1000 mg/l does not affect the adsorption of Cs$^+$ on CuFC and demonstrate that CuHF has a high affinity for alkali cations in the following order: Cs$^+$ > K$^+$ > Na$^+$ > Ca^{2+} > Sr^{2+}. According to,[11] the IR spectra of CuFC contained characteristic absorption peaks, such as –C≡N, Fe(II)–C≡N and C≡N–Cu(II). The XRD spectrum corresponded to the standard CuFC spectrum, indicating the successful synthesis of CuFC in situ in the classical version.

To increase the selectivity and efficiency of radioactive ion sorption, inorganic and organic bases were incorporated into the CuFC structure by in situ synthesis. We described the simple synthesis of magnetic hydrogel beads with embedded copper ferrocyanide stabilised in ammonium hydroxide solution in.[12] The resulting CuFC-MHBs demonstrated effective removal of Cs$^+$ with a high Kd value of 66780 ml/g and excellent structural stability without CuFC release for 1 month and were effectively removed from water using an external magnet. In addition, CuFC-MHB selectively adsorbed Cs$^+$ with high Kd values in the presence of various competing ions, such as in simulated groundwater (24,500 ml/g) and seawater (8,290 ml/g) and retained their ability to absorb Cs$^+$ over a wide pH range from 3 to 11. Similar sorbents were obtained on porous aluminosilicate[13-17] and clay and zeolite substrates.[18-20] They have proven successful in removing residual concentrations of caesium ions from low-level radioactive water.

There is data on the production of composite adsorbents from potassium-copper ferrocyanide (CMC–KCuFC) particles based on sodium carboxymethylcellulose (CMC) biopolymer cross-linked with Cu^{2+}.[21] The authors investigated the factors affecting cesium adsorption, namely: pH (5-10) of the solution, contact time, initial concentration, temperature, and competing ions. The cesium adsorption process was described using the Freundlich isothermal model. The adsorption equilibrium time was 600 minutes and corresponded to the pseudo-second-order kinetic model.[8] The biological safety and biodegradability of the CMC–KCuFC sorbent components were also noted.

There are data on in situ synthesis of sorbents in the (SiO_2-CuFe-CN) system.[22] The authors showed that the experimental data obtained correspond to the Langmuir and Freundlich isotherms. The developed sorbent is a promising material for the remediation of low-level radioactive waste solutions containing 134Cs, 60Co, 99Mo and 90Sr with a relatively low concentration of nitric acid. Nanoparticles (SiO_2-CuFe-CN) provide high selectivity of sorption of the radioisotope 99Mo compared to other radioisotopes, starting from 0.01 M HNO_3. Further developments led to the creation of a composite magnetic silica material with functionalised copper ferrocyanide (mag@silica-CuFC), which can be easily separated from aqueous solutions using a magnetic field. The synthesized mag@silica-CuFC composite has a high sorption capacity for Cs$^+$ due to its strong affinity for Cs$^+$ and the large surface area of the carriers. Cs$^+$ sorption on the mag@silica-CuFC composite quickly reached sorption equilibrium after 2 hours of contact. The effect of the presence of salts with a high concentration of up to 3.5 wt. % on the efficiency of Cs sorption on composites was also investigated. It was found that the maximum sorption capacity is maintained in the presence of up to 3.5 wt.% NaCl in the solution.[23]

The work[24] notes the prospect of electrochemical synthesis of copper-zinc ferrocyanide (CuZnFC) for effective removal of Cs$^+$ from modelled liquid radioactive waste (LRW). Single-factor experiments established the optimal reaction conditions: initial pH 8.0, molar ratio of $[Fe(CN)_6]_{4-}$ to Cs$^+$ 10:1, reaction temperature 25 °C, current density 6 mA cm^{-2} and stirring speed 400 rpm^{-1}, which allowed a Cs$^+$ removal rate of 99.978% to be achieved. Analysis of the precipitate showed that its composition corresponds to $CuZn[Fe(CN)_6]_6 \cdot 18H_2O$, which had a cubic crystal structure and rapidly absorbed Cs+ from the simulated liquid radioactive waste.

There are sufficient literary sources that discuss the synthesis of composite materials in which CuFC is encapsulated in graphene, bentonite, etc.

The chemical bonding of CuFC to the surface of a hollow fibre PVDF membrane (PVDF-CuFC) is quite interesting for the effective removal of aqueous caesium (Cs$^+$). For the chemical immobilisation of CuFC on the surface of a hollow fibre PVDF membrane, COOH carboxyl groups were introduced for peptide binding of CuFC to amino groups. The PVDF-CuFC-based sorbent showed higher Cs$^+$ adsorption kinetics and adsorption capacity than PVDF-COOH. In addition, with an increase in the initial pH, the amount of Cs adsorbed by PVDF-CuFC also increased. However, the amount of Cs$^+$ adsorbed at pH 10 was slightly lower.[25] The use of PVDF-CuFC as a filter adsorbent for purifying Cs-contaminated water sources is relevant for continuous filtration.

There is data on the development of combined sorbents based on cotton fibre and ferrocyanides of transition metals.[26] This work investigates the effect of ferrocyanide concentration and process temperature on the content of inorganic components in the final product. It has been shown that the efficiency of caesium removal from model solutions decreases in the following order: Cu^{2+} - Fe^{2+} - Zn^{2+} - Ni^{2+} - Co^{2+}.

Researchers[27, 28] created a composite of copper hexacyanoferrate on polyacrylonitrile (CHCF–PAN) and used it as an ion exchanger to separate caesium from an aqueous solution. This sorbent was synthesized with a porous crystalline structure stable up to 200 °C. Kd for Cs$^+$ ions on CHCF–PAN increases with increasing pH, contact time, and solution temperature. The adsorption of Cs ions on CHCF–

PAN is an endothermic, spontaneous ion exchange reaction, and the sorption of Cs ions on CHCF–PAN corresponds to the Freundlich model with an efficiency of up to 63.78%.

There are many other examples of organic composites containing CuFC, but we are interested in the possibility of obtaining it by in situ synthesis in solid-phase reactions (powder method, grinding method) by direct reaction of metal salts with potassium hexacyanoferrate. In,[29] the possibility of such synthesis was considered and the increased selectivity of such a sorbent for the removal of Cs(I), Rb(I) and Tl(I) was noted. To remove Cs ions in acidic solutions of radioactive waste, potassium cobalt hexacyanoferrate (KCoFC) was synthesized by a similar method. The synthesized KCoFC was stable in a nitric acid solution with pH = 1 and showed high selectivity for Cs ions compared to Sr and Na ions. Kd Cs for a solution with 0.01N Cs^+ ions at pH = 2 was approximately $3.6x10^3$ ml/g in the presence of the same concentrations of Sr and Na ions, respectively. The ion exchange capacity of KCoFC for Cs ions, obtained using the Dubinin-Polanyi equation, was 1.72 meq/g.[30]

The above data show that obtaining CuFC by in situ synthesis in solutions is most effective for removing caesium ions from highly concentrated radioactive water, while direct (solid-phase) reaction is most effective for removing residual concentrations of caesium ions from low-level radioactive water. Therefore, this work considered the possibility of such synthesis for this purpose.

Materials and research methods

For the solid-phase synthesis of copper ferrocyanide in situ using the powder method, chemically pure salts of copper sulphate ($CuSO_4 \cdot 5H_2O$) and potassium hexacyanoferrate ($K_4[Fe(CN)_6] \cdot 3H_2O$) were used. The formation of copper ferrocyanide occurs according to the following reaction:

$$2CuSO_4 \cdot 5H_2O + K_4[Fe(CN)_6] \cdot 3H_2O = Cu_2[Fe(CN)_6] \cdot 7H_2O + 2K_2SO_4 + 6H_2O$$

To carry out the synthesis process, 50 g of the starting components were weighed and manually ground in a porcelain mortar for 10, 20, 30, 40, 50 and 60 minutes. Potassium ferrocyanide was taken in excess of 18.4% of the stoichiometry. The expected yield of copper ferrocyanide (CuFC) in the sorbent was approximately 46%. During grinding, we observed a colour change from light brown to dark brown-purple (Figure 1).

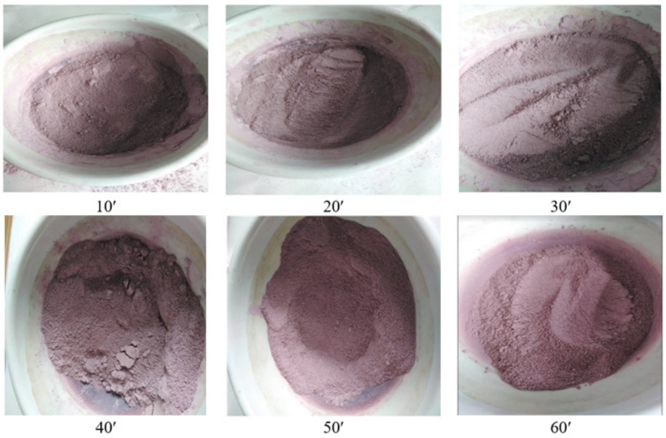

Figure 1 Appearance of copper ferrocyanide powders after grinding the starting reagents for min.

To confirm the formation of CuFC, IR spectra we taken on a Specord 125 spectrograph (Germany) of both the starting components and their mixture in the range of 400-4000 cm^{-1}.

To determine the sorption capacity of CuFC synthesized in situ by solid-phase reactions, three sorbent samples were prepared in portions of 1 g. The first sample was CuFC after 30 minutes of grinding; the second sample was 90% CuFC+10% PC-500; the third sample was dry CuFC powder obtained from solutions of the starting reagents. Weakly active radioactive water obtained by the method described in [13,15-17] was used as a source of radioactive radiation.

Samples of sorbents weighing 1 g were poured into 100 ml of radioactive water, stirred in a magnetic mixer for 60 minutes and left for 3 days (Figure 2). The activity of the water and sediment was determined after 3 days of settling using FoodLight and Atoll-3M gamma spectrometers.[13]

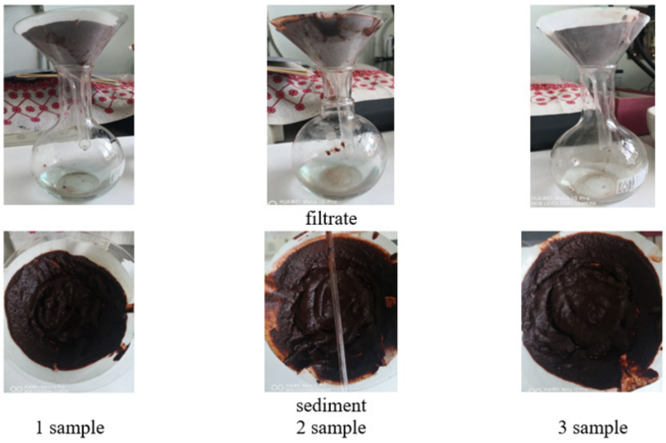

Figure 2 Results of radioactive water purification using sorbent samples 1-3.

Using a Bresser LCD Micro optical digital microscope, 8 MP, magnification x4, the microstructure of sorbent samples was analysed before and after 3 days of sorption, obtained by two methods – grinding and chemical precipitation (Fig 2).

Results

The results of IR spectrometry confirming the synthesis of copper ferrocyanide are shown in Figure 3, and the data on the measurement of water and sediment activities using copper ferrocyanide are shown in Figure 4. The following wave numbers, cm^{-1}, are characteristic for copper sulphate: 520, 577, 610, 670, 965, 1005, 1165, 1205, 1650. For stretching vibrations of OH^- groups – 3430 and 3750 cm^{-1}. For potassium ferrocyanide the following wave numbers, cm^{-1}; 586, 1623, 1645, and for OH^- groups – 3470 and 3530 (Figure 3).

After 10 min. of grinding the starting components, an absorption band characteristic of potassium ferrocyanide – 587 cm^{-1}, and absorption bands of copper sulphate – 965 and 1200 cm^{-1} and OH^- groups – 3420 cm^{-1} we observed in the mixture. The wave numbers for the reaction products – potassium sulphate and CuFC, cm^{-1} are fixed: 617, 657, 998, 1120, 1145, 1200 and 1620 cm^{-1}. After 20 minutes of grinding the initial components, the phase composition of the mixture is similar to the previous one. After 30 min. of grinding of the initial components, an increase in the absorption bands for the reaction products cm-1 is recorded in the mixture: 619, 655, 985, 1000, 1100, 1145, 1195 and 1630 cm-1, which confirms the formation of CuFC in larger quantities compared to previous time exposures. Increasing the grinding time from 40 to 60 min contributes to the formation of up to 46% CuFC (619, 657, 963, 985, 995, 1118, 1143, 1460, 1520 and 1620 cm^{-1}). The spectroscopy results are in good agreement with the data of[9] with the fixation of the groups –C≡N, Fe(II)–C≡N and C≡N–Cu(II).

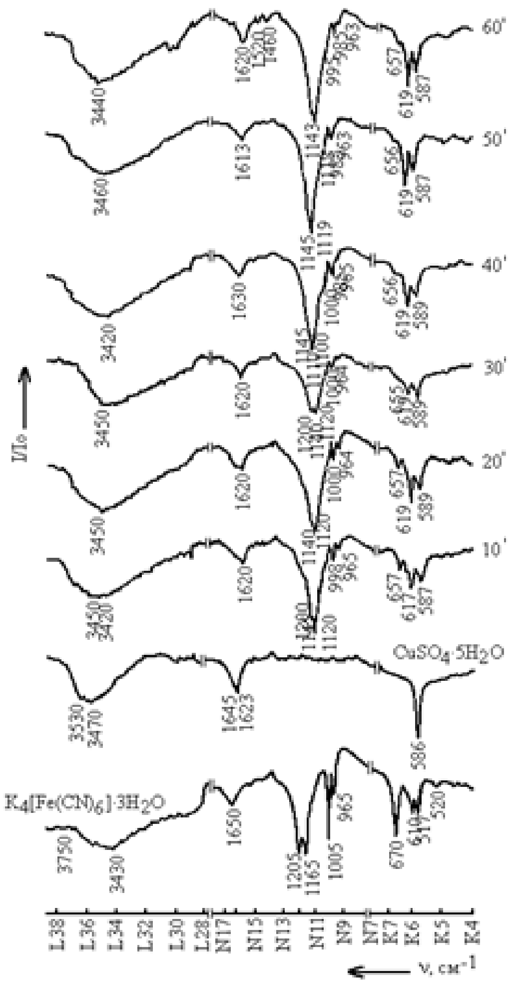

Figure 3 IR spectra of the starting reagents – copper sulphate and potassium ferrocyanide and their mixture after grinding, respectively, after 10, 20, 30, 40, 50 and 60 min.

The calculated results of determining the sorption properties of synthesized sorbent samples for selective removal of caesium ions from water with low radioactivity after 3 days of exposure are presented in Table 1. Particular attention was paid to the selective accumulation of caesium ions in the sediment, understanding that it may contain residues of other types of radionuclides (Figure 4) (Table 1).

Table 1 Sorption activity of sorbents

No	Sorbent	Activity, Bq	α, %
1 sample			
1	Mixture	0.826	100
2	Filtrate (water)	0.103	12.5
3	Sludge	0.672	81.4
2 sample			
4	Mixture	1.047	100
5	Filtrate (water)	0.245	23.4
6	Sludge	0,736	70.3
3 sample			
7	Mixture	0.661	100
8	Filtrate (water)	0.143	21.6
9	Sludge	0.465	70.3

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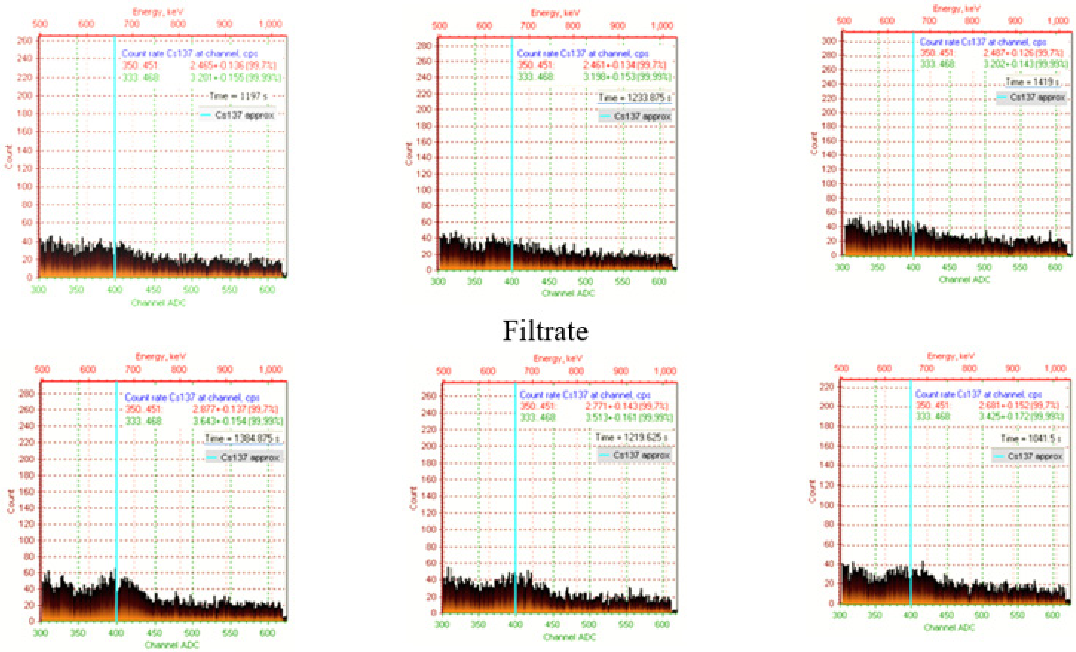


Filtrate

Sediment

1 sample

2 sample

3 sample

Figure 4 γ-spectrogram of water and sludge activity after 3 days of sorption.

As can be seen from the data in Table 1, sample 1 sediment has the highest accumulation capacity, with a cesium ion removal rate from radioactive water of 81.4%, while for samples 2 and 3 this value is 70.3%.

To refine the data on the accumulation of caesium ions in the sediments of three types of sorbents, we used the Atoll-3M gamma spectrometer. The measurement results are shown in Figure 5, and their interpretation is given in Table 2.

Table 2 Storage capacity of cesium ions in sludge samples of sorbents

Sample	Channel	Energy, keV	HWHH*, кeV	Area	Unrecognisable area	Nuclides	Activity, Bq
1	215.963	675.599	48.987	12434	296	Cs-137	146
	475.999	1539.59	92.029	3939	187	K-40	368
2	216.004	675.732	51.218	10621	339	Cs-137	125
	474.537	1534.58	90.23	4009	186	K-40	375
3	215.542	6,74,244	51.016	9615	313	Cs-137	113
	474.814	1535.52	89.965	4176	186	K-40	390

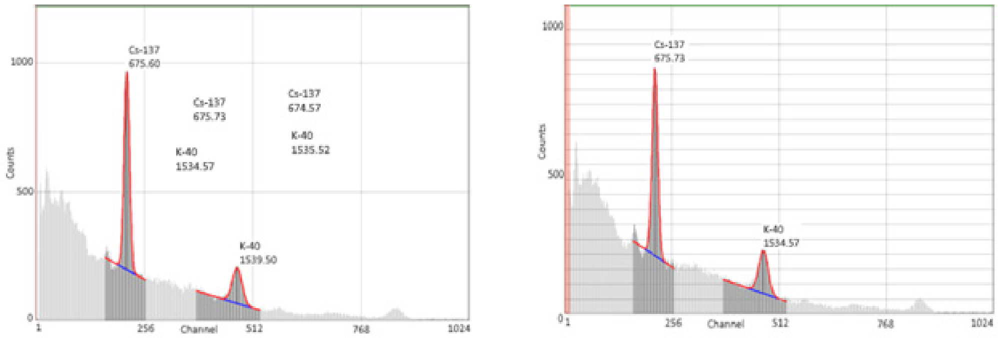
*Peak half-width and half-height.

As can be seen from the data in Figure 1 and Table 2, the highest capacity for cesium ion accumulation (up to 146 Bq) is characteristic of sorbent sample 1, which was synthesized in situ by solid-phase reactions. The accumulation capacity of other sorbent samples is significantly lower, 125 and 113 Bq, respectively (Figure 5) (Table 2).

To understand the manifestation of the sorption capacity of cesium ions in the developed sorbent samples of various types of synthesis, Figure 6 shows optical-digital photographs of the dried sediment before and after 3 days of sorption (Figure 6).

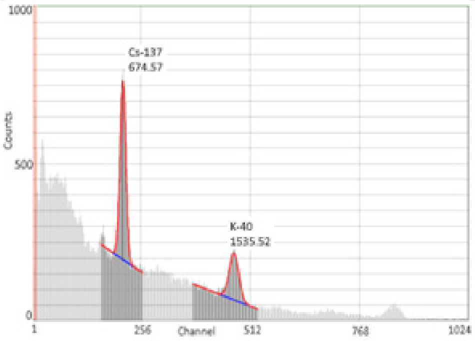
In Figure 6 shows that before sorption in the first sample of sorbent sediment, insignificant residues of copper sulphate (blue crystals) and translucent crystals characteristic of potassium sulphate are observed on the surface of CuFC grains. After 3 days of sorption, a modification of the surface of CuFC crystals with cesium inclusions is observed, which is reflected in spherical formations of a bright beetroot colour, i.e., ion exchange occurs with the probable formation of a compound of the type $Cs_2[Fe(CN)_6]\cdot 7H_2O$.

In the sediment of the second sorbent sample (Figure 6), in addition to copper sulphate crystals (blue in colour) on CuFC copper grains (brown-chocolate in colour), Portland cement hydration products are fixed in the form of hydrated grains and spherical clusters of highly basic calcium hydrosilicates. After 3 days of sorption, a modification of the surface of CuFC crystals with cesium inclusions is observed, which is reflected in spherical inclusions of a bright beetroot colour, i.e., ion exchange occurs with the probable formation of compounds of the type $Cs_2[Fe(CN)_6]\cdot 7H_2O$ and $Cs_2O\cdot 2CaO\cdot 2SiO_2\cdot 4H_2O$ compounds.



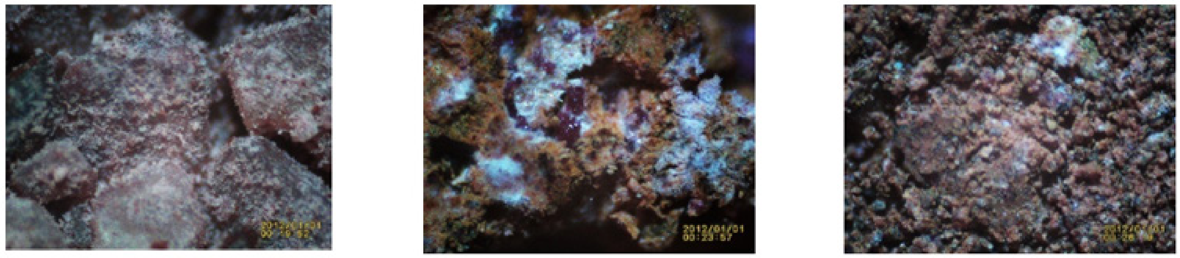
1 sample

2 sample

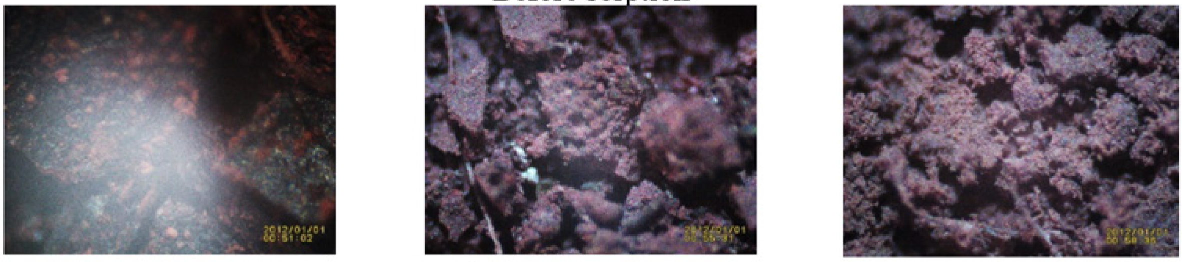


3 sample

Figure 5 γ-spectrograms of sludge of sorbent samples after 3 days of exposure.



Before sorption



After 3 days of sorption

1 sample

1 sample

1 sample

Figure 6 Macrostructure of sediment particles from sorbent samples before and after sorption.

In the sediment of sample 3 (Figure 6), copper ferrocyanide crystals contain copper sulphate residues that are not fixed after 3 days of sorption. The structure of the crystals is porous and discontinuous, which confirms the occurrence of ion exchange with the formation of $Cs_2[Fe(CN)_6]\cdot 7H_2O$.

Conclusions

The possibility of in situ solid-phase synthesis of CuFC by the powder method for the removal of caesium ions from low-level radioactive water has been established. It has been established that sorbent sample 1 has a removal rate of 81.4% with a sediment activity of 146 Bq. Sorbents samples 2 and 3 have a cesium ion removal rate of 70.3% with a sediment activity of 125 and 113 Bq. The addition of 10% Portland cement to the composition of sorbent sample 2 affects the rate of precipitation and chemical binding of cesium ions into compounds of the type $Cs_2O{\cdot}2CaO{\cdot}2SiO_2{\cdot}4H_2O$. The storage capacity of sorbent 1 (after 3 days of exposure) is manifested in the sorption of $137Cs^{1+}$ ions on the surface of CuFC particles due to hydroxyl groups with spherical formations of bright beet-coloured compounds, such as $Cs_2[Fe(CN)_6]{\cdot}7H_2O$.

In terms of the accumulation capacity of caesium ions, the sorbent samples can be ranked in the following order: sample 1 sample 2 → sample 3. The prospects for the application of the developed sorbent (sample 1) are evident in its simplicity of synthesis, low cost due to the use of inexpensive commercially available reagents, adsorption rate, high adsorption capacity and high selectivity to caesium ions.

Use of artificial intelligence

The author confirms that they did not use artificial intelligence technologies when creating the current work.

Acknowledgement

None.

Conflicts of interest

None.

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