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RESEARCH PAPER

Study of scandium sorption from a solution of leached waste from titanium production with hydrochloric acid

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ABSTRACT

This study examines the sorption of scandium from hydrochloric acid (chloride) solutions using cation exchange resins: Purolite C-150H$^+$ and Purosorb SAC140H$^+$. The resins were compared based on their sorption performance, including extraction efficiency, static exchange capacity, and selectivity in the presence of accompanying elements (Al and Ti). Optimal sorption conditions—pH, solid-to-liquid phase ratio, and temperature—were determined experimentally.The results show that scandium sorption on Purolite C-150H$^+$ follows the Langmuir isotherm ($R^2 = 0.9269$), indicating monolayer adsorption on a homogeneous surface. In contrast, sorption on Purosorb SAC140H$^+$ is better described by heterogeneous adsorption models: Freundlich ($R^2 = 0.433$) and Dubinin–Radushkevich ($R^2 = 0.647$) isotherms.

Keywords: Scandium, sorption, SMTC, exchange capacity

INTRODUCTION

Scandium is a trace element and is found in many minerals. There are two known scandium minerals: tortweite and sterrettite, but neither of these minerals is of industrial significance. Scandium is mainly extracted as a by-product from complex raw materials, where its content does not exceed a few hundredths of a percent, but there are many accompanying elements. Significant resources of scandium are concentrated in titanium-magnesium production waste (100-350 g/t) and bauxite processing waste (10-200 g/t).[1].

The most important areas of practical application for metallic scandium are the alloying of aluminum and magnesium alloys, and its oxide Sc_2O_3 is used to produce wear-resistant ion- and electrically conductive ceramics. Scandium is unique in that even a small addition to aluminum creates a material with a very high strength-to-density ratio. Another important area of application is the use of scandium in solid oxide fuel cells, where a zirconium-stabilized electrolyte with added scandium increases efficiency. [2-8]. Due to these characteristics, scandium has been included in the European Union's list of critical raw materials (CRM), which underscores its importance and the need to ensure a stable supply. [9].

Scandium is mainly present in trace amounts in waste generated during the processing of titanium, aluminum, and nickel ores. The largest volume of Sc_2O_3 production in the world is provided by the People's Republic of China, where scandium is extracted as a by-product in the process of obtaining titanium dioxide (TiO_2) [10, 11]. However, due to the high cost and complexity of processing TiO_2 waste, new technological solutions that allow scandium to be extracted from such waste at minimal cost are becoming promising.

It has been established that during the smelting of titanium concentrate, 90% of scandium is transferred to titanium slag, concentrating 1.6 times, and during subsequent chlorination, it remains in the melt. During chlorination, several elements associated with titanium (vanadium, niobium, tantalum) are removed mainly with gaseous chlorination products, while other elements (scandium, zirconium, thorium, REEs) are concentrated in the melt [12].

Chlorination of titanium slag is carried out in shaft chlorinators filled with molten potassium, magnesium, and sodium chlorides at a temperature of about 800 °C. Anodic chlorine gas is fed into the lower part of the reactor through tuyeres, and the charge is dosed from the upper part of the reactor onto the melt surface [13]. During the process, titanium dioxide reacts with chlorine in the presence of carbon, resulting in the formation of titanium tetrachloride and carbon oxides. Impurities contained in the slag are also chlorinated to prevent excessive accumulation. The melt is renewed by feeding spent melt from magnesium electrolysers and periodically draining the melt. Part of the unreacted titanium dioxide and carbon is also removed from the reactor together with the melt. As is known, titanium raw materials contain Sc_2O_3 (up to 0.1% in ilmenite, up to 0.3% in sphene). The spent melt from the titanium chlorinator (SMTC) is stored in big bags and sent as waste to storage facilities, accounting for the largest part of the chloride waste from titanium-magnesium production. Up to 30,000 tons of SMTC are dumped into sludge fields annually. Consequently, SMTC contains titanium and valuable components associated with titanium slag, including scandium. To extract it, SMTC is usually leached with water, but a significant number of impurities are transferred to the solution [14]. The main methods for separating scandium from impurities include precipitation, condensation and sublimation, ion exchange, and extraction.

Extraction with organic solvents is one of the key methods for purifying and concentrating scandium from acid solutions obtained after leaching. The most effective extractants are organophosphorus compounds such as di(2-ethylhexyl) phosphoric acid (D2EHPA), tributyl phosphate (TBP), and Cyanex. The advantages of the method are high selectivity for scandium and the ability to fine-tune the process parameters (pH, component ratio) to achieve maximum extraction. In particular, the synergistic D2EHPA + TBP system demonstrates an efficiency of over 98% scandium extraction from acid solutions [15]. At the same time, among the disadvantages, it is worth noting the use of toxic and flammable solvents, as well as the need for their regeneration and disposal of the resulting waste.

Another method for extracting scandium from a solution is sorption. Sorption extraction of scandium is carried out using specialized ion-exchange resins (e.g. Purolite, Lewatit) and organic sorbents, which allows for the efficient extraction of scandium from dilute acid solutions after leaching. The main advantages of the method are high selectivity under pH control, as well as the possibility of regeneration and reuse of sorbents. However, the efficiency of sorption may decrease with a high content of accompanying metals, such as iron and titanium, and also strongly depends on the composition of the solution and process parameters. [16,17].

Given the limited number of ores with high scandium content, the development and improvement of technologies for processing secondary raw materials (technogenic waste, red mud, tailings) is becoming a priority. The use of leaching, sorption, and solvent extraction technologies ensures high yield and selectivity in scandium extraction. However, further optimization of processes is important to improve environmental safety and economic efficiency. This paper presents the results of experiments on the optimization of the process of sorption of scandium from a solution leached with hydrochloric acid using Purolite C-150H$^+$ and Purosorb SAC140H$^+$ cation exchangers.

MATERIAL AND METHODS

Chemically pure hydrochloric acid and ion exchange resins from Purolite and Purosorb were supplied in the sodium form (Na$^+$ form). The main characteristics of the Purolite C-150H$^+$ and Purosorb SAC140H$^+$ sorbents are presented in **Table 1**.

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Table 1 Characteristics of sorbents

Brand	Functional group	Structure (polymer base)	Resin type
Purolite C-150H$^+$	Sulfonic acid group (–SO$_3$H)	Styrene-divinylbenzene copolymer	Cation exchange, gel
Purosorb SAC140H$^+$	Sulfonic acid group (–SO$_3$H)	Styrene-divinylbenzene copolymer	Cation exchange, macroporous

To convert the sorbents to the H$^+$ form, the procedure specified in GOST 10896-78 was followed. Pre-swollen cation exchange resin in the Na$^+$ form was loaded into a column and treated with a 10% NaOH solution at a ratio of 5 volumes of solution per 1 volume of sorbent. The resin was then washed with five bed volumes of water to remove residual alkali, after which a 5% hydrochloric acid solution was passed through until the acid concentration in the filtrate matched the initial concentration.

In previous work on the aqueous leaching of SMTC, we established the optimal leaching parameters for maximum scandium extraction into solution: T = 25 °C, t = 60 min, and S:L = 1:10. This leachate was subsequently acidified with hydrochloric acid to a concentration of 3%, producing solutions suitable for scandium sorption. The concentrations of the main elements monitored during the sorption experiments are shown in **Table 2**.

Table 2 Concentration of scandium and impurities in the initial solution

Name	Sc	Fe	Al	Ti
C$_0$, g/l	0.0643	14.9828	0.1049	0.0489

Two cation exchangers in two forms were selected for the sorption of scandium from a chloride solution: Purosorb SAC 140 H+, Purosorb SAC 140 Na+, Purolite C-150 H+, Purolite C-150 Na+ in order to determine the optimal ion exchanger. Sorption was carried out on a KS 3000 ic control incubator-shaker IKA (Germany) at a temperature of 25°C, with a mixing time of 1 hour, a solution volume of 100 ml, and a sorbent mass of 1 gram. The results are shown in **Table 3**.

Table 3 Sorption of scandium on cation exchangers using the static method

Name of anionite	Sorbent mass, g	Solution volume, ml	Scandium extraction into sorbent, %
Purosorb SAC 140 Na +	1	100	0
Purolite C-150 Na +	1	100	4,04
Purosorb SAC 140 H+	1	100	76,51
Purolite C-150 H+	1	100	78,84

According to the results in **Table 3**, the highest extraction of scandium into the sorbent was observed for cation exchangers in the hydrogen form, which was an order of magnitude higher than for cation exchangers in the sodium form. Thus, further research on scandium sorption was conducted on Purosorb SAC 140 H+ and Purolite C-150 H+ cation exchangers.

Analysis methods. The content of scandium and other components of the solution was determined using an Optima 2000 DV inductively coupled plasma optical emission spectrometer (Perkin Elmer, USA).

The main characteristics of the sorption process include the extraction of the target component onto the sorbent and the static sorption capacity of the sorbent.

The formula determines the extraction of scandium into sorbent E:

$$E = \frac{C_0 - C_e}{C_e} x100\% \qquad (1)$$

where C$_0$— is the concentration of scandium in the initial solution, mg/L; Ce is the residual equilibrium concentration of niobium in the solution, mg/L.

The formula determined the static exchange capacity of the sorbent q:

$$q = \frac{(C_0 - C_e)V}{m} \qquad (2)$$

where V is the volume of the solution, ml; m is the mass of the dry sorbent, g.

RESULTS AND DISCUSSION

Effect of S:L ratio on the sorption of scandium and impurities. The solid-to-liquid ratio (S:L) is an important parameter determining the efficiency of sorption. A change in this ratio affects both the degree of sorbate extraction and the equilibrium sorption capacity of the sorbent. **Figs 1 and 2** show data on the effect of the S:L ratio on the sorption of scandium and impurities using Purolite C-150H$^+$ and Purosorb SAC140H$^+$ sorbents, respectively.

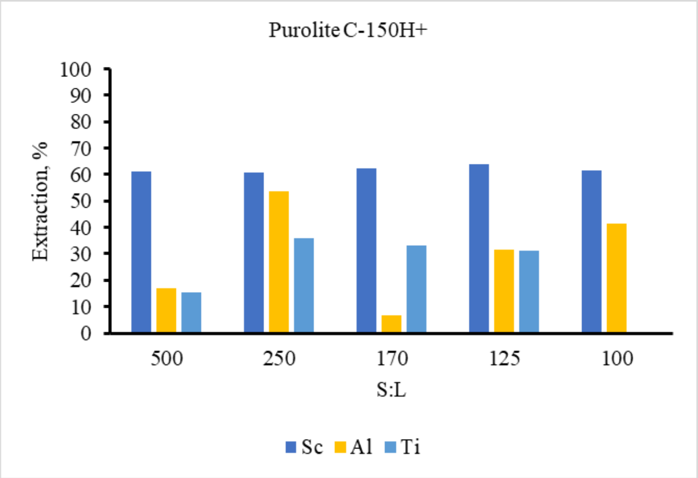

Fig. 1 Effect of the S:L ratio on the sorption of scandium and impurities by the Purolite C-150H+ sorbent

Fig. 1 shows that when S:L is reduced from 500 to 100 for the Purolite C-150H+ sorbent, a consistently high degree of Sc extraction (from 60% to 64%) is observed. The highest sorption is achieved at S:L = 125–170 (64.05 and 62.21%), which indicates optimal conditions for Sc extraction in this range. Al sorption remains low in all S:L ranges, fluctuating between ~5–54%. The minimum sorption of Al is observed at S:L = 170, which indicates high selectivity of the sorbent to Sc under these conditions. The extraction of Ti is at the level of 20–30% and is slightly dependent on changes in S:L. A slight increase in extraction is observed with a decrease in S:L, but the values remain moderate, which also confirms the selectivity of Purolite C-150H+ towards Sc. The selected cation exchangers Purolite C-150H+ and Purosorb SAC140H+ do not sorb iron at room temperature.

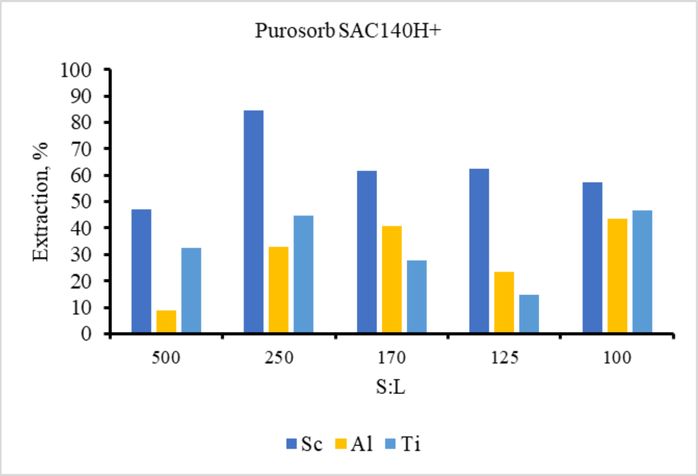

Fig. 2 Effect of the S:L ratio on the sorption of scandium and impurities by the Purosorb SAC140H+ sorbent

The sorption of Sc by the Purosorb SAC140H+ sorbent also shows high values (**Fig. 2**), especially at S:L = 250 and 170, reaching up to ~80%. At S:L = 500 and 100, the extraction decreases to ~60%, which is similar to the trend observed for the previous sorbent. Overall, Purosorb SAC140H+ shows good Sc extraction capacity, but its efficiency is slightly lower than that of Purolite C-150H+ at low S:L. Al extraction is higher than that of Purolite C-150H+, reaching up to 40% at S:L = 170–100. This indicates lower selectivity of SAC140H+ for Sc, especially at low S:L ratios.

Effect of temperature on the sorption of scandium and impurities

Temperature is one of the key factors affecting the mechanism and efficiency of the sorption process. Temperature changes can affect sorption capacity in different ways depending on the nature of the sorbent, sorbate, and the type of interactions between them. **Figs 3 and 4** show the effect of temperature on the sorption of scandium (Sc) and associated impurities (Fe, Al, Ti) using Purolite C-150H+ and Purosorb SAC140H+ sorbents.

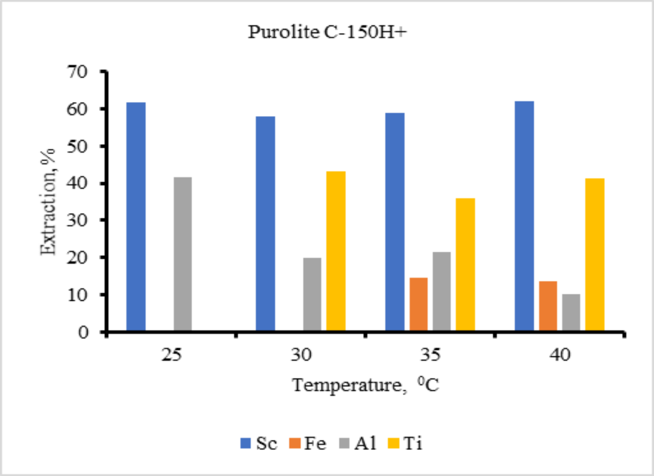

Fig. 3 Effect of temperature on the sorption of scandium and impurities by the Purolite C-150H+ sorbent

The effect of temperature on the sorption of Sc by the Purolite C-150H+ sorbent is insignificant (**Fig. 3**), which may indicate a weak thermodynamic dependence and a stable sorption mechanism. As the temperature increases, the sorbent begins to sorb iron, considering that an iron concentration greater than 10 g/l significantly affects the sorption of other elements.

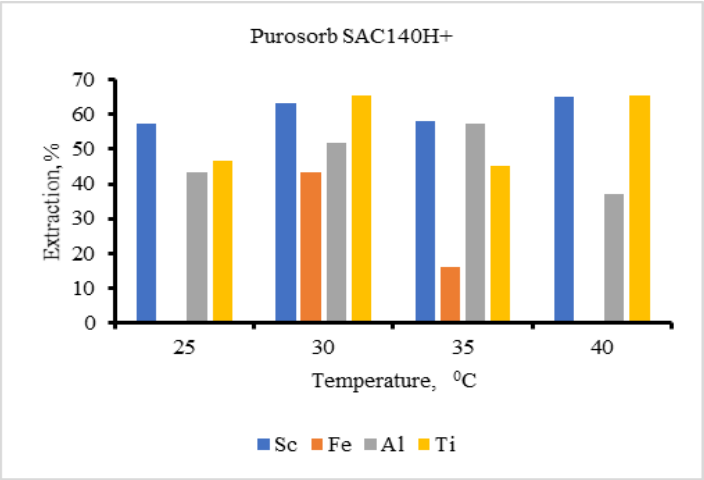

Fig. 4 Effect of temperature on the sorption of scandium and impurities by the Purosorb SAC140H+ sorbent

Titanium, aluminum, and scandium demonstrate consistently high extraction rates across all temperature ranges for the Purosorb SAC140H+ sorbent. At temperatures between 30 and 35 °C, iron is additionally sorbed, which indicates that increasing the temperature is undesirable for scandium sorption.

Effect of pH on the sorption of scandium and impurities

The effect of pH on the scandium sorption process was studied in the range from -1 (formed during leaching of SMTC 3% with hydrochloric acid) to 3. The results of scandium sorption on the Purolite C-150H+ cation exchanger, depending on pH are presented in **Fig. 5**.

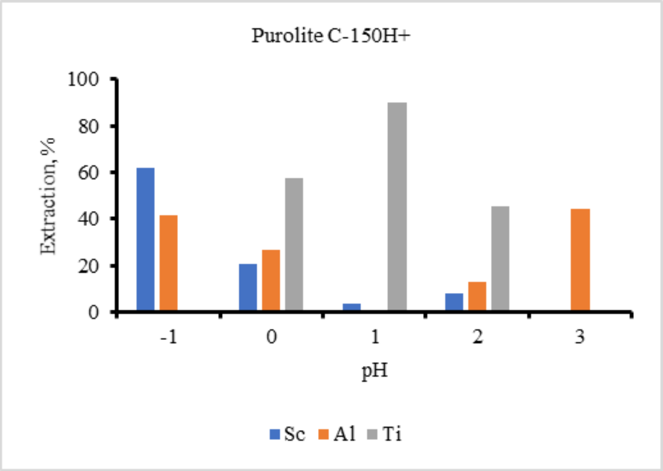

Fig. 5 Effect of pH on the sorption of scandium and impurities by the Purolite C-150H+ sorbent

Sorption by the Purolite C-150H+ sorbent under extremely acidic conditions (pH = –1) is about 60%, which indicates the sorbent's good ability to extract Sc in an aggressive environment. As the pH increases, Sc extraction decreases, reaching a minimum at pH = 1 (~10%). If the pH of the solution is increased to 3 at iron concentrations greater than 10 g/L, scandium precipitates. Al sorption rises from 10% at pH = –1 to 40% at pH = 0 and 1. Further, at pH = 2–3, a smooth increase to ~60% is observed, indicating a tendency for Al sorption in a more neutral environment.

The results of scandium sorption on the Purosorb SAC140H+ cation exchanger as a function of pH are shown in **Fig. 6**.

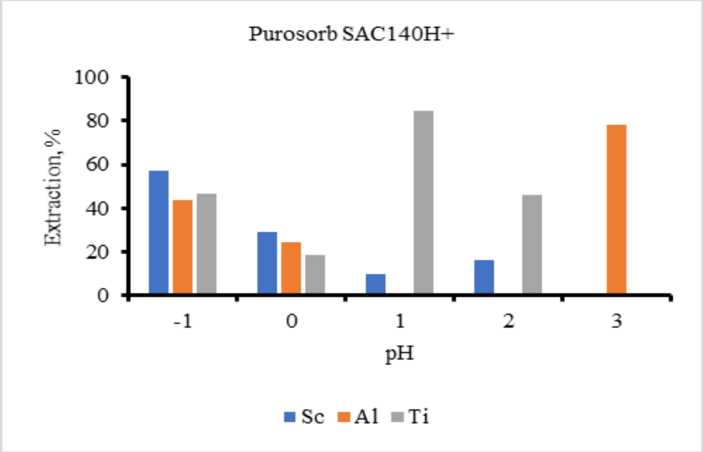

Fig. 6 Effect of pH on the sorption of scandium and impurities by the Purosorb SAC140H+ sorbent

For scandium, the Purosorb SAC140H+ sorbent works well at low pH values (pH = –1). At pH = 1–3, Sc extraction is ~20%, which indicates a preference for an acidic environment for effective Sc sorption. Aluminum sorption by the Purosorb SAC140H+ sorbent is moderate in an acidic environment (pH = –1 to 1): ~40–50%. However, at pH = 3, there is a sharp increase in Al extraction to 80%, which significantly reduces the selectivity of scandium sorption.

Study of the mechanism of scandium sorption by Purosorb SAC140H+ and Purolite C-150H+ sorbents from hydrochloric acid solution

The study of sorption isotherms plays a key role in understanding and optimizing the processes of sorption extraction of target components from liquid or gaseous media. It is necessary both for fundamental analysis of the interaction between the sorbent and sorbate, and for practical application in industrial technologies for purification, extraction, and concentration of substances. The study of isotherms allows us to establish the nature of the interaction between the sorbent and the sorbate: whether it is physical or chemical sorption, whether sorption occurs in a single layer (monolayer) or in several layers, and whether adsorption occurs on a homogeneous or heterogeneous surface. These data are important for understanding the nature of the sorption process.

To study the equilibrium sorption of scandium from hydrochloric acid solution by two cation exchangers, Purosorb SAC140H+ and Purolite C-150H+, three well-known models were used in this work: Freundlich, Langmuir, and Dubinin-Radushkevich (D-R). The Dubinin-Radushkevich (D-R), Freundlich, and Langmuir isotherms are mathematical models describing the equilibrium between the amount of substance sorbed on the sorbent surface and its concentration in solution at a constant temperature. They allow us to understand the mechanism of sorption and the nature of the interaction between the sorbent and the sorbate.

According to Langmuir's isotherm model, adsorption occurs in a single layer (one molecule thick) on a certain number of fixed, homogeneous, and energetically equivalent active centers, between which there are no lateral interactions or spatial obstacles, even if they are located close to each other [18]. The model assumes that adsorption occurs on a homogeneous surface where each adsorbate molecule interacts with the same energy and the movement of molecules across the surface is excluded.

The equation describes the Langmuir isotherm model:

$$q_e = \frac{C_e K_L q_{max}}{1+C_e K_L} \tag{3}$$

q_e — equilibrium sorption (mg/g), q_{max} — maximum sorbent capacity (mg/g), C_e — equilibrium concentration in solution (mg/l), K_L — Langmuir constant (l/mg).

Figs 7 and 8 show the linear dependence in the Ce/qe and Ce coordinates, characterizing the sorption of scandium by Purosorb SAC140H+ and Purolite C-150H+ cation exchangers, respectively, within the Langmuir isotherm model.

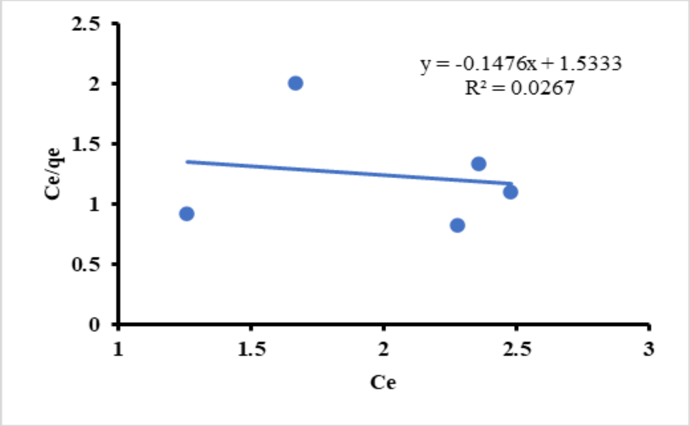

Fig. 7 Langmuir isotherm after sorption of scandium by Purosorb SAC140H+ sorbent

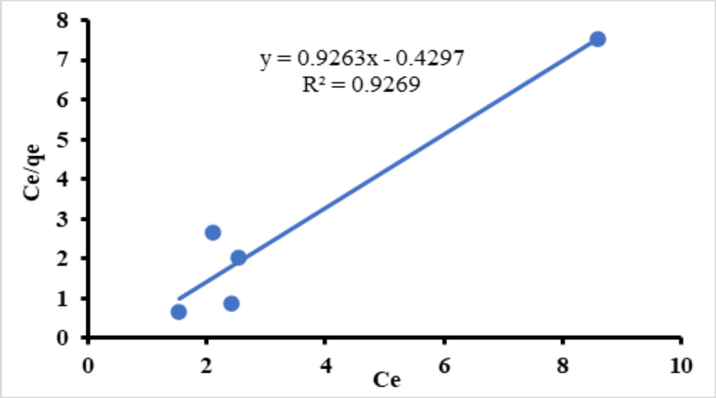

Fig. 8 Langmuir isotherm after sorption of scandium by Purolite C-150H+ sorbent

The Langmuir model poorly describes the sorption process of scandium on Purosorb SAC140H+ (Fig. 7). This is indicated by the correlation (R2 = 0.0267) and the negative slope of the line, since the slope should be positive within the Langmuir model. For the Purolite C-150H+ sorbent (**Fig. 7**), there is a high correlation coefficient (R2=0.9269), which indicates good agreement between the experimental data and the Langmuir model.

The Freundlich isothermal model assumes that the adsorption process takes place on a heterogeneous surface where the energy of active centers is unevenly distributed and interactions between adsorbed molecules are possible [18].

The following equation describes this model:

$$q_e = K_f \times C_e^{\frac{1}{n}} \tag{4}$$

By taking the logarithm, we obtain the equation:

$$\log q_e = log K_f + \frac{1}{n} log C_e \tag{5}$$

where Ce is the equilibrium concentration of Sc, mg/L; q_e is the amount of Sc adsorbed at equilibrium, mg/g; K_f is the Freundlich constant related to adsorption capacity, L/g; n is the constant related to adsorption intensity.

Figs 9 and 10 show Freundlich isotherm graphs for the sorption of scandium using two different sorbents: Purosorb SAC140H$^+$ and Purolite C-150H$^+$. The model parameters were evaluated based on the linear dependence between the logarithms of the equilibrium concentration (log Ce) and the sorbed amount (log qe).

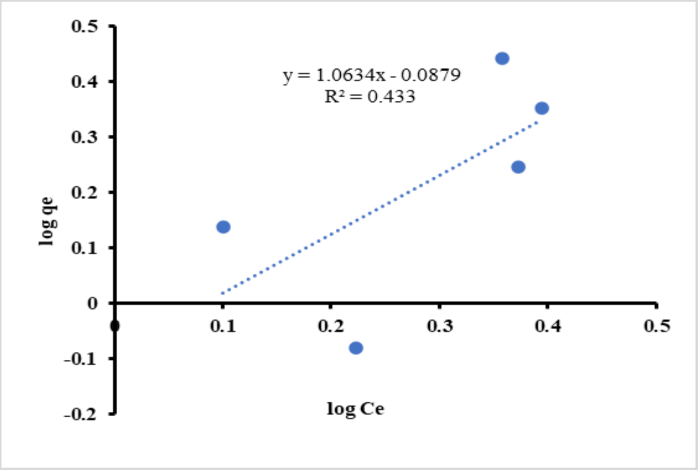

Fig. 9 Freundlich isotherm after sorption of scandium by Purosorb SAC140H+ sorbent

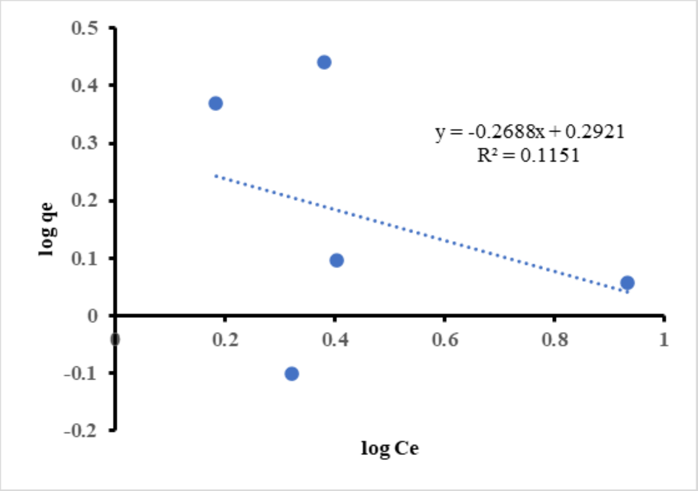

Fig.10 Freundlich isotherm after sorption of scandium by Purolite C-150H+ sorbent

Purosorb SAC140H$^+$ shows better fit to the Freundlich model compared to Purolite C-150H$^+$, with $R^2 = 0.433$ indicating moderate fit of the Freundlich model to the experimental data, which allows it to be used to describe the sorption of scandium on a heterogeneous surface (**Fig. 9**).

The sorption process on Purolite C-150H$^+$ does not follow Freundlich's model. The value $R^2 = 0.1151$ indicates a poor approximation of the model and a strong discrepancy between the calculated and actual data. The negative slope of the line ($1/n < 0$) theoretically does not correspond to the Freundlich model, since the parameter n must be positive and greater than 1 for a physically reasonable interpretation (**Fig. 10**).

The Dubinin–Radushkevich isotherm model is based on the assumption of a Gaussian distribution of adsorption energy on a heterogeneous surface, which allows determining the nature of the interaction—physical or chemical [19]. Unlike the Langmuir isotherm, this model is more universal, since it does not require assumptions about the uniformity of the surface and the constancy of the adsorption energy potential [20,21]. The mathematical expression of the Dubinin–Radushkevich isotherm is presented in the following form [28,29]:

$$\log q_e = \log q_{DR} - B_{DR}(\varepsilon)^2 \qquad (6)$$

$$\varepsilon = RT\ln\left(1+\frac{1}{C_e}\right) \qquad (7)$$

where qe (mg/g) is the amount of adsorbed niobium ions at equilibrium concentration; qDR (mg/g) is the theoretical saturation capacity; BDR (mol2/kJ2) is the Dubinin-Radushkevich isothermal constant associated with the adsorption energy; ε is the Polanyi potential (kJ/mol), reflecting the isothermal work of transferring one mole of niobium from the equilibrium solution volume to the sorbent surface; Ce (mol/L) is the concentration of niobium in the solution at equilibrium; R is the universal gas constant; and T (K) is the absolute temperature.

Figs 11 and 12 show linear approximations of Dubinin-Radushkevich isotherms for the sorption of scandium ions using two ion exchange resins: Purosorb SAC140H$^+$ and Purolite C-150H$^+$. This model allows us to evaluate the sorption mechanism and energy parameters of the process, which are particularly relevant for microporous materials.

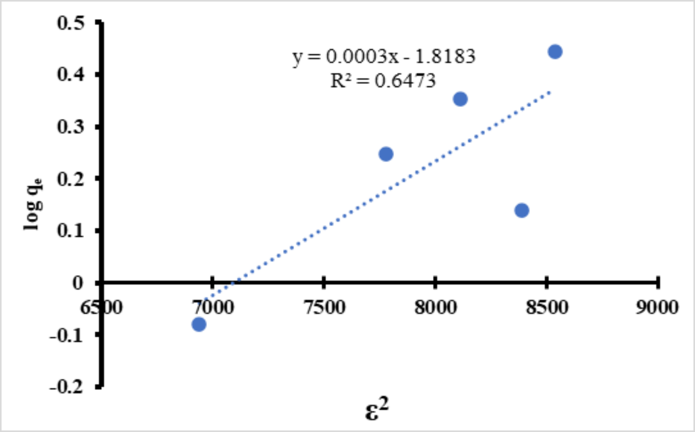

Fig. 11 Dubinin-Radushkevich isotherm after sorption of scandium by Purosorb SAC140H+ sorbent

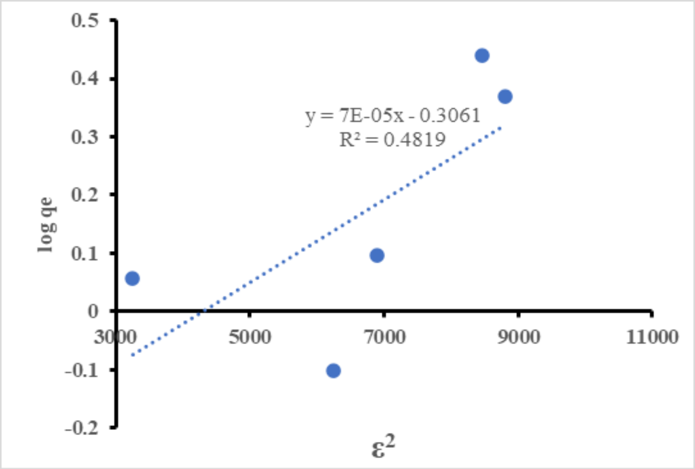

Fig. 12 Dubinin-Radushkevich isotherm after sorption of scandium by Purolite C-150H+ sorbent

The Dubinin–Radushkevich model showed satisfactory agreement with experimental data for Purosorb SAC140H+. The value of $R^2 = 0.65$ indicates a relatively good approximation. The positive slope may indicate atypical behavior, but given the logarithmic form used, it can be interpreted as a complex process. This confirms the heterogeneous nature of the Purosorb SAC140H$^+$ sorbent surface and the presence of micropores.

For the Purolite C-150H$^+$ sorbent, the model also shows moderate agreement with the experiment, but the accuracy of the approximation is lower ($R^2 \approx 0.48$).

CONCLUSION

The study conducted a comparative assessment of the sorption properties of Purolite C 150H$^+$ and Purosorb SAC140H$^+$ cation exchangers in the extraction of scandium from chloride solutions obtained by leaching spent titanium-magnesium production melts. The highest sorption efficiency is achieved when using cation exchangers in the hydrogen form, while sodium forms exhibit extremely low activity. The optimal sorption conditions are determined by the ratio of the solid and liquid phases (S:L = 125–170 for Purolite C 150H$^+$ and 170–250 for Purosorb SAC140H$^+$), as well as a low pH value (≈ –1), which ensures high selectivity for scandium. Increasing the process temperature has a negligible effect on scandium extraction, but promotes iron sorption, which reduces the selectivity of the process. Analysis of the isotherms showed that the sorption of scandium on Purolite C 150H$^+$ is best described by the Langmuir model ($R^2 \approx 0.93$), which indicates a monolayer distribution and a more homogeneous sorbent surface. Purosorb SAC140H$^+$ is characterized by heterogeneous sorption, corresponding to the Freundlich and Dubinin–Radushkevich models. Thus, the Purolite C 150H$^+$ cation exchanger is more promising for the selective extraction of scandium from hydrochloric acid solutions, ensuring high extraction of the target element with minimal sorption of impurity metals. The results obtained can be used to optimize the technology for sorption extraction of scandium from chloride waste from titanium-magnesium production.

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