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Research Article

INVESTIGATE THE ADSORPTION OF CESIUM ION (Cs^+) AND STRONTIUM ION (Sr^{2+}) ON $Zn_2[Fe(CN)_6]$ NANOPARTICLES

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ABSTRACT

Low-cost, nanoscale zinc hexacyanoferrate (ZnHF), an effective adsorbent for cesium (Cs^+) and strontium (Sr^{2+}) removal, was prepared using the chemical co-precipitation method. The Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS) spectra, and high-resolution transmission electron microscopy (HRTEM) images were performed to determine the morphologies of ZnHF. The $Zn_{15}[Fe(CN)_6]_{12}(2K).10H_2O$ was the trigonal structure (space group p-3c1) in the range of 50-200 nm, the BET surface area 43.08 m^2/g. The Cs^+ and Sr^{2+} removal were dependent on pH; this material's maximum value of adsorption capacity (qmax) is achieved at a pH of 6. According to the Langmuir model q_{max} = 190.52 mg/g and 72.43 mg/g for Cs^+ and Sr^{2+} respectively. The Langmuir model was conformable to describe the adsorption process of both Cs^+ and Sr^{2+} ions by ZnHF. The low-cost easily synthesized nanoscale zinc hexacyanoferrate (ZnHF) material. This material becomes an attractive and promising adsorbent in treating Cs^+ and Sr^{2+} ions of nuclear water.

Keywords: adsorption; cesium, nanoparticle; strontium; zinc hexacyanoferrate

1. Introduction

The rapid development of the nuclear industry with different purposes has caused severe radioactive contamination due to fission products with a half-life of decades, especially the ^{137}Cs (30.2 years) and ^{90}Sr (28.9 years) isotopes (Ali et al., 2020). In 1986, the Chernobyl nuclear disaster caused widespread pollution of ^{90}Sr (Petryna, 1995). In 2011, a strong earthquake of magnitude nine had created massive tsunamis that devastated the northeastern coast of Japan, resulting in the loss of temperature control inside the

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reactor at the Fukushima Daiichi nuclear power plant. This disaster led to reactors' explosions and causing of large-scale radioactive ^{137}Cs released into the environment (Yasunari et al., 2011).

Because cesium has similar chemical behavior to potassium in living organisms, and strontium has the same atomic radius as calcium, it will replace calcium in bones. When the human body absorbs cesium and strontium, those substances are difficult to be eliminated and can cause many different types of cancer (Avery, 1996; Cabrera et al., 1999). Therefore, recognizing and removing cesium and strontium is always an urgent issue and needs more research contributions from scientists.

In order to reduce radioactive contamination, various techniques such as liquid-liquid extraction, co-precipitation, adsorption, etc., have been studied to remove cesium and strontium in aqueous media. Among the applied techniques, adsorption is considered an economical and easy to operate method. Cesium and strontium adsorbent materials have been used, such as Na-MX80 bentonite (Siroux, 2017), zeolite (Abdollahi, 2019), or nano chitosan (Goyal, 2020).

Zinc hexacyanoferrate (ZnHF) has been shown to have good adsorption capacity with cesium (Li et al., 2008; Narang et al., 2013). This study aims to introduce a cubic nanostructures adsorbent ZnHF prepared by the chemical co-precipitation method, which is easy to conduct and low-cost synthesis. The materials were characterized by XRD, FTIR, HR-TEM, EDS-TEM, and BET images. The adsorption of ZnHF materials to cesium and strontium was also studied through isothermal adsorption and the change of the pH value of the solution. The results show that zinc hexacyanoferrate is one of the promising and effective materials in removing cesium and strontium ions from the solution.

2. Materials and Methods

2.1. Materials

All the highly pure reagents in this study were prepared from analytical-grade chemicals. The double-distilled water was used in the preparation of solutions for all experiments. The stock solutions of Cs^+ 1000 mg/L, Sr^{2+} 1000 mg/L, $K_4[Fe(CN)_6].3H_2O$ and $ZnSO_4.7H_2O$ are from Merck, Germany.

Cesium (Cs^+) and strontium (Sr^{2+}) working solutions were freshly prepared by diluting CsCl and $SrCl_2.6H_2O$ with double distilled water. The HNO_3 (0.1-0.5 N) and NaOH (0.1-0.5 N) were used to adjust the cesium solution pH as necessary. Cesium and strontium in solutions before and after were analyzed by Atomic Absorption Spectrometer (AA-7000 Shimadzu, Japan). Origin software was used for data analysis and processing.

2.2. Synthesis of zinc hexacyanoferrate (ZnHF)

The materials were synthesized according to the following reaction equation:

$$2ZnSO_4 + K_4[Fe(CN)_6] \rightarrow Zn_2[Fe(CN)_6] + 2K_2SO_4 \quad (1)$$

First, 0.15 mol of $ZnSO_4.7H_2O$ was dissolved in 750 ml of double distilled water with vigorous stirring, and the reactor beaker was placed in an ultrasonic bath. The beaker was equipped with a dropping funnel containing 250 mL of ($K_4[Fe(CN)_6]$ 0.05 M). Second, the $K_4[Fe(CN)_6]$ solutions were slowly dropped into a reactor beaker container of $ZnSO_4.7H_2O$ solutions under continuous stirring, and the temperature was maintained at 45°C. The adsorbent synthesis process was carried out in an ultrasonic bath (Elma S300H). After 8 hours of reaction, the white precipitate was collected and rinsed with double distilled water five times, centrifuged (Universal 320-Germany) at 10000 rpm, and dried at 60°C for 24 h. The materials obtained after drying were finely ground to prepare for further experiments (Li et al. 2008).

2.3. Characterization of ZnHF adsorbent

The adsorbent was characterized via XRD (Scintag-XDS-2000, Cu K α (λ=1,54059), HR-TEM, EDS-TEM **(**JEM 2100, HSX: Jeol, Japan) and surface area (BET) (Micromeritics - TriStar II 3020 3.02, USA) and EDS (JEOL JSM-6510LV, Japan), FTIR spectroscopy (Thermo Scientific, Nicolet iS10, USA). All experimental measurements were performed at ambient air temperature and their corresponding instruments.

2.4. Effect of solution pH

For safety purposes, the Cs^+ and Sr^{2+} for research are cesium salt and strontium salt in stable isotopes.

The effects of pH on the cesium and strontium adsorption were examined in a series of batch sorption experiments that used the same initial Cs^+ 150 mg/L and Sr^{2+} 150 mg/L, pH was maintained at different values of 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, and 9.0. The initial pH of the adsorption experiments solution was adjusted to a defined value with 0.01 N HNO_3 or NaOH and maintained at a designated pH value. After 24 h of the reaction, all the samples were centrifuged by Universal 320-Germany at 10.000 rpm for five minutes and filtered through a 0.22-µm membrane filter. The filtrate was analyzed for the cesium and strontium by AAS.

The amount of absorbent used is 0.1 g for Cs^+ and 1.0 g for Sr^{2+}, respectively.

2.5. Effect of the initial Cs^+ concentration

0.1g of zinc hexacyanoferrates was weighed precisely and placed into a 250 mL Erlenmeyer flask, which contained 100 mL of the cesium solution. The concentration of the cesium solution was selected within the range of 75, 100, 120, 145, 170, 200, 230, and

250 mg/L. A similar procedure was used for determining the adsorption capacity of strontium by ZnHF, excepted 1g of zinc hexacyanoferrates. The concentration of the strontium solution was selected within the range of 40, 100, 150, 200, 250, 300, 400, 450, and 650 mg/L were used.

During the adsorption process, the pH of the solution was not adjusted and maintained at a value of 6.0. The flask was shaken by IKA HS 260 basic USA at 270 rpm for 24 h. All batch experiment processes were carried out at the ambient air temperature (25°C). The initial pH of the adsorption experiments solution was adjusted to a defined value with 0.01 N HNO_3 or NaOH and maintained at a designated pH value. After 24 h of the reaction, all the samples were centrifuged by Universal 320-Germany at 10.000 rpm for 5 minutes and filtered through a 0.22-µm membrane filter. The filtrate was analyzed for the cesium and strontium by AAS.

The cesium and strontium adsorption capacities by zinc hexacyanoferrates were calculated by the change of the cesium and strontium concentrations before and after the adsorption process. The adsorption capacities of the cesium and strontium by the absorbed are calculated from the following expression:

$$q_e = \frac{V(C_i - C_e)}{B} \tag{2}$$

Where q_e is the adsorption capacity of adsorbent (in mg/g of adsorbent); C_i and C_e are the cesium or strontium concentrations (mg/L) before and after the adsorption process, respectively; B is the mass (g) of adsorbent used, and V is the solution volume (L) used.

The Langmuir adsorption equation:

$$q_e = \frac{q_m b C_e}{1 + bC_e} \tag{3}$$

Where q_e is the amount of cesium or strontium ions adsorbed by the materials (mg/g); Q_m is the maximum adsorption capacity of cesium or strontium ions; Ce is the initial concentration at a point of adsorption (mg/L); b is the rate constant between adsorption and desorption.

The Freunlich adsorption equation:

$$q_e = KC_e^{1/n} \tag{4}$$

Where q_e is the amount of cesium or strontium ions adsorbed by the materials (mg/g); K, n are constant adsorption of reaching equilibrium.

3. Results and discussion

3.1. *Material characteristics*

The elemental composition of the complex substance was examined using TEM/HR-TEM images coupled with EDS analysis. The EDS spectrum of ZnHF is shown in Fig. 1. The atomic percentages of different elements of the complex substance are also described in Fig. 1 and Table 1.

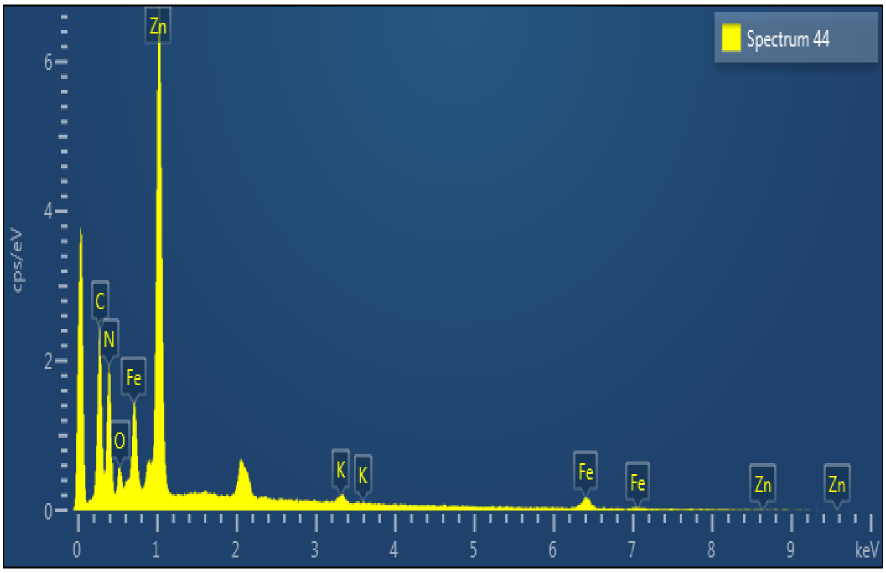

Fig. 1. *EDS spectrum of ZnHF*

Table 1. *The elemental composition of the complex substance*

Element	**Line Type**	**Weight (%)**	**Atomic (%)**
Zn	L series	25,81	7,39
Fe	L series	17,79	5,91
C	K series	22,88	35,47
N	K series	26,69	35,47
O	K series	4,24	4,93
K	K series	2,07	0,99
Total		99,47	90,15

ZnHF exhibits a large variety of compositions and structures. The main components of ZnHF materials were Zn, Fe, C, and N (Fig. 1 and Table 1). However, other elements such as potassium (K) and oxygen (O) are present in the material. Oxygen (O) indicates that ZnHF can combine with water molecules, potassium (K) can be a by-product of the synthesis of this ZnHF complex, and is reabsorbed by the adsorbent that has just been synthesized.

Based on these results, the molecular formula of ZnHF can be deduced to be $Zn_{15}[Fe(CN)_6]_{12}(2K).10H_2O$

FTIR spectroscopy was observed from IR Thermo Scientific, Nicolet IS10 instrument. FTIR spectrum provided valuable information to confirm the structure of the proposed synthesized materials

The FTIR spectrum of ZnHF is shown in Fig. 2. The FTIR spectrum of ZnHF has a conspicuous band centered at 2106.23 cm^{-1} assigned to the ν(CN) band. The FTIR spectrum of ZnHF consists of three vibrations within the octahedral unit of $[Fe(CN)_6]^{4-}$:ν(CN), δ(Fe-CN), and ν(Fe-C) (Rodríguez-Hernández et al., 2007). The relatively high frequency of the ν(CN) band in ferrocyanide suggests tetrahedral $[Fe(CN)_6]^{4-}$ coordination of the Zn atom. When the Zn atom is octahedrally coordinated with the N ends of CN groups, this vibration is observed as a band below 2095 cm^{-1} (Rodríguez-Hernández et al., 2007). Similarly, evidence obtained from δ(Fe-CN) and ν(Fe-C) showed bands centered at 611.79 and 503.89 cm^{-1}, respectively. A narrow δ(HOH) band centered at 1611.95 cm^{-1} was also observed. In the ν(OH) region, two FTIR bands centered at 3533.29 and 3583.48 cm^{-1} appeared, which were ascribed to the symmetric and asymmetric OH stretching of the coordinated water, respectively, indicating the existence of crystals binding to the water molecules of $Zn_{15}[Fe(CN)_6]_{12}(2K).10H_2O$.

The FTIR analysis results in this study are also consistent with the research results of the M. Avila group (Avila et al., 2008).

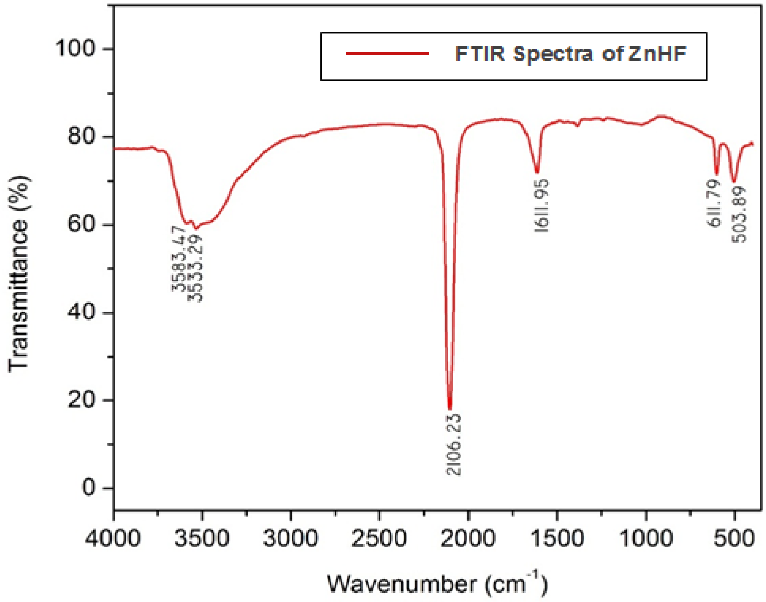

Fig. 2. *The FTIR spectrum of ZnHF*

The morphology of the complex substance is shown in Fig.3a and 3b. The bulk structures and the morphologies of ZnHF adsorbents were characterized on TEM and HR-TEM images. TEM micrographs of the complex substance showed a zeolitic structure and lactic form in the range of 50–200 nm. From Fig. 3b and the elemental composition of the complex substance in Table 1, sufficient evidence affirms that ZnHF had a trigonal structure with space group p-3c1.

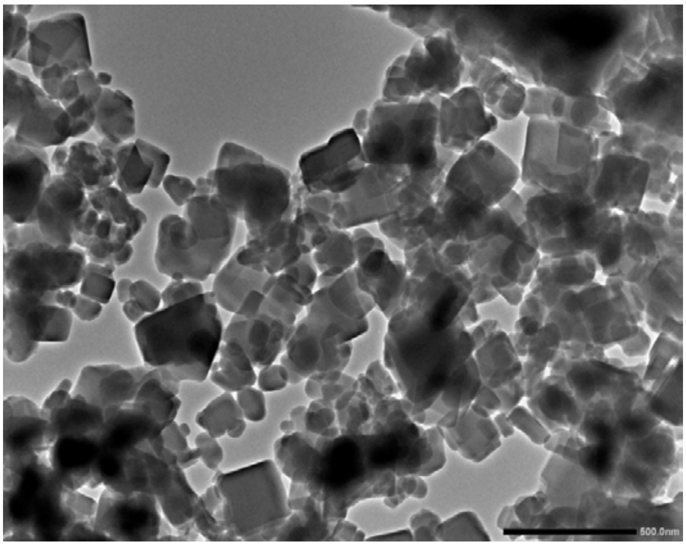

Fig. 3a. *TEM image of ZnHF*

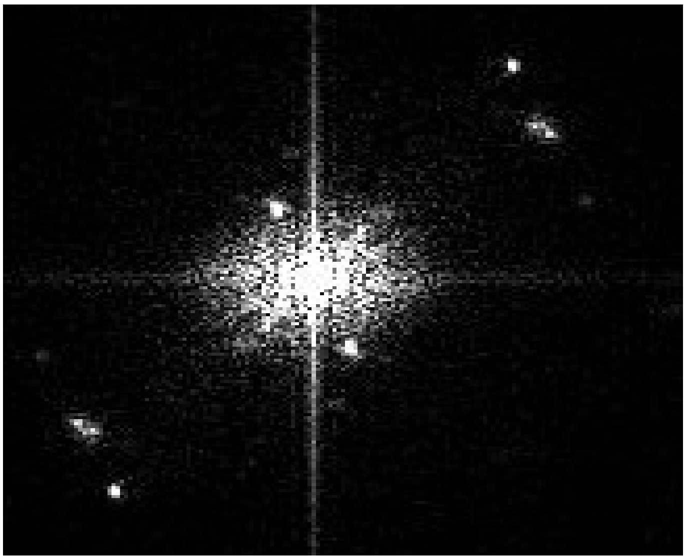

Fig. 3b. *HR-TEM image of one unit cell of ZnHF*

To identify the crystal structure of ZnHF, the XRD pattern of ZnHF was analyzed and is shown in Fig. 4. From the XRD pattern, all the peaks are well indexed to ZnHF with a few impurities, which can be ascribed to the trigonal structure with space group p-3c1. Through Rietveld refinement, the lattice parameters were found to be a = b = 12.613 Å and c = 32.971 Å (α = β = 90, and γ = 120), which are in line with the literature (Rodríguez-Hernández et al., 2007; Mark et al., 2003). It can be confirmed that the ZnHF component is in lactic form with trigonal configuration.

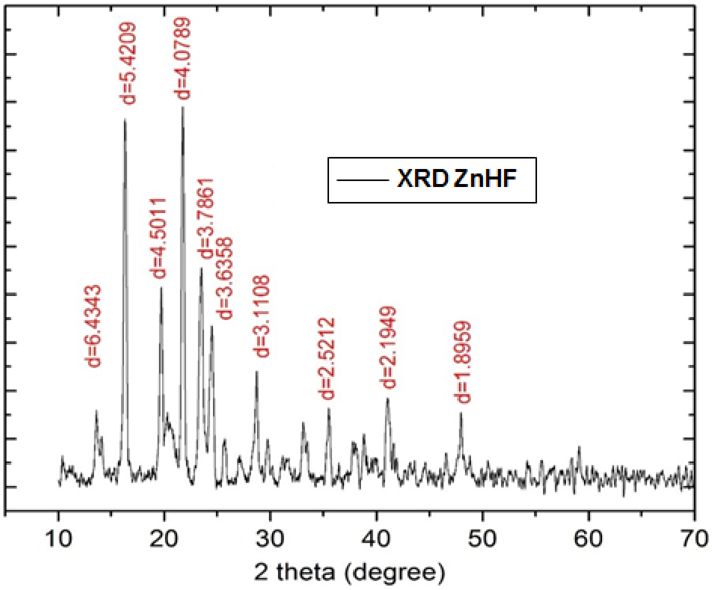

Fig. 4. *XRD pattern of ZnHF*

Based on the analysis results (Fig. 1, Fig. 2, Fig. 3a, Fig. 3b, and Table 1), using VESTA software, the crystal structure of one unit cell of ZnHF was drawn and is presented in Fig. 5. ZnHF had a zeolitic trigonal structure in the range of 50–200 nm and a BET of 43.08 m^2/g. The complex substance served as an adsorbent for further experiments.

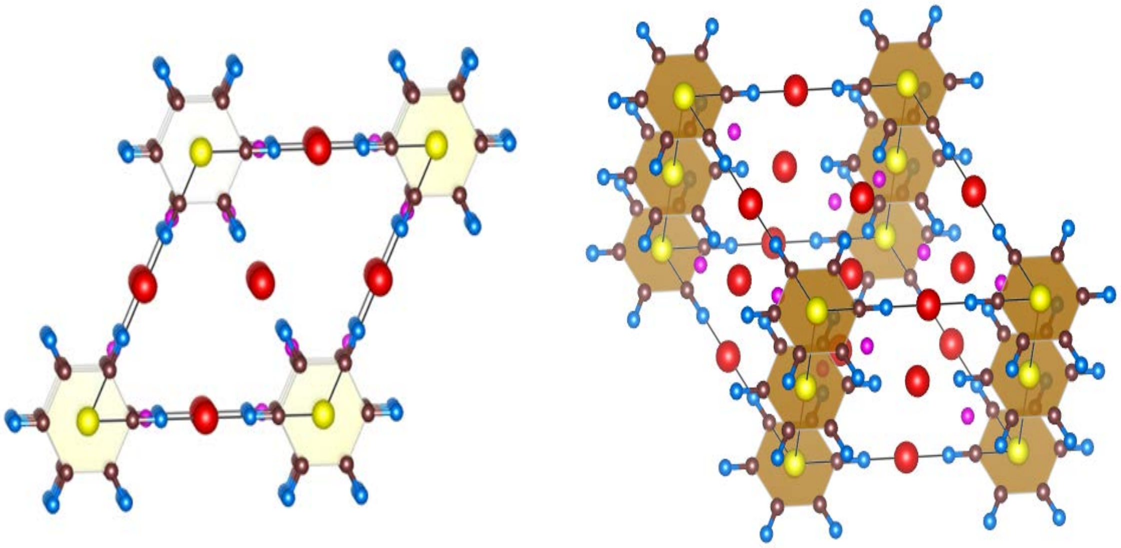

Fig. 5. *Crystal structure of one unit cell of ZnHF; blue (N), black (C), red (Zn), yellow (Fe), pink (H_2O and/or K^+)*

3.2. Effect of pH value on the adsorption capacity of ZnHF material to Cs^+ and Sr^{2+} ions

The results of Cs^+ and Sr^{2+} ions adsorption on ZnHF materials are shown in Fig. 6a and 6b.

The effect of pH value on the Cs^+ adsorption process by ZnHF materials was shown in Fig. 6a. The adsorption capacity of the material for Cs^+ ion is very low at pH values of 2 and 3. There is a noticeable change in the change of pH value. At a value of pH = 6, the maximum value of adsorption capacity is observed. When the pH value continuously increases, the adsorption capacity gradually decreases.

For Sr^{2+} ion, at the pH values of 2 and 3, a poorly adsorption process was observed. On the other hand, the adsorption capacity reached its maximum value when the pH value reached pH = 4. More specifically, the results show that in the pH range from 4 to 9, the absorbance of ZnHF materials for Sr^{2+} ions does not change significantly.

The results show that the adsorption of Cs^+ and Sr^{2+} ions by ZnHF is dependent on the pH value of the solution. During the following research process, we adjusted to pH = 6 for each type of experiment.

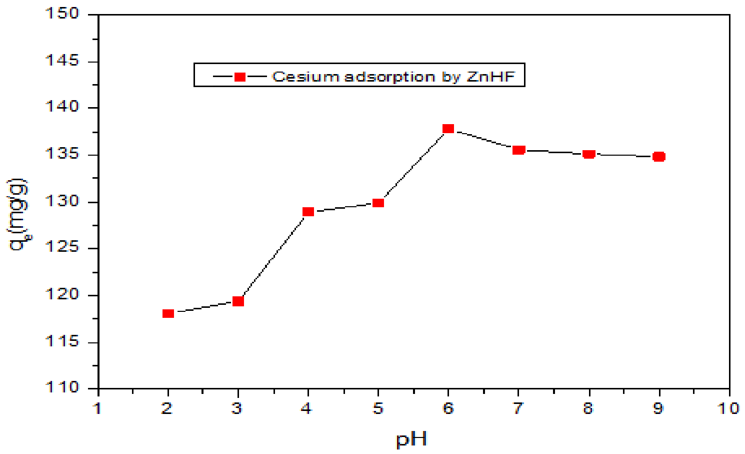

Fig. 6a. *Effect of pH value on Cs^+ ions adsorption by ZnHF*

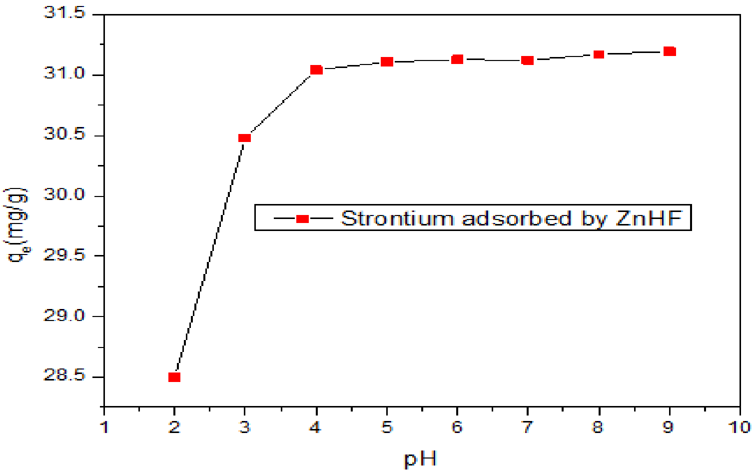

Fig. 6b. *Effect of pH value on Sr^{2+} ions adsorption by ZnHF*

3.3. Cs^+ and Sr^{2+} adsorption capacity by ZnHF materials

The maximum adsorption capacity of ZnHF materials was studied at a concentration range of 75 to 250 mg/L for cesium and 40 to 650 mg/L for strontium, respectively. The amount of absorbent used is 1 g/L for Cs^+ and 10 g/L for Sr^{2+}, respectively. All experiments were performed at pH = 6.0, the procedures were described in Section 2.5, and the results are shown in Fig. 7a and Fig. 7b.

Figure 7a shows that Cs^+ adsorption capacity increased sharply at a range of 75 to 250 mg/L for initial cesium concentration. When the concentration of Cs^+ was 200 mg/L, the amount of Cs^+ adsorption was 186.26 mg/g. The Cs^+ adsorption capacity reached its highest value of 187.44 mg/g when the initial concentration of Cs^+ was 230 mg/L. However, even if the initial concentration of Cs^+ continued to increase, the adsorption capacity decreased slightly and was almost unchanged. This result could be attributed to the equilibrium of the adsorption process.

Fig. 7b displays the experimental results when studying the adsorption process of Sr^{2+}, revealing that an inferior adsorption process was observed when ZnHF material was used to absorb Sr^{2+} ions in solution. Sr^{2+} ion adsorption process reached the equilibrium value if the initial concentration of Sr^{2+} was more than or equal to 450mg/L. The maximum absorbance value of ZnHF material for Sr^{2+} ion is 55.27 mg/g when the process reaches the equilibrium value.

Origin 8.5.1 software was used to analyze the results and parameters of the Cs^+ and Sr^{2+} ions isothermal adsorption onto ZnHF materials based on Langmuir and Freundlich models as shown in Table 2.

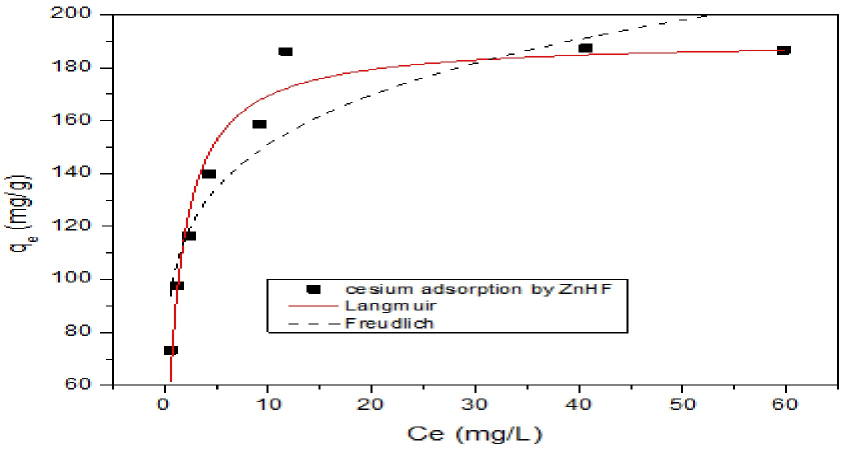

Fig. 7a. *Langmuir and Freundlich isotherm for the adsorption of Cs^+ on ZnHF*

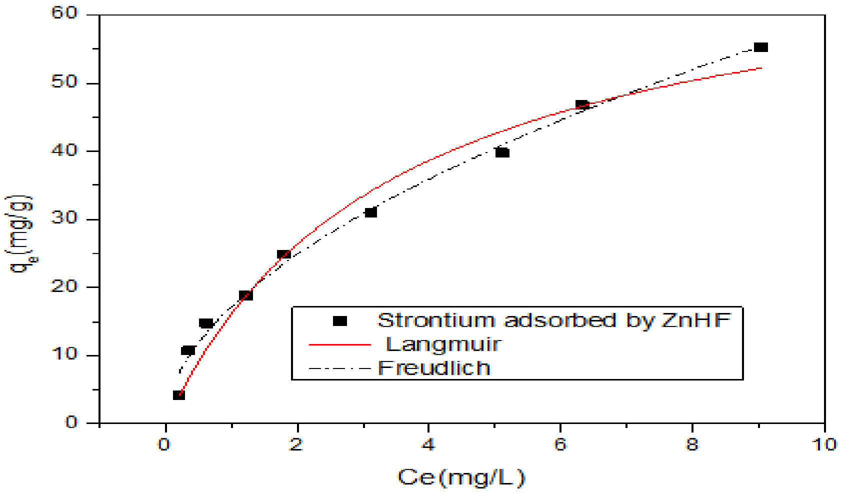

***Fig. 7b**. Langmuir and Freundlich isotherm for the adsorption of Sr^{2+} on ZnHF*

***Table 2.** Langmuir and Freundlich isotherm parameters for Cs^+ and Sr^{2+} adsorption on ZnHF adsorbent*

ZnHF materials			
Langmuir Model			
Absorbed Ions	q_m (mg/g)	b	R^2
Cs^+	190,5	0,80	0,95
Sr^{2+}	72,4	0,29	0,97
Freundlich Model			
Absorbed Ions	K_F (mg/g)	1/n	R^2
Cs^+	102,5	0,17	0,83
Sr^{2+}	17,2	0,53	0,99

The calculated maximum adsorption capacity (q_{max}) of the ZnHF material for both ion adsorption processes is larger than the experimental figure. The adsorption parameters for the Freundlich isotherm adsorption model have a value of $1/n < 1$, which is consistent with the theory for both cases. R^2 values in the range 0.83-0.99 for both cases are appropriate. The Cs^+ and Sr^{2+} ions adsorption process in water by ZnHF material follow the rules of normal adsorption. Both either Langmuir or Freundlich adsorption models can be used to describe the adsorption of Cs^+ and Sr^{2+} ions by ZnHF materials.

The comparison of ZnHF materials adsorption capacity in this study with other different materials in various research groups is shown in Table 3. The ZnHF materials synthesized in this study can remove two ions, Cs^+ and Sr^{2+}, and proving that it is a potential material to treat water contaminated with radioactive substances.

Table 3. *Compare the adsorption capacity of ZnHF with different adsorbent materials*

Absorbent	**pH**	**Cesium maximum adsorption capacity (mg.g^{-1})**	**Strontium maximum adsorption capacity (mg.g^{-1})**	**References**
ZnHF	6,0	190,5	72,4	This study
MIL-101-SO3H	6,0	36,5	-	Aguila et al., 2016
Zeolite	2-10	102,0	96,2	Vipin et al., 2016
Zeolite + MWCNT	2-10	113,6	107,5	Vipin et al., 2016
Zeolite A	2-8	207,5	303,0	El-Kamash et al., 2008
AC-PBNP	6,8	36,1	9,26	Ali et al., 2020
Nanozeolite composite	7,0	208,4	97,1	Faghihian et al., 2013
Microzeolite composite	7,0	160,0	78,9	Faghihian et al., 2013

4. Conclusion

Zinc hexacyanoferrate is an inexpensive, nano-sized material that can remove cesium and strontium from an aqueous solution. ZnHF in this study was synthesized by the co-precipitation method.

From the results of XRD, EDS-TEM, IR infrared, BET, and HR-TEM spectra, we conclude that $Zn_{15}[Fe(CN)_6]_{12}(2K).10H_2O$ material has been synthesized successfully. It has the trigonal crystal form (p-3c1), the size is in the range of 50-200 nm, and a BET specific surface area of 43.08 m^2/g. The material has been prepared at a low cost and is potentially used as an adsorbent.

The adsorption capacity of the material for both Cs^+ and Sr^{2+} ions reached their maximum value at pH = 6. The maximum adsorption capacity values of ZnHF were 187.44 mg/g for Cs^+ and 55.27 mg/g for Sr^{2+} respectively. The maximum adsorption capacity according to Langmuir model (q_{max}) of the ZnHF is larger than the observed experimental value.

The Cs^+ and Sr^{2+} ions adsorption process in water using ZnHF material follow the rules of normal adsorption. Two isothermal adsorption models Langmuir and Freundlich can be used to describe the adsorption of Cs^+ and Sr^{2+} ions by this material.

❖ ***Conflict of Interest:*** *Authors have no conflict of interest to declare.*

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NGHIÊN CỨU KHẢ NĂNG HẤP PHỤ Cs^+, Sr^{2+} BỞI VẬT LIỆU NANO KẼM HEXACYANOFERRATE

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TÓM TẮT

Vật liệu kích thước nano kẽm hexacyanoferrate (ZnHF) là loại vật liệu hấp phụ hiệu quả đối với hai ion Cs^+ *và* Sr^{2+}*. ZnHF được điều chế bằng phương pháp đồng kết tủa. Các phổ hồng ngoại Fourier (FTIR), nhiễu xạ tia X (XRD), quang phổ tia X kết hợp tán xạ năng lượng (EDS), kính hiển vi điện tử truyền qua có độ phân giải cao (HRTEM) được sử dụng để xác định hình thái của vật liệu ZnHF. Vật liệu* $Zn_{15}[Fe(CN)_6]_{12}(2K).10H_2O$*, tinh thể ở dạng trigonal (p-3c1), có kích thước 50-200 nm, diện tích bề mặt BET là 43,08* m^2/g*. Cả hai ion* Cs^+ *và* Sr^{2+} *bị loại khỏi dung dịch đều phụ thuộc vào pH, hấp dung cực đại của quá trình được tiến hành ở pH = 6. Dung lượng hấp phụ cực đại theo mô hình tính toán Langmuir* q_{max} *là 190,52 và 72,43 mg/g lần lượt đối với cesi và stronti. Quá trình hấp phụ cả hai ion* Cs^+ *và* Sr^{2+} *trong nước bởi vật liệu ZnHF tuân theo quy luật của quá trình hấp phụ đơn thuần. Hai mô hình hấp phụ Langmuir và Freundlich có thể dùng mô tả quá trình hấp phụ ion* Cs^+ *và* Sr^{2+} *bởi vật liệu này. Vật liệu kẽm hexacyanoferrate (ZnHF) kích thước nano, giá thành thấp, dễ tổng hợp; loại vật liệu này có thể trở thành chất hấp phụ hấp dẫn và đầy hứa hẹn trong việc xử lí ion* Cs^+ *và* Sr^{2+} *trong nước.*

Từ khóa: hấp phụ; Cesi; nano; stronti; $Zn_2[Fe(CN)_6]$