Removal of nickel from electroplating rinse waters using electrostatic shielding electrodialysis/electrodeionization

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ABSTRACT

Electrostatic shielding zones made of electrode graphite powder were used as a new type of ionic and electronic current sinks. Because of the local elimination of the applied electric field, voltage and current within the zones, ions are led inside them and accumulate there. The current sinks were implemented in electrostatic shielding electrodialysis of a simulated nickel plating rinse water containing $100\,mg\,L^{-1}$ nickel and electrodeionization of a 0.001 M $NiSO_4$ solution with simultaneous electrochemical regeneration of the ion exchange resin beds. Pure water was obtained with a Ni^{2+} ion concentration of less than $0.1\,mg\,L^{-1}$ at a flow rate of $2.02 \times 10^{-4}\,dm^3\,s^{-1}$ diluate stream and a current density of $30\,A\,m^{-2}$.

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1. Introduction

Nickel is a toxic heavy metal and nickel compounds, for instance nickel sulphide, are suspected to cause cancer [1]. Waste streams from nickel electroplating industries, textile industries or washing effluents for remediation of soil contaminated with nickel may contain up to $1000\,mg\,L^{-1}$ nickel, which, according to environmental regulations worldwide must be controlled to an acceptable level before being discharged to the environment. Only 30–40% of all metals used in plating processes are effectively utilized i.e. plated on the articles while the rest contaminates the rinse waters during the plating process when the plated objects are rinsed upon removal from the plating bath.

Nickel removal is known to be difficult and consequently a scientific challenge. Several treatment processes have been suggested for the removal of nickel from aqueous waste streams, such as adsorption on activated carbon [2], or on red mud [3], ion exchange on zeolites [4], ion exchange on chelating resins [5,6], microfiltration [7] and chemical precipitation [8–10].

Although chemical precipitation is the most economic and the most commonly utilized procedure for the treatment of heavy metal-bearing industrial effluents, it can become ineffective in the presence of strong complexing agents. In addition, the high buffer capacity provided by complexing agents requires excessive amounts of chemicals to neutralize alkalinity. The precipitated sludge containing the concentrated $Ni(OH)_2$ is an extremely haz-

ardous waste and must be disposed of using special facilities at great expense to industry.

From the viewpoint of environmental protection and resource saving, effective recycling and reusing of the metal wastewater is strongly expected. Closed-loop system or so-called effluent-free technology should be developed.

Electrochemical methods such as cathodic reduction and electrowinning [11–14], electrodialysis [15,16] and electrodeionization [17–21] have been recently developed to contribute to the solution of this serious environmental problem of toxic heavy metal removal.

Electrodeionization is the removal of ions and ionizable species from water or organic liquids. It uses electrically active media and an electrical potential to cause ion transport and may be operated batch wise, or continuously.

Continuous processes such as electrodialysis, and filled cell electrodialysis or otherwise called continuous electrodeionization comprise alternating permselective cation exchange membranes and anion exchange membranes, which under the influence of the electric field allow only cations or only anions respectively to permeate their mass and simultaneously retain coions so that diluate and concentrate compartments are created and deionization occurs. Membrane electrodeionization processes exhibit the known limitations associated with membranes, such as membrane fouling, scaling and concentration polarization. Furthermore, these processes cannot avoid the precipitation of bivalent metal hydroxide occurring at the resins interface and the anion exchange membranes [13–17]. For this reason the application of electrodeionization has been limited in wastewater treatment. Improvements with new configuration of the electrodeionization

membrane stack to overcome the undesirable hydroxide precipitation are reported in Refs. [18,19].

Batch processes such as capacitive deionization [22] or membrane capacitive deionization [23] are collection/discharge processes which rely on the formation of double-layer supercapacitor at the solution/electrode interface and need electrodes with very large specific areas, such as nano-structured activated carbon aerogels.

In our previous works [24–27] we have shown that electrostatic shielding electrodialysis/electrodeionization can be realized by means of electrostatic shielding zones-ionic current sinks (ESZs-ICSs) instead of permselective ion exchange membranes. The ESZs-ICSs are formed by electronically and ionically conducting media e.g. graphite powder packed beds interposed between the anode and cathode which cause electric field discontinuity inside the electrolytic setup resulting in ion diluting and ion concentrating compartments.

The present paper offers a new alternative way of a membraneless process of electrodialysis/electrodeionization for removal of Ni^{2+} ions from water solutions or industrial effluents such as nickel electroplating waste waters.

The proposed new process differs from classical electrodialysis–continuous electrodeionization processes in that it does not use any permselective ion exchange membranes and therefore it does not exhibit the above mentioned membrane associated limitations. It also differs from classical batch wise operated capacitive deionization in that it is a continuous process i.e. diluate and concentrate are received from separate and unchanged compartments without any removal of diluate and concentrate or any down time for electrode saturation, regeneration and rinsing steps.

2. Materials and methods

2.1. Electrodes

Platinized titanium grids were used as end-electrodes in all experiments.

The intermediate electrodes ICSs must be electronically and ionically conducting. They are packed beds of graphite powder (Merck, particle size <50 μm, electrical conductivity $2 \times 10^4 \, S \, m^{-2}$) or electrode graphite powder (Fig. 1) which is used as anode by the electrolytic production of aluminium (Aluminium of Greece, particle size <1 mm, electrical conductivity $3.3 \times 10^4 \, S \, m^{-2}$). Anode graphite is preferable because of its better electrical and electrocatalytic properties.

2.2. Separators

The cell compartments are separated from each other by ion conducting porous organic polypropylene separators S (Celgard 3407) which prevent intermixing of solutions without obstructing the migration of ions in the electric field.

2.3. Cells

The self-made electrodialysis cell illustrated in Fig. 1 contains four ICSs serving as ion traps and ion concentrating compartments, three diluate compartments between them and two electrode compartments, all placed in parallel. Each compartment is 8 cm long and 7 cm wide. The ICSs have a thickness of 5 mm and an effective area of 50 cm² each. The area of each ICS is equal to the vertical cross-section area of the electrolytic cell. All compartments are separated from each other by the separators S. The distance between two successive ICSs and also between the ICS and the adjacent end-electrode is 10 mm and is determined by the ion conducting separators and spacers.

The separators are sealed so that the different compartments do not communicate hydraulically with each other but only electrically (ionically) through the electric field. Each ICS has a bipolar function and belongs simultaneously to two adjacent diluate compartments as in bipolar electrochemical double-layer supercapacitors.

The separators can be omitted in case when porous inorganic or organic conductive sheets such as conductive ceramics, porous carbon paper or aerogel sheets, or other porous composite electrodes are used which can function both, as intermediate electrodes and separators as well.

The three diluate compartments are filled with the feed solution. The electrode compartments are filled with a 0.05 M H_2SO_4 solution, while the four concentrate compartments ICSs are filled with electrode graphite powder, are only intermittently rinsed (once an hour) with the feed solution and are steadily let to drain. This pressure difference caused by the different water levels between the diluate compartments and the concentrate compartments ICSs prevents diffusional leakage of ions from the more concentrated ICS-solution into the adjacent diluted ones. Deionization in the diluate compartments can be realized with the water inside the ICSs at the lowest level. If the feed solution in the diluate compartments soaks in a porous material, no pressure difference between the diluate and concentrate compartment exists. Deionization in the diluate compartment is realized without any pressure so long as the concentrate solution level is kept lowest by simply letting it to drain from the porous water permeable bottom of the ICS. Furthermore, the porous ionically and electronically conductive media of which the ICSs are made must cover uniformly all their space without allowing any ions to escape from the diluate compartments to the electrode compartments or in the opposite direction without passing through the ICS mass.

The graphite powder was activated by treating it in a 1 M NaOH solution at 90 °C for 3 h. To complete physical absorption of $NiSO_4$ in graphite, the ICSs were rinsed with a 0.1 M $NiSO_4$ solution until saturation and then with deionized water.

Graphite powder used as ICS material is chemically and thermally very stable but electrochemically quite stable, because it starts to corrode when its relatively narrow potential window or the electrochemical decomposition potential of water is exceeded. The exceeding of these potentials can be prevented by using thinner ICSs which do not exhibit electron transfer reactions between their two polarized sides. Such thinner ICSs are more stable and can operate at increased current densities [25,26]. Certainly, platinized or ruthenized graphite and activated carbon powder, platin spongue or other dimensionally stable electrodes are stable and suitable even at higher fields but are more expensive.

Fig. 2 shows the self-made electrodeionization cell consisted of seven separate, in parallel placed compartments: anode compartment, left ICS, cation exchange resin (CR)-loaded compartment, central concentrate compartment (ICS), anion exchange resin (AR)-loaded compartment, right ICS and cathode compartment. The compartments are separated from each other by the six ion conducting porous polypropylene separators S (Celgard 3407) and are pressed together to form an assembly by using spacers and screws. Each compartment is 8 cm long and 7 cm wide. The ICSs have a thickness of 5 mm and an effective area of 50 cm². The area of each ICS is equal to the vertical cross-section area of the electrolytic cell. The rest four compartments are 10 mm thick each.

The CR and AR resins were pretreated and immersed in a 0.1 M $NiSO_4$ and 0.1 M Na_2SO_4 solutions respectively for 3 days, then packed into the corresponding CR-loaded and AR-loaded compartment and rinsed with deionized water. The anode and the cathode compartments were filled with 0.05 M H_2SO_4 + 0.05 M Na_2SO_4 and 0.05 M NaOH + 0.05 M Na_2SO_4 solutions respectively.
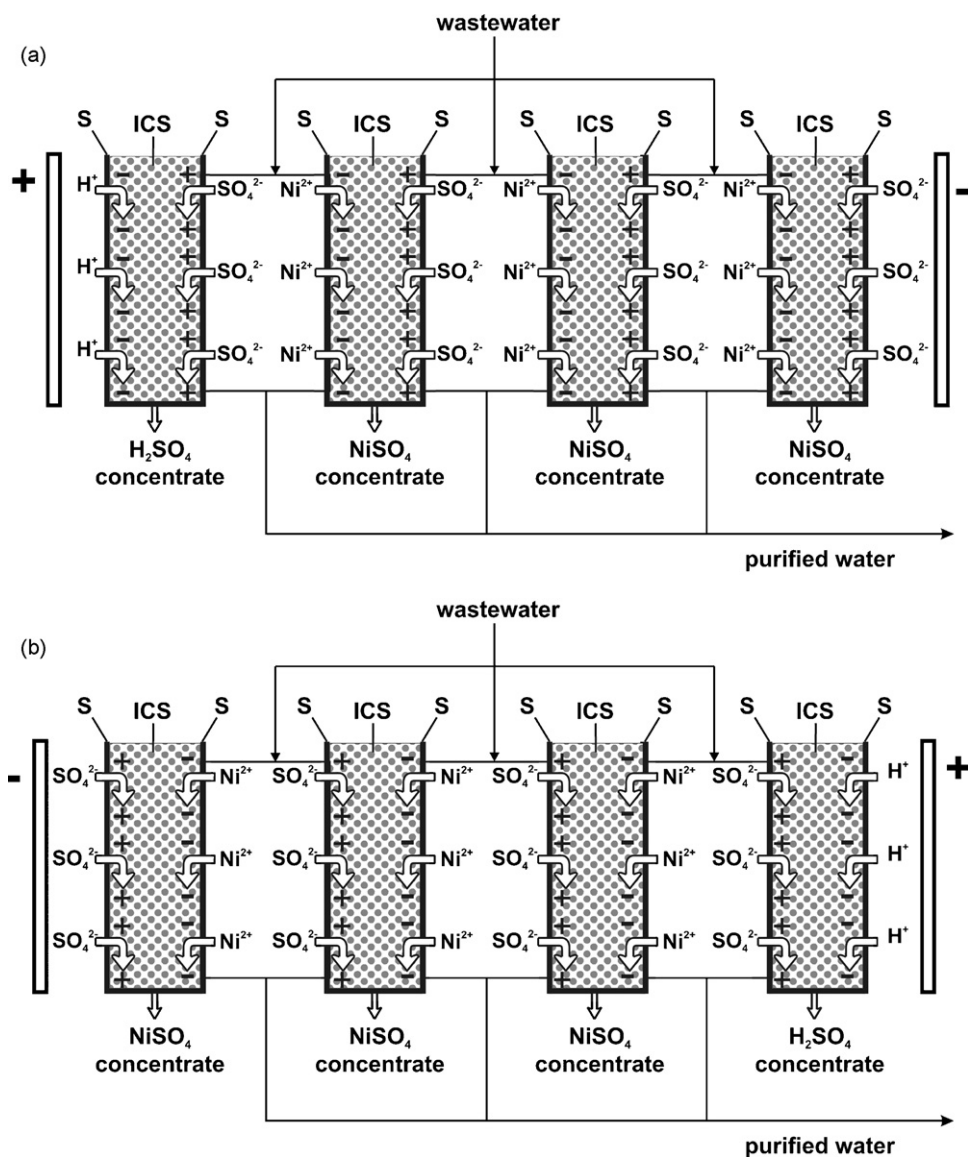
Fig. 1. (a) Schematic diagram of an electrostatic shielding electrodialysis cell for removal of nickel from nickel-bearing wastewaters. Ni^{2+} or H^+ cations and SO_4^{2-} anions accumulate inside the electrostatically shielded ICSs and (b) the same cell by polarity reversal without any negative impact on the electrodialysis process. Only the direction of the entering ions is reversed.
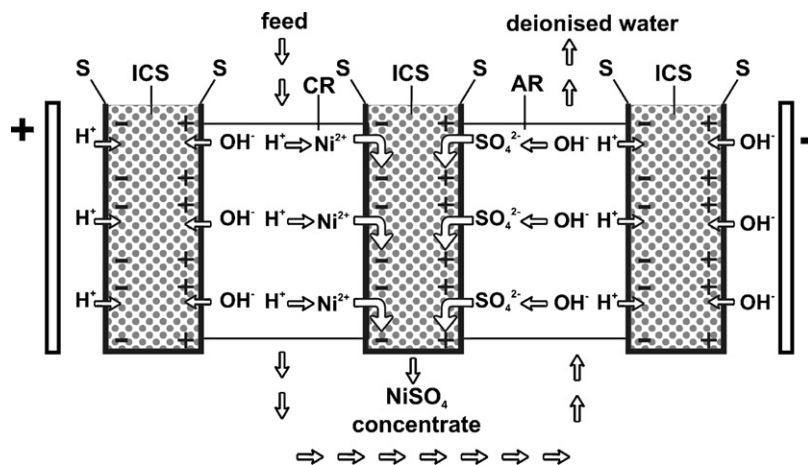

Fig. 2. Schematic diagram of an electrostatic shielding electrodeionization cell for removal of Ni^{2+} ions with simultaneous regeneration of the ion exchange resin beds. The cell contains three ICSs, one CR-loaded and one AR-loaded diluate compartments. Ni^{2+} cations and SO_4^{2-} anions accumulate inside the central ICS and form a $NiSO_4$ concentrate which is received from its bottom. Water to be deionized permeates first the CR and then the AR resin bed.

2.4. Apparatus

Atomic Absorption Spectroscopy (AAS) (Perkin Elmer 5100) was used to determine the Ni^{2+} ion concentrations in water. A power supply (PHYWE) was used to maintain constant DC voltage or constant DC current. Voltage and current were measured by a multimeter (PHYWE). The temperature was held at 298 ± 3 K during all experiments.

2.5. Chemicals

$NiSO_4$, H_2SO_4 and Na_2SO_4 were of analytical grade (Merck). The ion exchange resins used were strongly cation exchanger H^+-form (Amberlite IR-120, Merck,) with an ion exchange capacity of 1.7 mmol mL^{-1} and strongly basic anion exchanger OH^--form (Merck) with an ion exchange capacity of 1.45 mmol ml^{-1}.

3. Results and discussion

3.1. Electrostatically shielded ion concentrating compartments—ESZs, ICSs

Current sinks and sources are local currents from a location where they can be detected into a location they cannot be detected (current sink) or vice versa (current source). Current sinks and sources have particular relevance in current across biological membranes (neurobiology) and have proved to be valuable in the study of brain function [28]. Furthermore, current sinks are used in several electronic applications [29].

However, all known current sinks are related to electronic current sinks (ECSs). Our search throughout literature has not resulted in any other paper except for our previous works [24–27] dealing with ICSs. We created ICSs through electrostatic shielding by employing electronically and ionically conducting media e.g. graphite powder and used them as ion concentrating compartments in form of ESZs.

It is known from electric field theory and Faraday cage [30] and our previous works [25–27] that, when a conductor is placed inside an electric field, an opposite field is formed so that the original electric field in the interior of the conductor is cancelled. The field intensity inside the conductor is zero and its whole space is electrostatically shielded independent on the external field intensity. Therefore, an abrupt potential jump is formed between the inside and the outside of the conductor. Since in an electrolytic or an electrodialysis cell ion migration is caused by the applied electric field, it will stop within an electronically and ionically conducting ICS of zero field, interposed between the anode and cathode.

The ESZs become ion concentrating compartments while the adjacent compartments become ion diluting compartments. The ESZ acts here as a "sink" for ions and ionic currents (ICS). The ionic current (real direction) is eliminated at the cathodically polarized side of the ICS (current sink) and appears again at its anodically polarized side (current source). We first introduced in literature the terms "ion sink" and "ionic current sink–source" in our previous works [26,27].

Ni^{2+} cations and SO_4^{2-} or eventually Cl^- anions can move inside the ICS but only by diffusion and convection and not in field direction. The concentrate compartment ICS is never saturated with the accumulated ions. Because of the electric field its two short-circuited sides become polarized and saturated with oppositely charged ions but at the same time they are discharged and release the same ions because of shorting [25,26].

The thickness of the ICS plays a significant role for purification, regarding the electron transfer redox reactions between the two polarized sides of each ICS inside the electric field. The thicker the

ICS the higher the potential difference between its two polarized sides inside a given electric field. If this potential difference exceeds the electrochemical decomposition potential of water or other solutes, electron transfer redox reactions occur between the two polarized sides of the ICS. This phenomenon does not contribute to deionization but to aimless electrical energy consumption and should be avoided by reducing the ICS thickness. Also the current density can be considerably increased by using thinner ICSs. In this paper these phenomena are only briefly stated. They have been explicitly explained in our previous works [25–27].

The ICS (electronic conductor) does not need practically any potential to carry the current through its mass. The relatively to electrolytic solutions high electronically conducting ICS not only does not bring about any additional potential drop in the cell but, on the contrary, it helps the current flowing the cell to increase by lowering the total resistance of the cell. The ICS consumes no electrical energy ($E = U$; $I = I^2$; $R = 0$, since $U_{ICS} = 0$ or $R_{ICS} = 0$). In multi-cell stacks where a series of concentrate compartments (ICSs) and diluate compartments alternate, the unit cell comprises one diluate compartment and one ICS. The electrical energy is consumed for electromigration of ions only in the diluate compartment which is energetically very favorable [25,26].

Unlike the sensitive and troublesome ion exchange membranes, the thermally and chemically stable graphite powder beds used as ESZs-ICSs allow execution of electrodeionization also under harsh thermal or chemical conditions such as elevated temperatures, strong acidic or strong alkaline solutions and organic solvents.

Up to now, no other paper apart from our previous works, appeared in literature dealing with such ionic and electronic sinks. We can take advantage of these useful new findings and drive ions inside the ICSs, create ion concentrating and ion depleting compartments and perform in this way membrane-less electrodialysis and electrodeionization of water and industrial effluents.

3.2. Electrostatic shielding electrodialysis of nickel plating rinse waters

Watts nickel plating baths contain $NiSO_4$, $NiCl_2$ and H_3BO_3. We have prepared a simulated Watts nickel plating rinse water containing 210.4 mg L^{-1} $NiSO_4$, 83 mg L^{-1} $NiCl_2$ (100 mg L^{-1} Ni) and 59.3 mg L^{-1} H_3BO_3 which we placed in the three diluate compartments of the cell illustrated in Fig. 1 for electrodialytic treatment. The solution pH was approximately 4. The proposed new electrostatic shielding electrodialysis cell with ICSs instead of ion exchange membranes has proved to be efficient also for the removal of heavy metal ions such as Ni^{2+}.

Fig. 3 depicts the concentration variations and percent removal of Ni^{2+} ions versus time in the three diluting compartments. The process was conducted batch wise under constant voltage and decreasing current at a current density of 10–20 A m^{-2}. Ni^{2+} cations in the diluate compartments permeate the ICSs and accumulate there. At higher fields where the electrochemical decomposition potential of H_2O, $NiSO_4$ or $NiCl_2$ can be exceeded, Ni^{2+} ions can also partially be electronated and electrodeposited to pure metal at the cathodically polarized sides of the ICSs through electron transfer reactions [25,26]. Both phenomena contribute to nickel removal in the diluate compartments. The Ni^{2+} ion concentrate received from the bottom of the ICSs may either be recycled to the cathodic compartment to be electrodeposited as pure metal through electrowinning or be given back to the electrolytic nickel bath for reuse. The produced pure water can be reused as rinsing water. In this way, the process can save a lot of water resource and realize "zero pollution". The removal of Ni^{2+} ions is more effective when the electrodialysis cell is assisted by ion exchange resins as will be explained in Section 3.3.
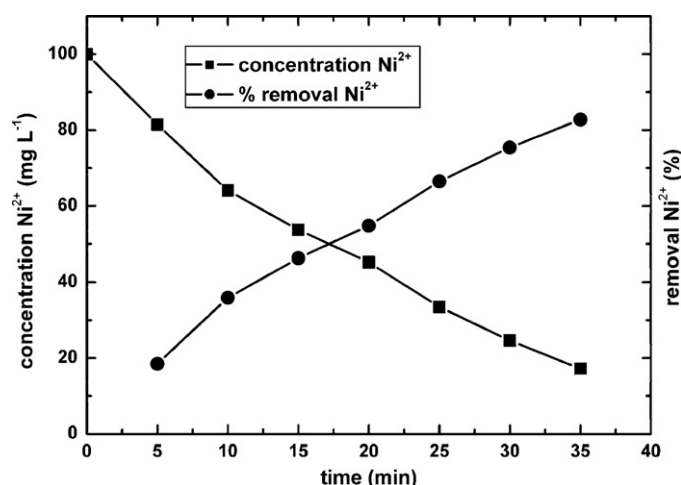
Fig. 3. Concentration variation and % removal of Ni²⁺ ions with time in the diluate compartments of Fig. 1 during the electrostatic shielding electrodialysis of nickel plating rinse waters.

Fig. 4. Concentration variation with time of Ni²⁺ ions (mg L⁻¹) in the central ICS of Fig. 2 during the electrostatic shielding electrodeionization of a 0.001 M NiSO₄ solution.

3.3. Electrostatic shielding electrodeionization of a 0.001 M NiSO₄ solution

High purity deionized water can be produced with the filled cell electrodeionization device (Fig. 2). Feed water must permeate first the CR and then the AR resin to be deionized.

A synthetic 0.001 M NiSO₄ solution was used and pure water with a Ni²⁺ ion concentration of less than 0.1 mg L⁻¹ was obtained in both modes, batch or continuous, while the resins were simultaneously regenerated without the use of any chemical regenerants. The product water quality stays constant over time, whereas in regenerable ion exchange with chemicals it degrades as the resins approach exhaustion.

H⁺ and OH⁻ ions are formed at the anode and cathode through the water splitting reactions:

$$H_2O \rightarrow 2H^+ + 1/2O_2 + 2e^- (anode)$$

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 (cathode)$$
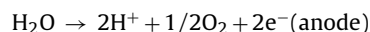
The generated H⁺ ions in the anode compartment and the anodically polarized side of the left ICS repel and replace the Ni²⁺ cations from the CR resin and the generated OH⁻ ions in the cathode and the cathodically polarized side of the right ICS repel and replace the SO₄²⁻ anions from the AR resin. The replaced cations and anions together with the electroosmotically transported water molecules are transferred to the central concentrate compartment (ICS) where, due to electrostatic shielding, the electromigration of ions is stopped and they accumulate there. The accumulated NiSO₄ is rinsed with 50 ml deionized water, is let to drain from the bottom of the central ICS and is collected in a container placed underneath. The rinsing of the central ICS occurs once an hour with the same initial volume of 50 ml deionized water which was collected and reused and its Ni²⁺ concentration increase with time is plotted in Fig. 4. The electrodeionization process was conducted in a continuous mode under constant current and increasing voltage at a current density of 30 A m⁻². The obtained experimental results concerning the Ni²⁺ ion concentration (mg L⁻¹) and pH variation inside the central ICS are depicted in Figs. 4 and 5 respectively.

The right and left ICSs do not at all contribute to electrodeionization but are essential. It was found that electrostatic shielding and therefore the capture of the incoming ions inside the central ICS is not so effective if the two outer ICSs are missing. Perhaps the accumulated ions inside the sole ICS are stronger attracted by the adjacent anode and cathode, so they are not
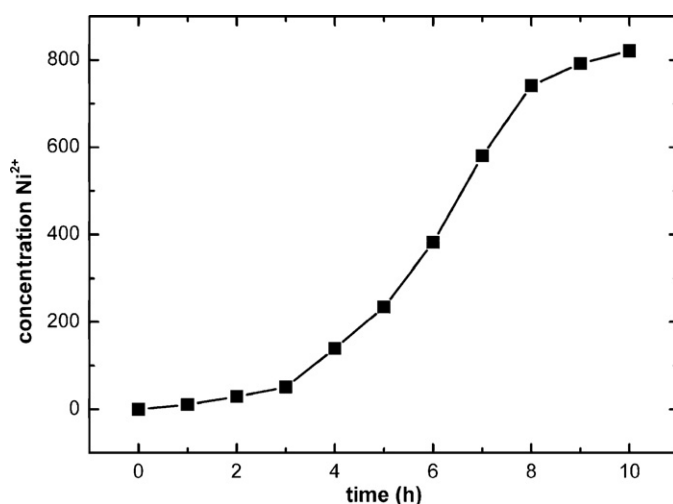
sufficiently held inside the ICS and migrate to the adjacent electrode.

Precipitation of insoluble hydroxides (scaling) such as Ni(OH)₂ inside the central ICS was not observed because this ICS compartment becomes gradually acidic. Part of the generated H⁺ ions at the anodically polarized side of the left ICS migrate through the CR resin bed into the central concentrate compartment (ICS). Similarly, part of the OH⁻ ions generated at the cathodically polarized side of the right ICS migrate through the AR resin bed and result in the same central concentrate compartment. It is well known that the migration rate of the H⁺ ions is greater than that of the OH⁻ ions. Therefore, pH inside the central ICS drops with time (Fig. 5). pH was measured and found acidic also in the anodic compartment, the left ICS and the CR-loaded compartment. pH is alkaline inside the cathodic compartment, the right ICS and the AR-loaded compartment, but no Ni²⁺ ions exist there to combine with the OH⁻ ions and form the insoluble Ni(OH)₂ precipitate. Similar observations have been reported in literature by Chen and Wu [18] or Feng et al. [19].

The electrodialysis and electrodeionization processes were evaluated in terms of percent removal (pr), current efficiency (ce) and enrichment factor (ef), which were calculated from the Eqs. (1)–(3)
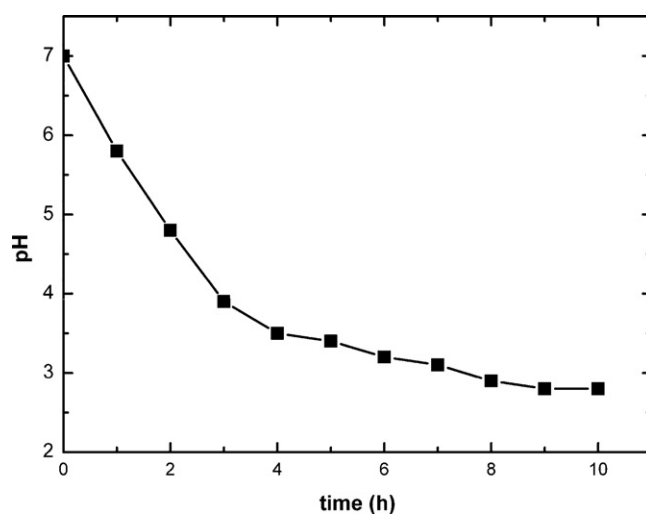

Fig. 5. pH variation with time in the central ICS of Fig. 2 during the electrostatic shielding electrodeionization of a 0.001 M NiSO₄ solution.

respectively:

$$pr = \frac{C_o - C}{C_o} \times 100 \tag{1}$$

$$ce = z \times F \times Q \times \frac{C_o - C}{I} \tag{2}$$

$$ef = \frac{C_c}{C_o} \tag{3}$$

where C_o and C are the inlet and outlet concentrations of Ni^{2+} ions in the diluate compartments and C_c in the concentrate compartment respectively (mol L^{-1}), z is the ion charge, F is the Faraday constant (96,453 A s), Q is the flow rate (dm^3 s^{-1}), and I is the current (A).

In the continuous mode of electrodeionization with a flow rate of 2.02×10^{-4} dm^3 s^{-1} diluate stream and a current density of 30 A m^{-2} the current efficiency for Ni^{2+} ion removal is 26%. Ni^{2+} ions in concentrate could be enriched by a factor of 14. At higher current densities the removal of Ni^{2+} ions and therefore the current efficiency can be further increased. However, this additional increase is attributed to electrodeposition of the pure metal at the cathodically polarized side of the central ICS, due to exceeding the electrochemical decomposition potential of NiSO$_4$ and electron transfer reactions between the two polarized sides of the ICS.

4. Conclusions

Based on the experiments conducted in this study, the following conclusions can be drawn:

(1) Electrostatically shielded zones-ionic current sinks made of graphite powder eliminate the applied electric field inside their mass and can therefore serve as ion traps and ion concentrating compartments in membrane-less electrodialysis and electrodeionization applications.
(2) The current density can be enhanced by using thin electrostatic shielding zones-ionic current sinks and therefore avoiding the electron transfer reactions.
(3) The proposed new electrodialysis/electrodeionization cells do not exhibit the known membrane associated limitations such as membrane fouling, scaling, concentration polarization. Furthermore, they can operate also under harsh chemical or thermal conditions.
(4) A membrane-less electrostatic shielding electrodialysis cell was implemented in a batch wise purification of a simulated nickel plating rinse water containing 100 mg L^{-1} nickel under constant voltage and decreasing current at a current density of 10–20 A m^{-2}. The concentration of nickel decreased with time and fell to 17 mg L^{-1} in 35 min. Furthermore, a new membrane-less electrostatic shielding electrodeionization cell was used for a continuous purification of a 0.001 M NiSO$_4$ solution producing pure water with a nickel concentration under 0.1 mg L^{-1} at a flow rate of 2.02×10^{-4} dm^3 s^{-1} diluate stream and a current density of 30 A m^{-2}.
(5) Research is needed to gain more information about the implementation possibilities of the proposed ionic current sinks and their dynamics to contribute to the solution of environmental or other technological tasks.

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