7TH INTERNATIONAL CONFERENCE

Scientific and Research Cooperation between Vietnam and Poland

18–20 October 2023, Kraków, Poland

POL-VIET 2023

RESEARCH ON ADSORPTION OF Ni^{2+} IONS USING HALLOYSITE CLAY AND THE ABILITY OF DESORPTION AND NICKEL RECOVERY BY ELECTRODEPOSITION METHOD

Duyen Thi LE[1,3,*], Thao Phuong Thi LE[1,3], Dung Tien CONG[1], Hung Viet NGUYEN[1,3], Hung Manh HA[1], Thao Thanh Thi NGUYEN[2,3], Bac Hoang BUI[2,3]

[1] *Department of Chemistry, Faculty of Basic Science, Hanoi University of Mining and Geology, Vietnam*

[2] *Faculty of Geosciences and Geology Engineering, Hanoi University of Mining and Geology, Vietnam*

[3] *HiTech-CEAE Research Team, Hanoi University of Mining and Geology, Vietnam*

*Corresponding author: lethiduyen@humg.edu.vn

INTRODUCTION

In modern life, the advancement in industry and urbanization has led to serious metal pollution all over the world (Sharma et al., 2022). Nickel is a transition metal that is silvery-white, hard, ductile, highly corrosion-resistant. The concentration of nickel in various environmental compartments have been found to remarkably exceed natural levels. This causes harmful and dangerous effects on humans and the environment.

Natural halloysite material (HAL) is a clay mineral belonging to the kaolin group, which is also applied in many fields: pharmaceutical, medicine, food, high-grade materials and environment due to its superior properties such as microstructure, tube, non-toxic, high mechanical strength. HAL material in Thach Khoan - Phu Tho area, Vietnam has been used to adsorb Cd, Pb showing high adsorption efficiency as 51.45 % and 79.3 % respectively (Bac et al., 2021). In addition to heavy metal adsorption studies, the research to find effective methods of desorption and recovery of heavy metals and regeneration of adsorbent materials, minimizing negative impacts on the environment is very important. In this work, we adsorb Ni2+ ions on HAL clay, then desorption and recovery of Ni metal from loaded HAL material by electrolysis in reline (choline chloride-urea) electrolyte. The advantage of this study is the desorption and recovery process of nickel are carried out together in an electrochemical cell, after that HAL adsorbent can be reused.

EXPERIMENTAL

Adsorption experiments

The effect impacts on adsorption process was investigated such as:
- the contact time changed between 10 and 120 mins,
- the initial pH of the solution was studied between 2.3 and 6.9
- the mass of HAL in the range of 0.3 - 1.0 g.

The experiments were carried out at room temperature and kept stirring at a speed of 400 rpm.

Adsorption capacity and efficiency are calculated by equations (1) and (2), respectively (Gupta et al., 2012):

$$Q = (C_o - C).V/m \quad (1) \qquad H = (C_o - C).100/C_o \quad (2)$$

Preparation of HAL powder

Fig 1. Picture of halloysite powder

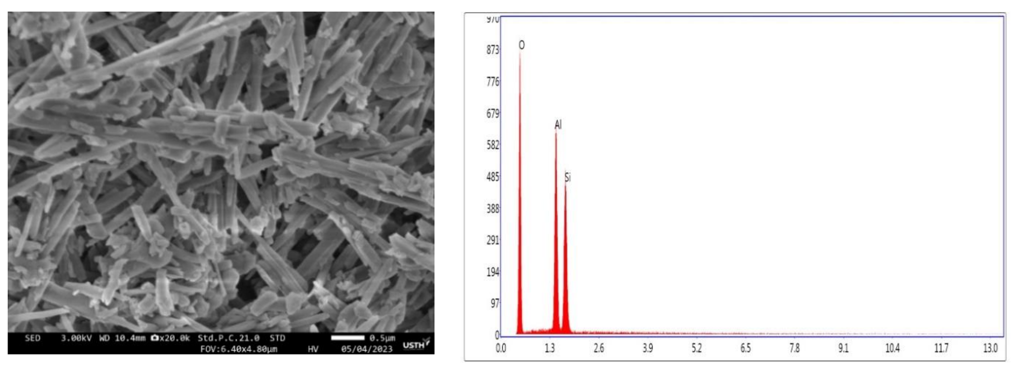

Fig 2. SEM-EDX image of halloysite

Desorption experiments

The desorption of Ni^{2+} from Ni-HAL and deposition Ni metal was carried out in a three-electrode electrochemical cell: the WE was gold plate (geometric area = 1 cm2), the RE was Ag,AgCl|Cl- and the CE was a platinum grid of large area.
- applying current: 1, 2, 3, 5, 7.5 mA;
- electrolytic time : 1-5 hours
- The deposition potential ≤ – 0.6 V, T = 60 oC.

RESULTS AND DISCUSSION

Effect of the experimental factors on Ni2+ ions adsorption by HAL powder

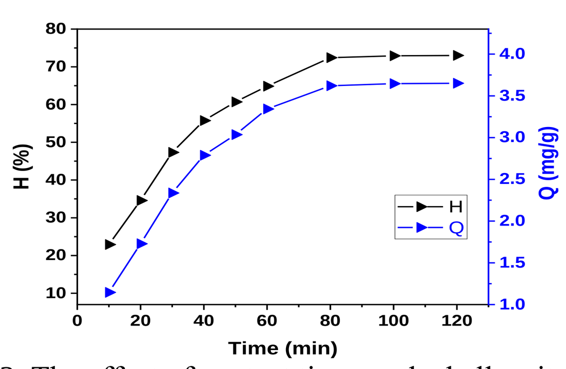

Fig 3. The effect of contact time on the halloysite's adsorption capacity and efficiency of Ni^{2+} at initial concentration of 50 mg/L; mass of 0.5 g; pH = 5.9

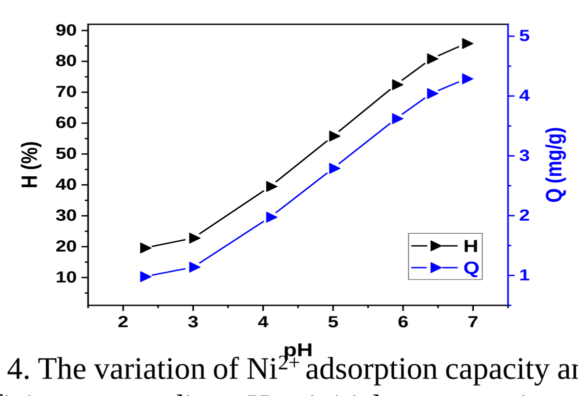

Fig 4. The variation of Ni^{2+} adsorption capacity and efficiency according pH at initial concentration of 50 mg/L; mass of 0.5 g; contact time of 80 mins

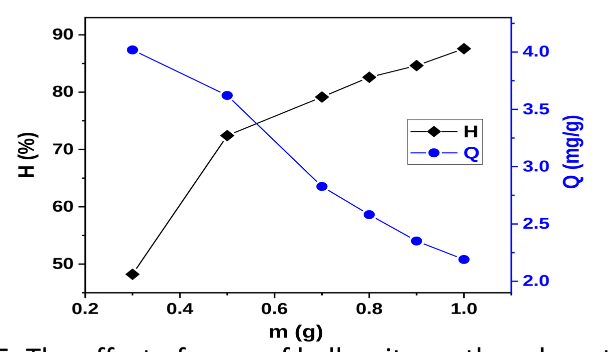

Fig 5. The effect of mass of halloysite on the adsorption capacity and efficiency of Ni2+ at initial concentration of 50 mg/L; pH 5.9; contact time of 80 mins

Characterization of HAL before and after adsorption process

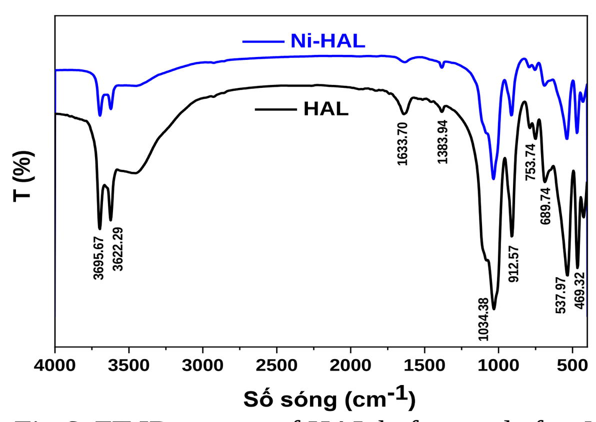

Fig 6. FT-IR spectra of HAL before and after Ni^{2+} adsorption process

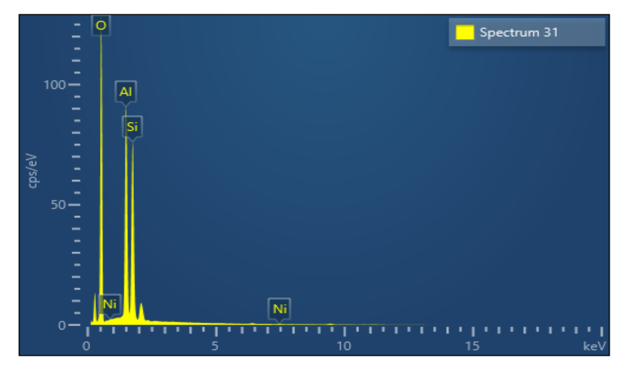

Fig 7. -EDX spectra of HAL after Ni^{2+} adsorption process

Adsorption isotherm curve

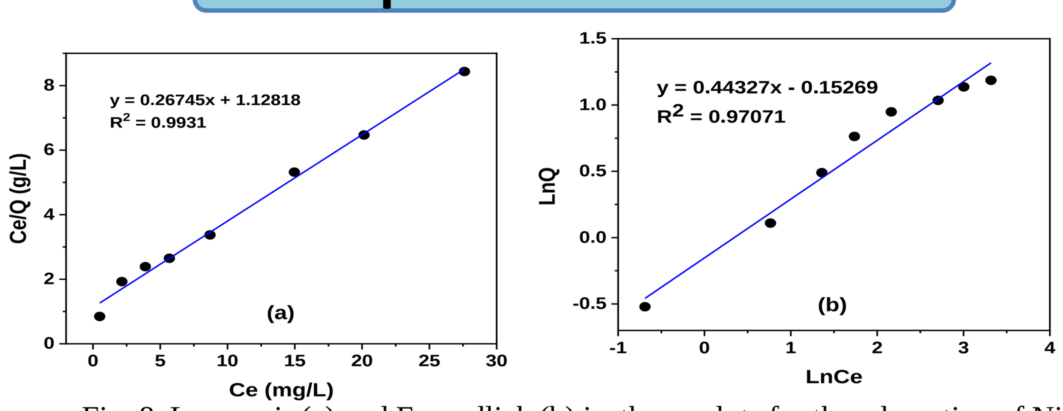

Fig 8. Langmuir (a) and Freundlich (b) isotherm plots for the adsorption of Ni^{2+} onto halloysite

Table 1. Adsorption isotherm parameters

Langmuir			Freundlich		
Q_m	K_L	R^2	n	K_F	R^2
3.739	0.237	0.9931	2.25596	0.85847	0.97071

Adsorption kinetic

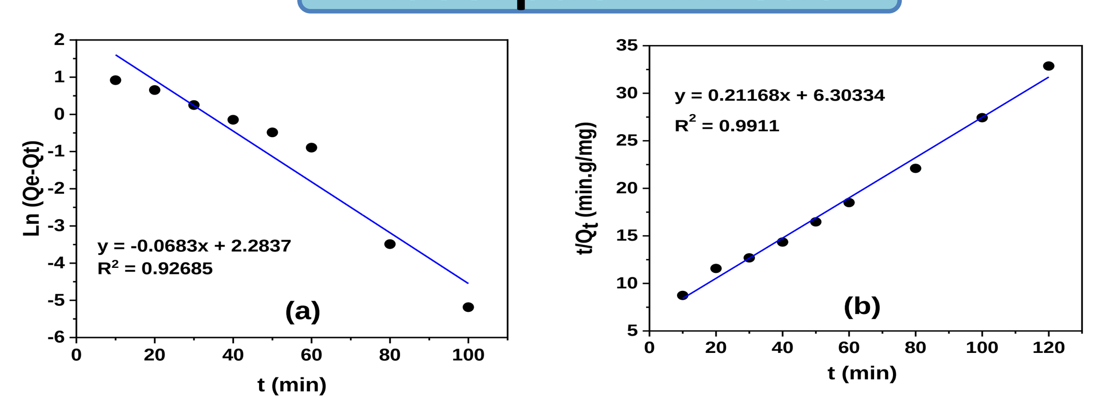

Fig 9. The description of the experimental data according to pseudo-first-order kinetic equantion (a) and pseudo-second-order kinetic equantion (b) for adsorption of Ni^{2+} onto halloysite

Table 2. Adsorption kinetic parameters

pseudo-first-order kinetic equantion			pseudo-second-order kinetic equantion			Q_e experiment (mg/g)
Q_e (mg/g)	k_1 (min^{-1})	R^2	Q_e (mg/g)	k_2 (g/mg/min)	R^2	
9.813	0.0683	0.92685	4.724	0.00711	0.9911	3.651

The appropriate adsorption conditions: 0.8 g halloysite/50 mL Ni^{2+} solution of 50 mg/L, contact time of 80 minutes at natural pH 5.9, room temperature (25 °C)

Desorption of Ni2+ out of Ni-HAL and recovery of Ni metal by electrocdeposition method

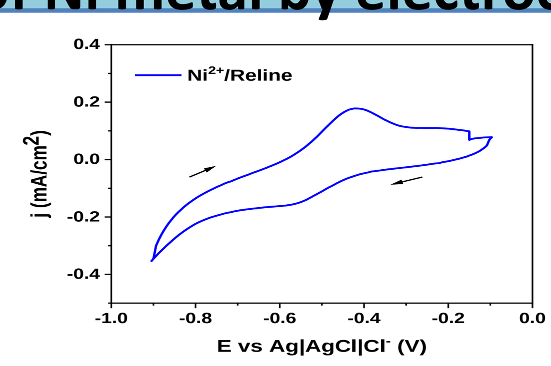

Fig 10. Cyclic voltammogram of 0.005M Ni2+ in the reline, scan rate of 50 mV.S-1, T = 60 oC

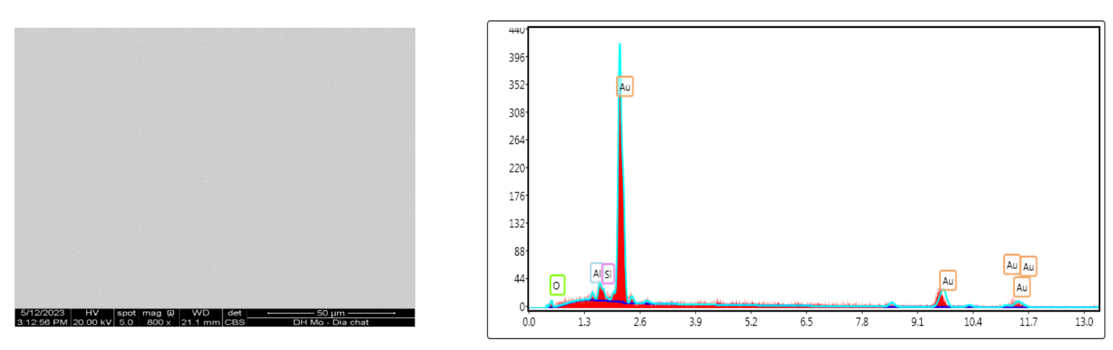

Fig 11. SEM-EDX image of the surface of Au plate electrode before electrolysis of Ni-HAL

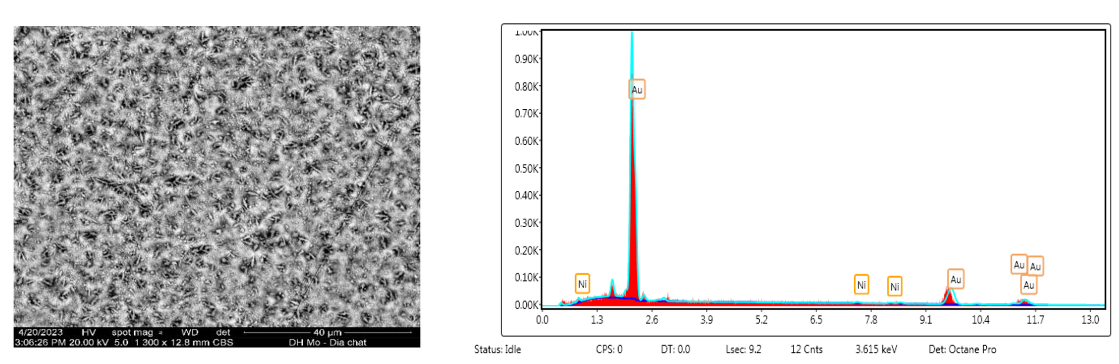

Fig 12. SEM-EDX image of the surface of Au plate electrode after electrolysis of Ni-HAL

Table 3: The recovery efficiency of Ni (H %) from 0.5 g Ni-HAL at different applied currents and electrolytic times

t (hour) \ I (mA)	H % 1	2	3	5	7.5
1	56,39	58,26	60,32	62,25	65,81
2	64,23	67,83	68,44	71,34	73,76
3	71,11	73,88	75,48	78,47	80,52
4	76,68	79,23	82,14	84,95	87,24
5	81,62	85,29	88,05	90,18	92,65

Appropriate desorption conditions: Ni-HAL mass of 0.5g, electrolysis time of 5 hours, applied current of 7.5 mA at 60 °C.

CONCLUSION

Halloysite clay was used to study the adsorption - desorption of Ni2+ ions from aqueous solution. The adsorption efficiency of Ni^{2+} obtained 82.59 % at appropriate conditions: contact time of 80 mins, pH 5.9, HAL mass of 0.8 g. The results of adsorption isotherms showed that adsorption of Ni^{2+} ions using HAL powder followed Langmuir isothermal model with the maximum mono layer adsorption capacity of 3.739 mg/g. The experiment data of adsorption kinetics confirmed that Ni^{2+} adsorption process followed the pseudo-second-order law with the correlation coefficient (R^2) of 0.9911. The recovery efficiency of nickel reached 92.65 % from loaded HAL material by electrodeposition on the surface of Au plate electrode at the applied current of 7.5 mA after 5 hours in the reline solvent. HAL material was not dissolved in the solvent of reline and can be reused after desorption process.

REFERENCE

[1]. Sharma, R., Saini, K. C., Rajput, S., Kumar, M., Mehariya, S., Karthikeyan, O. P., & Bast, F. (2022, Strateg. Tools Pollut. Mitig. (2022). DOI:10.1007/978-3-030-98241-6_10

[2]. Bac, B. H., Nguyen, H., Thao, N. T. T., Duyen, L. T., Hanh, V. T., Dung, N. T., . . . An, D. M. (2021). Chemosphere, 282, 131012. DOI: 10.1016/j.chemosphere.2021.131012