



Original Article

Gamma spectrometry technique application to the ^{60}Co sorption onto IRN-77 resin from radioactive wastewater: Equilibrium, Kinetic and Thermodynamic investigations

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ARTICLE INFO

Article history:

Received 16 July 2023

Revised 22 October 2023

Accepted 03 November 2023

Keywords:

Nuclear wastewater;
 ^{60}Co ;

kinetic study;

Adsorption;

Gamma spectrometry;
IRN-77.

ABSTRACT

The performance of synthetic ion exchange resin IRN-77 have been studied in this work in order to use it as an adsorbent to remove radioactive isotope ^{60}Co from nuclear wastewater by the sorption process, using the gamma spectrometry technique. The resin sample was identified using SEM and FTIR infrared spectrometry. The gamma radiation acquisition emitted from the fixed radioactive ^{60}Co onto IRN-77 solid samples was carried out using the gamma spectrometry chain, equipped with an HPGe semi-conductor detector with high-resolution. Various factors were considered for the sorption process study such as ^{60}Co concentration, contact time and temperature. The maximum adsorption capacity of the IRN-77 samples was determined by studying the adsorption isotherms; Kinetics models including thermodynamics were also studied and investigated. The experimental results showed that the adsorption reaction was adjustable to the pseudo-first-order and the Langmuir model was found to describe best the experimental results by obtaining a very important maximum adsorption quantity of $10.620 \mu\text{Ci}$ of ^{60}Co per 1 gram of IRN-77 adsorbent. A dimensionless separation factor R_L was used to judge the favorable adsorption. The adsorption capacity of ^{60}Co ions onto IRN-77 particles increased with the increasing of temperature. The values of the thermodynamic parameters have shown that the ^{60}Co ions adsorption process was endothermic and favored at high temperatures with a positive value of the enthalpy ΔH° of 23.54 kJ/mol . The free energy's values ΔG° are positive over the whole temperature range. The specific activities of the fixed ^{60}Co radionuclide allow evaluating the solid samples IRN-77 resin's sorption capacity.

1. Introduction

The γ spectrometry's development began with the nuclear sciences development and technologies in order to satisfy the needs for analysis, characterization and control of radioactive material. Gamma spectrometry is applied to identify and measure gamma-emitting radionuclides (e.g., ^{131}I , ^{60}Co , ^{134}Cs , and ^{137}Cs). Gamma spectrometry has many advantages, such as simultaneous analysis of multiple radionuclides, and characterized by the simplicity of sample analysis which does not require its prior radiochemical preparation, and is therefore a basic technique used for radionuclide monitoring [1]. It is essential to control the cooling waters in the primary circuits of nuclear reactors, in order to limit any expected contamination of these circuits as well as to protect

personnel intervening during maintenance actions by reducing radioactive dose flow rates. The primary circuits of nuclear reactors attempt to use high nuclear quality ion exchange resins to treat these moving waters [2]. However, despite the optimization of the materials and the chemical composition of the primary circuit water of the nuclear reactor, some contamination of this water may occur which mainly comes from corrosion of the metal walls (reactor tank, pipes, pumps, reactor pool, etc.). A fraction of the constituent elements of the steels solubilizes and charges the primary fluid with metal ions which will activate during their passage in the active zone (The core of the nuclear reactor) by absorbing part of the neutron radiation and form radioisotopes which are called

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Peer review under responsibility of University of El Oued.

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activation products. In the cooling fluids of the nuclear reactor, corrosion radionuclides with different half-lives exist or can appear. ^{60}Co is among the most dangerous radionuclides due to its mobility and transport in the environment as well as its very long half-life with its γ emissions. It has the capacity to induce harmful effects in the nervous and genetic system in humans, as it can be the main cause of the appearance of carcinogenic cells [3, 4]. The presence of this radionuclide leads to the formation of colloidal particles which circulate and are deposited on the walls of the reactor cooling circuit. (The combustible element plates, the grid...), subsequently affecting the quality of the cooling water circuit, its pipes and its ancillary organs (heat exchanger plates). Consequently, the deposition of these corrosive ^{60}Co particles around the nuclear reactor is at the origin of two constraints which harm the operation and maintenance of the reactor: likely to affect the distribution of the neutron flux and the activation of certain corrosion products during their passage through the active zone of the reactor, and together constitute liquid radioactive waste which is therefore imperative to be eliminated by effective adsorbent particles for safe reactor operation [2]. There are several processes for treating radioactive pollutants in aqueous solutions [5-8]. Among these processes we can cite adsorption which is considered a very popular separation method and is applied in particular for the treatment of low concentration effluents, this method is characterized by its feasibility and simplicity, its high yield and especially for its low cost and the possibility of using abundant and effective adsorbents [3,8-12]. Several recent research works have been carried out for the elimination of these corrosive radioactive entities using different types of adsorbents [2,3,5,13,14]. Ion exchange resins have attracted the attention of scientific researchers in recent years [15-17]. These resins are ion exchangers of an organic nature, made up of a network of insoluble macromolecules. These can be of natural or synthetic origin. The exchange capacity of a resin is reflected by a precise number of functional groups, this is the number of moles of theoretical monovalent ions that a resin can fix. The work of this article is devoted to the study of the behavior of the resin with regard to the adsorption of 60-cobalt by applying an unconventional technique which is gamma spectrometry, through the measurement of the specific activities of the ^{60}Co retained.

2. Materials and Methods

2.1. Characterization of the adsorbent solid material

The IRN-77 particles studied in this paper is a nuclear grade ion exchange resin and processed according to the highest purity standards to meet the strictest requirements

of the nuclear industry. It is a strongly acidic gel-type polystyrene cation exchanger with a uniform particle size supplied in at least 99% of its exchange sites in the form of hydrogen, a high exchange capacity of $\text{CE} > 1.9 \text{ eq/L}(\text{H}^+)$, approximately 8% DVB cation resin, a density of $\sim 800 \text{ g/L}$ and a high operating temperature of approximately $120\text{--}150^\circ \text{C}$. The particles of IRN-77 resin have a uniform size and are characterized by the absence of fine beads which result in a lower pressure drop which differs from conventional resins. The IRN-77 sample was characterized by the Scanning Electron microscope (PHILIPS XL30 FEG ESEM) to observe the morphology of sample. Infrared spectroscopy Analysis (FTIR) was carried out using a Perkin Elmer spectrometer (UATR Two model). The adsorption capacity of the solid resin IRN-77 were measured using the gamma spectrometry chain equipped with a HPGe semi-conductor detector with high-resolution (FWHM) of about 1.8 keV at 1332.5 keV γ -ray peak of ^{60}Co [18]. The spectra were analyzed using the Gamma vision software.

2.2. Procedure of the experiments study

The isotope ^{60}Co was obtained by irradiating a quantity of stable cobalt filament ^{59}Co in the nuclear research reactor (NuR) [2]. ^{60}Co is produced by nuclear radiative capture reaction according to: $^{59}_{27}\text{Co} + {}^1_0n \rightarrow {}^{60}_{27}\text{Co} + {}^0_0\gamma$. The irradiated thread was solubilized in nitric acid HNO_3 and diluted in the deionized water. The behavior of the IRN-77 resin with regard to the adsorption of ^{60}Co ions was conducted in static mode. with different adsorption operating parameters such as contact time, temperature as well as the initial ^{60}Co concentration. Quantities of adsorbent were brought into contact with 25 ml of ^{60}Co solution in a thermostatically controlled water bath, and subjected to magnetic stirring at a constant stirring speed of 200 rpm, for a contact time which could go up to 4 or 5 hours of stirring.

After an equilibrium time, the suspensions were centrifuged at 5500 rpm; the pellets of the samples were analyzed by the gamma spectrometry chain after its calibration in energy and efficiency. The adsorption capacity of cobalt ions quantified at equilibrium and measured in $\mu\text{Ci/g}$ is determined by:

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \quad (1)$$

Where C_0 ($\mu\text{Ci/L}$) and C_e ($\mu\text{Ci/L}$) are the initial and equilibrium concentrations of metal ions, respectively, V is the volume of the aqueous phase solution (L) and m is the amount of adsorbent (g).

3. Results and Discussion

3.1. Adsorbent Analysis

The SEM images of IRN-77 are presented on Fig.1 (a) and

Fig.1 (b). It can be seen that the crystals of the IRN-77 resin have spherical morphologies with an average size of between 300 μm and 690 μm , highlighting the existence of intragranular porosity, smooth, homogeneous, undamaged, regular and well-organized surfaces.

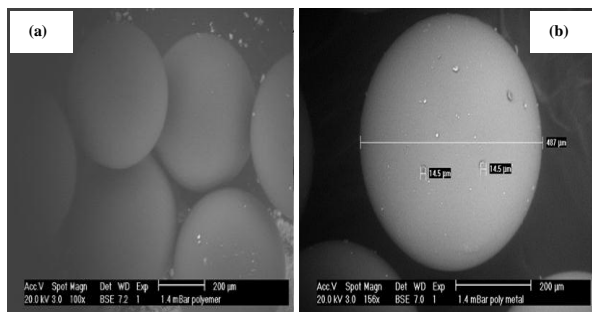


Fig 1. Scanning electronic micrographs of IRN-77 resin.

Fig.2. shows FTIR infrared spectra of IRN-77 resin. It is clear from this figure that there are several binding adsorption bands in the network of IRN-77. We can observe a valence vibration of the hydroxyl groups $-\text{OH}$ of constitutional water located about $(3100 \text{ and } 3500) \text{ cm}^{-1}$, but also a vibration of the connection $\text{C}-\text{H}$, around $(2927 \text{ and } 2924) \text{ cm}^{-1}$ and also a binding adsorption band of $\text{C}=\text{C}$ located about $(1634 \text{ and } 1645) \text{ cm}^{-1}$ as well as effusions in the band of $(500 - 1500) \text{ cm}^{-1}$ located at $(1476 \text{ cm}^{-1} \text{ and } 1377 \text{ cm}^{-1})$ [19].

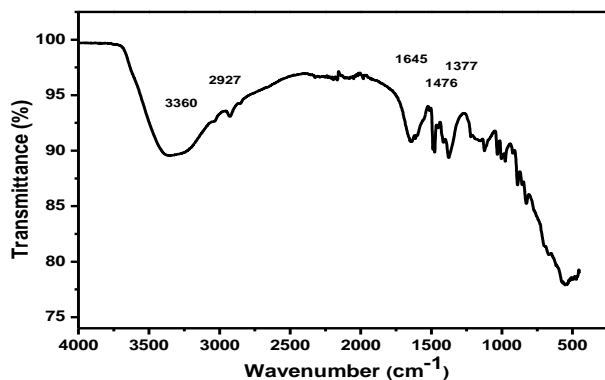


Fig 2. FTIR spectra of IRN-77 resin

3.2. Effect of temperature and contact time

The equilibrium time is one of the important parameters in this work and it was studied by varying the temperature of the reaction medium. The adsorption's equilibrium time of cobalt onto IRN-77 resin was determined for an aqueous solution concentration of 100 mg/L by varying the contact time from 5 to 90 minutes. The evolution of the amount of adsorption of ^{60}Co ions on the IRN-77 resin in terms of specific activity at different temperatures is shown in Fig.3.

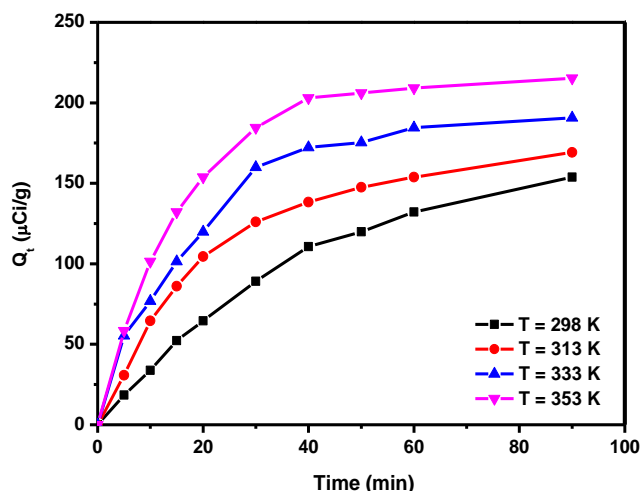


Fig 3. Effect of contact time on the adsorption capacity of Co^{2+} ions onto IRN-77 at different temperatures; ($\text{pH} = 5.5$, $C_0 = 100 \text{ mg/L}$ and $S/L = 5 \text{ g/L}$).

We note that the rate of the adsorption rate variation of Q_t as a function of time increases slowly in a non-linear way, to reach saturation at longer times. The results also show that the adsorption kinetics of cobalt onto the IRN-77 resin takes place in two phases: The adsorption of Co^{2+} ions increases moderately during the first thirty minutes over the whole temperature range studied (298, 313, 333, 353) K, beyond this, the variation is no longer significant and the curves then tend towards a plateau. Equilibrium is still not reached after 90 min; this means that the IRN-77 resin is not yet saturated, which means that its adsorption capacity is very important [13].

3.3. Equilibrium study

Experimental adsorption isotherms were carried out in order to establish a certain basis of useful information to better understand the adsorption mechanism of ^{60}Co onto the studied resin, such as the surface properties of the adsorbents as well as the affinity between the adsorbent and the adsorbate [20]. The graphical representation of the ^{60}Co adsorption isotherm onto IRN-77 is shown in Fig.4. Figure Fig.4 shows that the adsorbed quantity of Co^{2+} ions evolves rapidly for solutions at low concentrations; then decreases until equilibrium represented by a plateau of saturation of the adsorbent sites.

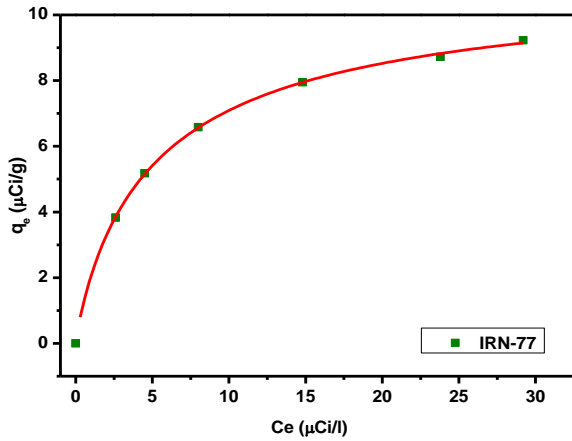


Fig 4. Equilibrium isotherm of the adsorption of Co^{2+} ions onto IRN-77; (T= 298K, pH = 5.5 and S/L = 5g/L).

The shape of the representation of the experimental points made it possible to confuse the isotherms obtained in type L; according to the classification of Giles and col (1960) [21] which shows that the ion adsorption process could occur in monolayers. The adsorbent-solute interaction is greater than that between solvent-solute, this phenomenon occurs when the forces of attraction between the adsorbed molecules are weak [22].

In order to quantify the maximum adsorption capacity of pollutants and to identify the surface properties of different adsorbents, various theoretical models have been unveiled [23,24]. Langmuir and Freundlich models were used in this study as fundamental adsorption isotherm; in order to propose the best model suitable for obtained data. concerning the adsorption of ^{60}Co ions onto the studied resin. The linear regression closest to 1 will be the chosen model that best describes the adsorption mechanism.

The Langmuir isotherm was initiated in order to quantify the adsorption capacities of a gas on solid materials, while taking into account at the same time the relative rates of adsorption and desorption of this gas [25]. While for the Freundlich isotherm is mostly applicable to the adsorption of adsorbate on the heterogeneous adsorbent surface [26]. The Langmuir and Freundlich models linear form can be represented by the equations (2) and (3) respectively [25]:

$$\frac{C_e}{q_e} = \frac{1}{K_L \cdot Q_0} + \frac{C_e}{Q_0} \tag{2}$$

Where q_e is the equilibrium adsorption capacity given in ($\mu Ci/g$), C_e is the equilibrium ion concentration given in ($\mu Ci/L$), Q_0 is the saturation capacity of the layer given in ($\mu Ci/g$), K_L is the equilibrium adsorption constant given in ($L/\mu Ci$). These parameters are obtained from the slope and the intercept at the origin of the plot C_e/q_e as a function of C_e (Fig.5.a).

$$\text{Log } q_e = \text{Log } K_f + \frac{1}{n} \text{Log } C_e \tag{3}$$

Where K_f is the Freundlich coefficient related to the adsorption capacity given in (L/g) and n is adsorption intensity. These parameters are obtained from the slope and the intercept at the origin of the plot $\text{Log } q_e$ as a function of $\text{Log } C_e$ (Fig.5.b).

The parameters of adsorption isotherm models are given in Table.1.

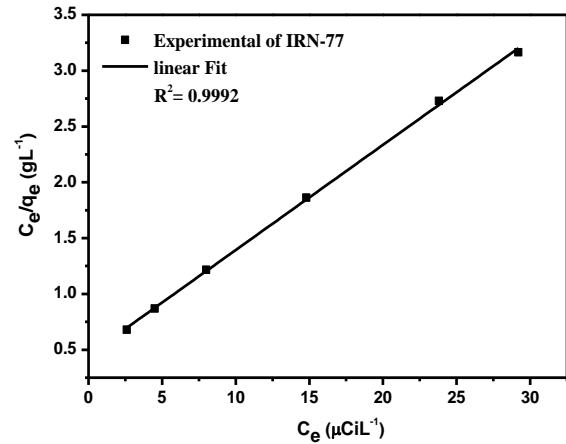


Fig 5.a. Linear representation of the Langmuir isotherm for the ^{60}Co ions adsorption onto IRN-77 (T=298 K $C_0=100\text{mg/L}$, pH= 5.5, S/L=5g/L and t=1h).

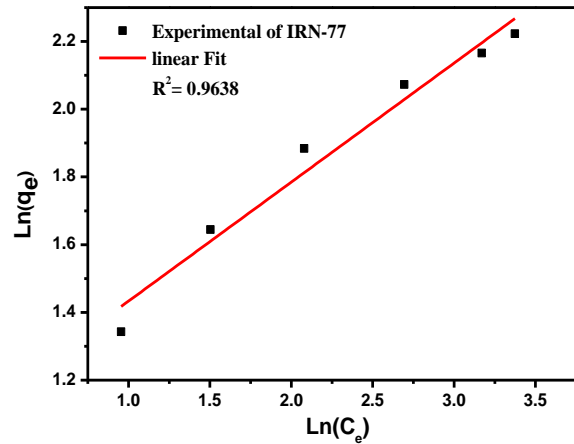


Fig 5.b. Linear representation of the Freundlich isotherm for the ^{60}Co ions adsorption onto IRN-77 (T=298 K, $C_0=100 \text{ mg/L}$, pH= 5.5, S/L=5g/L and t=1h).

Table 1: Parameters of ^{60}Co ion adsorption isotherms onto IRN-77 resin at 298K.

Model	Parameter	IRN-77
Langmuir	$Q_0(\mu Ci/g)$	10.620
	$K_L (L/\mu Ci)$	0.208
	R^2	0.9992
Freundlich	$1/n$	0.351
	$K_f(L/g)$	2.949
	R^2	0.9638

According to Fig.5.a and Fig.5.b; it should be observed that at 298 K, the linear representation of the Langmuir isotherms for the ^{60}Co ions adsorption onto the studied

resin presents very good linearity compared to that of the Langmuir model; as well as the results presented in table 1 show that the value of the correlation coefficient (R^2) obtained by the Langmuir model is very close to unity. Therefore, these results suggest that the Lagmuir model appears to be the appropriate model for adjusting the experimental results of the adsorption isotherms of ^{60}Co ions; and to assume that the process of adsorption of these ions occurs in monolayer mode with a finite number of adsorption sites, assuming that one molecule is adsorbed per site; no mobility of molecules and no interaction [27,28].

in the literature, the proportion ($1/n$) is defined as a heterogeneity factor [29,30]. The results in Table 1 reveal that the surface structure of our adsorbent is heterogeneous, generating the minimum percentage of interaction between the adsorbed atoms and this due to the value of $1/n$ which is less than unity [2]. These results also suggest a chemical adsorption process to be confirmed in other studies.

3.4. Kinetic study

The kinetic characteristics of the adsorption of ^{60}Co ions on the IRN-77 resin are dependent or controlled and diagnosed by several types of mechanisms such as chemical reactions, diffusion and in general mass transfer

phenomena.

The pseudo-first order and pseudo-second order linear forms are expressed by equations (Eq.4) and (Eq.5) respectively [2]:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{4}$$

$$\frac{t}{q_t} = \frac{1}{q_e} t + \frac{1}{k_2 q_e^2} \tag{5}$$

Where k_1 is the adsorption rate constant of ^{60}Co from the pseudo first order model given in (min^{-1}), k_2 is the adsorption rate constant of ^{60}Co from the pseudo second order model given in ($\text{g} \cdot \mu\text{Ci}^{-1} \cdot \text{mn}^{-1}$) and q_t is the quantity of adsorbate fixed at time t given in ($\mu\text{Ci/g}$) [2].

The parameters of the two models are determined from the plots's $\ln(q_e - q_t)$ versus t (Fig.6.a) and t/q_t versus t (Fig.6.b) for the pseudo first order model and second order respectively. The kinetic modeling results are presented in Table 2.

The characteristic curves of the pseudo-first and second order kinetic models relating to the ^{60}Co ions sorption onto IRN-77 resin at different temperatures, presented in the figures Fig.6.a and Fig.6.b show that they present a good linearity, which suggests the good compatibility of the experimental data of the ^{60}Co ions' adsorption onto this resin with the two kinetic models.

Table 2: Kinetic parameters of ^{60}Co ions sorption onto IRN-77.

Model	Parameters	Temperature (°K)			
		298	313	333	353
Pseudo-first-order	$q_{e \text{ Theo}} (\mu\text{Ci/g})$	160,686	154,713	189,639	203,310
	$k_1 (\text{min}^{-1})$	0,033	0,040	0,056	0,062
	R^2	0,9943	0,9927	0,9852	0,9861
	$q_{e \text{ Exp}} (\mu\text{Ci/g})$	176,013	167,934	193,413	216,076
Pseudo-second-order	$q_{e \text{ Theo}} (\mu\text{Ci/g})$	273,973	217,865	233,100	255,754
	$k_2 (\text{g} \mu\text{Ci}^{-1} \text{min}^{-1}) \cdot 10^{-4}$	0,558	1,893	2,491	2,852
	R^2	0,9764	0,9892	0,9905	0,9923
	$q_{e \text{ Exp}} (\mu\text{Ci g}^{-1})$	176,013	167,934	193,413	216,076

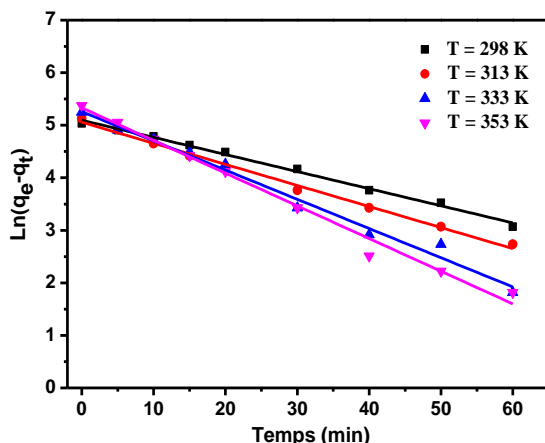


Fig 6.a. Lagergren's pseudo first order plots for the adsorption of ^{60}Co ions onto IRN-77 at different temperatures

(pH= 5.5 and S/L=5g/L).

According to the obtained results indicated in table 2, it should be noted that the correlation coefficients R^2 obtained from the pseudo 1st order model are very close to unity and relatively more important compared to those obtained from the pseudo 2nd order model.

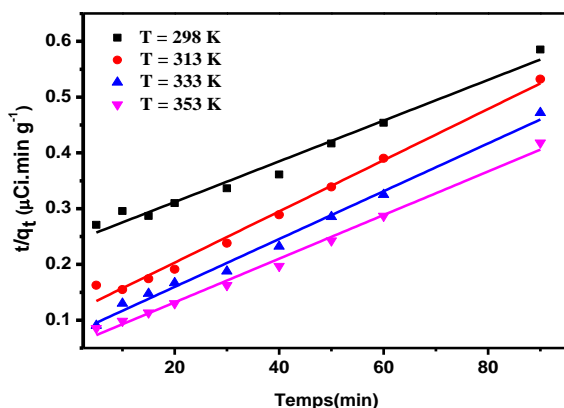


Fig 6.b. Second order plots for the adsorption of ^{60}Co ions onto IRN-77 at different temperatures (pH= 5.5 and S/L=5g/L).

As well as fixed quantities obtained at equilibrium and calculated theoretically $q_{e\text{Theo}}$ by applying the 1st order model are closer to the experimentally measured values $q_{e\text{Exp}}$ compared to those obtained for the pseudo 2nd order model. This suggests that experimental data of ^{60}Co ions sorption process onto IRN-77 resin are well fitted to the pseudo 1st order model and controlled by chemical adsorption [31].

Regarding the rate constant k_1 , it increases proportionally with increasing temperature in the range [298K; 353K], while concerning the rate constant k_2 , it decreases by increasing the temperature, which shows that the temperature factor influences the adsorption process of ^{60}Co ions on the IRN-77 resin.

3.5. Thermodynamic study

Depending on the type of the adsorbent surface and the adsorbed pollutant, adsorption can be an endothermic or exothermic phenomenon.

Due to the high dependence of the thermodynamic parameters of the adsorption process on temperature, the latter play an important role in the prediction of the adsorption mechanism [2]. These parameters can be obtained by combining the thermodynamic equation and that of Van't Hoff for achieve the equation (Eq.6) [2]:

$$\ln K_c = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (6)$$

Where: ΔH° is the standard enthalpy given in (kJ mol^{-1}); ΔS° is the entropy that expresses the affinity of a solute for an adsorbent given in ($\text{J mol}^{-1}\text{K}^{-1}$); ΔG° is the Gibbs free energy given in (kJ mol^{-1}); T is the absolute temperature (K), and R is the gas constant ($8.314 \text{ J mol}^{-1}\text{K}^{-1}$). These thermodynamic parameters are obtained from the plot K_c as a function of $1/T$ (Fig.7).

The equilibrium constant K_c is defined as [2]:

$$K_c = \frac{(C_0 - C_e)}{C_e} \quad (7)$$

The thermodynamic parameters of the adsorption process of ^{60}Co ions on the IRN-77 resin are obtained using equations (6) and (7) by varying the temperature between 293K and 353K. The results are summarized in Table 3.

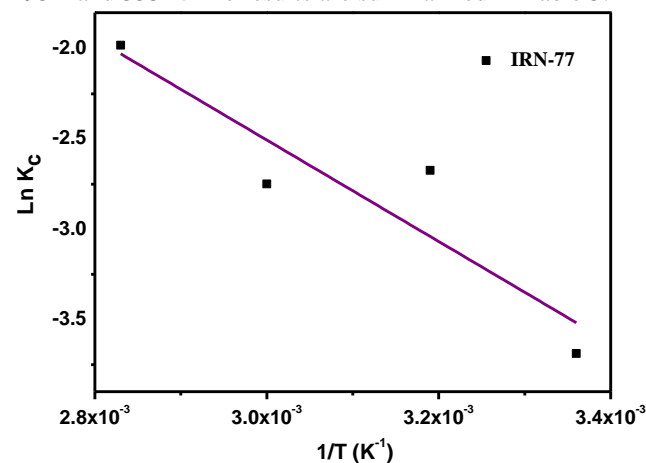


Fig.7. Plot of $\ln K_c$ as a function of $1/T$ relating to the sorption of ^{60}Co ions IRN-77 at different temperatures (pH = 5.5 and S/L = 5g/L).

It should be noted from table 3 that the calculated values of free energy ΔG° are positive over the whole temperature range, which indicates that ^{60}Co ions sorption process onto IRN-77 is not spontaneous and not favored over this temperature range.

The positive value of the enthalpy ΔH° (23.537 kJ/mol) shows that the adsorption process of ^{60}Co ions is endothermic.

Table 3: Thermodynamic parameters of ^{60}Co ions sorption process onto IRN-77.

parameters	T (K)	ΔH° (kJ/mol)	ΔS° (J/mol.K)	ΔG° (kJ/mol)
IRN-77	298	23.537	49.814	8.692
	313			7.945
	333			6.949
	353			5.953

It is also noted that the value of the entropy ΔS° of the ^{60}Co ions' adsorption is positive and moderately high; this indicates that there is an increase in disorder in the system, and reveals a strong affinity between the ^{60}Co ions in the liquid film and the adsorbent resin in the solid film [32].

4. Conclusion

The main objective of the undertaken study was the valorization of an IRN-77 resin for the adsorption of ^{60}Co ions using gamma spectrometry as a means of analysis. First of all, we proceeded to characterized this resin by a series of analyzes (SEM and FT-IR) in order to observe the behaviour of this material before use it. The experimental data of the isotherm adsorption study are faithfully represented by the Langmuir model due to the value of the

correlation coefficient very close to unity by obtaining a very important maximum adsorption quantity of 10.620 μCi of ^{60}Co per 1 gram of IRN-77 adsorbent. The kinetic study shows that the experimental data relating to the Co^{2+} ions adsorption onto IRN-77 resin perfectly follow the pseudo first-order model ; due to the correlation coefficient value very close to unity and the difference in the values of the fixed quantities between those calculated by the pseudo first-order model $q_{e\text{Theo}}$ and those measured experimentally $q_{e\text{Exp}}$. The thermodynamic parameters determination related to the Co^{2+} ions adsorption process shows that the process is favored at high temperature with a positive value of the

enthalpy ΔH° of 23.537 kJ/mol. As the findings of this study showed a good solution for ^{60}Co radionuclide removal from wastewater in nuclear applications.

Acknowledgements

We would like to thank the Draria Nuclear Research Center (CRND) for its valuable scientific cooperation.

Conflict of Interest

We state that we have no conflict of interest

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Recommended Citation

Achour S, Nibou D, Amokrane S. Gamma spectrometry technique application to the ^{60}Co sorption onto IRN-77 resin from radioactive wastewater: Equilibrium, Kinetic and Thermodynamic investigations. *Alger. J. Eng. Technol.* 2023, 08(2):193-200. <https://dx.doi.org/10.57056/ajet.v8i2.122>



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