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## Nickel sorption from solutions with high salt concentration

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**Abstract.** The object of the study is salt concentrates formed as a result of treatment of concentrated acidic and alkaline wastewater of galvanic processes, realized on Ural Optical & Mechanical Plant (Russia). Salt concentrates are to be disposed of as low-hazard substances, but periodically the presence of nickel ions in them was detected. Concentration of nickel ions reaches up to 0.3 g/L, the total salt content reaches 200 g/L. The possibility of sorption purification of salt concentrates from nickel on the aminocarboxylic ionite Lewatit TP 207 was studied, and the capacitance characteristics of the ionite were determined. It is shown that a high salt background does not reduce the ion exchange capacity of ionite for nickel. The dynamic capacity of Lewatit TP 207 before the breakthrough was 0.155 g/g. Nickel desorption is carried out with 20% sulfuric acid. The maximum concentration of nickel in the eluates was 25.484 g/L. Such eluates can be returned to the nickel-plating bath. The technology of periodic ion-exchange purification of salt concentrates is proposed.

### 1. Introduction

Galvanic production is an integral part of instrument-making, mechanical and other large enterprises. Electroplating is used to protect metals from corrosion during operation and at the same time to give a good appearance to parts.

One of the urgent problems of galvanic production is generation of the large amount of wastewater. The enterprises construct local treatment facilities to neutralize these waters and reduce the consumption of fresh water. At the neutralization station, water comes from the parts washing baths and spent contaminated electrolytes. They contain heavy non-ferrous metal ions, acids, alkalis, various additives, as well as mineral oils and dyes. It is advisable to create at the water treatment sites such technological chain, in which the water circulation system tends to be closed, and forming waste can be returned to the process.

The existing techniques for wastewater treatment are based on methods provide metals to be converted into insoluble compounds (hydroxides). The resulting solids are disposed of. Non-ferrous metals contained in sediments are lost, and enterprises are forced to pay environmental fines for the dumping of toxic substances.

Typically, galvanic wastewater treatment technology consists of a combination of several methods. The most commonly reagent treatments [1], coagulation-flotation methods [2], electrochemical methods (electroflotation, electrodialysis, electrolysis, etc.) [3–5], membrane technologies (ultrafiltration, nanofiltration, reverse osmosis), sorption processes are used [6].

The aim of the present study is to investigate the sorption of nickel by ion exchange from concentrated salt concentrates, which are waste from the treatment facilities of the galvanic section of the Ural Optical & Mechanical Plant.



In 2014 the treatment facilities have been reconstructed and currently operating according to the following scheme. Galvanic wastewater is directed to the neutralization station via separate collectors. Plastic receiver tanks are installed outside the building. Wash water, concentrated alkaline and acidic effluents, chrome effluents, concentrated alkaline and acidic aluminum-containing effluents are taken in separate tanks, from where they are fed for further processing.

Wash waters are averaged, then mixed with chrome effluents and fed to an electrocoagulator. The preliminary adjustment of the pH value of HCl or NaOH to the value of hydrate formation of chromium was carried out. In the electrocoagulator,  $\text{Cr}^{6+}$  is reduced to  $\text{Cr}^{3+}$ , as well as sorption of heavy metals on the surface of electrolytically obtained precipitates is occurred.

From the electrocoagulator, an aqueous suspension of metal hydroxides is sent to a vertical parallel plate clarifier, from where the thickened suspension enters to the filter press, and the clarified effluents pass sequentially through grain-loaded filters, carbon filters and a coal particle recovery unit.

After pretreatment clarified wash water is fed to the unit of a two-stage reverse osmosis. In the process of reverse osmosis, the stream is divided into two parts: the filtrate (water) and the permeate (stream enriched with salts). Permeate is taken to the evaporation unit, and purified water is used for topping up the electroplating baths.

Concentrated galvanic effluents are fed to the reactor to adjust the pH level (pH=10) and then sent to a filter press. Concentrated aluminum-containing effluents are adjusted separately (pH=5) and fed to a vacuum filter.

The clarified effluents and permeates of the reverse osmosis unit are directed to the evaporation unit. The products of the evaporation unit are distilled water and liquid salt concentrate for disposal.

Salt concentrates contain up to 0.3 g/L of nickel. To purify them from nickel using sorption by ion exchangers is proposed. It is known that resins containing carboxyl groups are capable of selectively absorbing nickel from dilute solutions [7, 8]. In particular, Lewatit TP 207 is used to remove nickel [9, 10].

## 2. Methods and materials

Lewatit TP 207 (Lanxess, Germany) is a weakly acidic, macroporous cation exchange resin with the iminodiacetate functional groups used for the selective sorption of heavy metal cations from aqueous solutions, the total ion exchange capacity of 2.2 meq/mL, the particle size of 0.4–1.25 mm and the thermal stability of up to 80 °C. In the experiments, a solution containing 250 mg/L Ni was used. Density of solution is 1.2 g/mL, pH = 4.40. Salt background presented by  $\text{Na}_2\text{SO}_4$ , NaCl,  $\text{NaNO}_3$ ,  $\text{Na}_3\text{PO}_4$ ,  $\text{Na}_3\text{BO}_4$ ,  $\text{NH}_4\text{F}$ .

### 2.1. Ionite pretreatment

Before to they use ionite was washed with NaOH and HCl (7.5 %) to remove organic and inorganic impurities and then washed several times with deionized water. The resin was finally converted to the  $\text{H}^+$ - form.

### 2.2. Batch experiments

To perform sorption under static conditions, 50 mL of the solution was brought into contact with 0.5 g of resin. The duration of contact of the resin with the solution was 24 hours. The filtrates were separated from the ion exchanger. The nickel concentration was determined by atomic absorption spectrometry.

The sorption capacity  $q_t$  [mg/g] of the appropriate ions was determined from the following formula:

$$q_t = (C_0 - C_t) \times \frac{V}{m},$$

where  $C_0$  is the initial concentration of As (V) ions, [mg/L];  $C_t$  is the concentration of As(V) ions at time  $t$ , [mg/L];  $V$  is the volume of the solution, [L];  $m$  is the mass of the ionite, [g].

### 2.3 Column experiments

In the column experiment, sorption was performed in a fixed-bed glass column with 1.0 cm internal diameter and 25 cm height, packed with 5 g of Lewatit TP 207 in H. The glass wool beads were added to improve the flow distribution.

The sorption duration was 78 hours; 2.59 L of a solution was passed. The filtrates were taken into 50 mL volumetric flasks.

### 2.4. Effect of $\text{NaNO}_3$ concentration on nickel sorption

The main component in salt concentrates is  $\text{NaNO}_3$ . The concentration of sodium nitrate is 194.78 g/L. A series of experiments was performed to study the effect of  $\text{NaNO}_3$  content on nickel sorption. In 50 mL volumetric flasks, a  $\text{NiSO}_4$  model solution with a nickel content of 250 mg/L and a  $\text{NaNO}_3$  solution with a concentration of 0, 10, 20, 50, 100, 200 g/L were added. Contact duration 24 hours.

### 2.5. Desorption

Desorption was carried out with a 20% solution of sulfuric acid. The desorbent was supplied at the same rate as the solution for sorption.

## 3. Results and discussion

The results are shown in the table 1.

**Table 1.** Effect of  $\text{NaNO}_3$  concentration on Lewatit TP207 capacity for nickel.

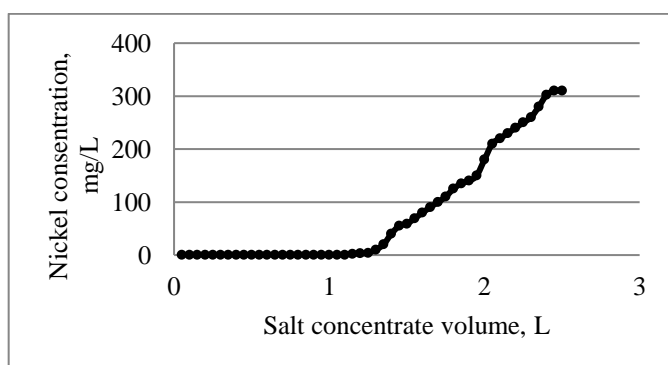
$C(\text{NaNO}_3)$ , g/L	Mass of ionite, g	$C_i(\text{Ni})$ , mg/L	$q_t$ , mg/g
0	0.5	27	22.30
10	0.505	30.2	21.76
20	0.505	26.9	22.09
50	0.505	22	22.57
100	0.5	23.55	22.65
200	0.5	21.88	22.81

From the obtained results it follows that changes in the salt background does not significantly affect the capacity of the resin.

Experimental breakthrough curves for adsorption of Ni(II) on Lewatit TP207 is shown in figure 1. A breakthrough point was observed after passing 1.15 L of concentrate after 34 hours. After passing through 2.5 L of solution, the filtrate concentration was equal to the initial one.

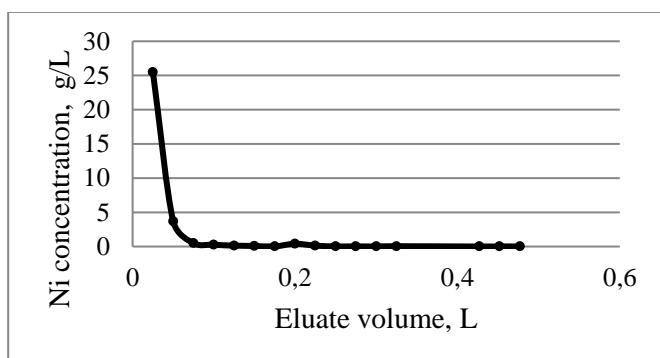
The dynamic capacity of the ion exchanger before the breakthrough point was 0.155 g/g.

After desorption, the ion exchanger was washed with 125 mL of distilled water. The nickel concentration in the wash water is 34.6 mg/L. The concentration of nickel in the eluates was determined by AAS.



**Figure 1.** Sorption breakthrough curve.

The output desorption curve is shown in the figure 2.



**Figure 2.** The output desorption curve.

In the richest eluate fraction the Ni concentration is 25.484 g/L. The concentration of Zn, Fe, Cu in several portions of eluates were analyzed by AAS. The results of the analysis are shown in table 2.

**Table 2.** The content of impurities in the eluate.

Eluate volume, L	Concentration, g/L		
	Zn	Cu	Fe
0.025	1.47	0.56	3.073
0.05	0.23	0.191	0.86
0.075	0.036	no data	0.63
0.1	0.005	no data	0.67
0.125	0.005	no data	0.83
0.15	0.036	no data	0.8
0.175	0.001	no data	0.77
0.2	0.001	0.002	no data

The resulting concentrations of impurities make it possible to return the eluates to the process chain, as an additive to the electrolyte in nickel baths, not exceeding the values established by the technological instruction.

Since salt concentrates have a temperature near 80 °C after the evaporation unit, the effect of temperature on nickel sorption has been studied.

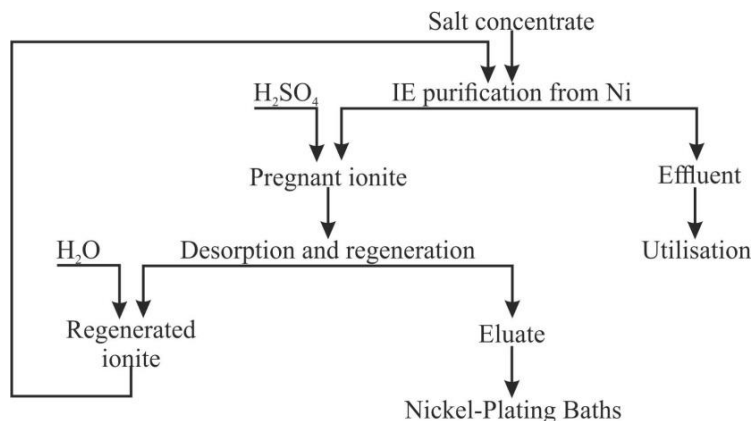
There is practically no information in the literature concern the effect of temperature on the absorption of nickel by resins. There is evidence of the effect of temperature on nickel sorption by activated carbon, bentonite clays, minerals etc. [11, 12]. The obtained regularities cannot be fully applied to synthetic ion exchangers.

To study the sorption process at elevated temperature, the solution was passed through a thermostatic column.

Salt concentrate with pH=4.98;  $\rho=1.334$  g/mL;  $C_{Ni}=189$  mg/L, temperature 60 °C. Duration of sorption was 11 hours, 1.975 L of salt concentrate was passed through column. Solution rate is 0.18 L/hour.

Although the ion exchanger was loaded into the column in a fully swollen state, its volume decreased while passing the heated solution. Breakthrough of nickel ions was observed in the first portion of filtrate. Thus, it is not recommended to supply the salt concentrate directly to the sorption purification without preliminary cooling.

Currently, about 40 m<sup>3</sup> per year of salt concentrate is formed at the enterprise; the average nickel concentration is 0.310 g/L. It is supposed to treat the concentrate according to the scheme shown in figure 3.



**Figure 3.** Flowsheet for the processing of salt concentrates.

Since the volume of generated effluents is small and require cooling, the best option would be to carry out sorption treatment periodically, as salt concentrates accumulate.

#### 4. Conclusion

According to the results of the experiments, the working exchange capacity (before the breakthrough point) over nickel for the Lewatit TP 207 ion exchange resin was 0.155 g/g. Considering that about 40 m<sup>3</sup> of salt concentrate is formed per year, it is proposed to provide for a periodic operation mode of the installation. The degree of desorption, it is advisable to take 90 %. Periodically, complete desorption of the ion exchanger will be necessary.

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