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Original scientific paper

Study of the electroreduction process of indium ions from aqueous electrolyte

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Abstract

The work focuses on the mechanism of the electroreduction of indium ions in an aqueous electrolyte containing hydrochloric acid. The potential region for the electroreduction of indium ions on platinum (-0.3 to -1.5 V), nickel (-0.8 to -1.5 V), and Pt/In (-0.63 to -1.5 V) electrodes was determined by recording potentiodynamic polarization curves. The influence of several factors, including temperature, indium ion concentration, scan rate, and disk electrode rotation speed, on the electroreduction of indium was investigated. It was established that increasing both the temperature and the indium ion concentration in the electrolyte accelerates the reduction process. The process is primarily controlled by the diffusion of indium ions to the cathode surface. Furthermore, polarization curves were obtained using a rotating platinum disk electrode. The dependence of the current density on the electrode rotation speed at different potentials showed that the electroreduction process of indium ions in an aqueous electrolyte is accompanied by diffusion polarization.

Keywords

Electrochemical reduction; indium ions; electrodeposition; rotating disk electrode; diffusion kinetics

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Introduction

Indium is a strategically important metal that has attracted considerable attention in the semiconductor industry [1]. Owing to its unique physical and chemical characteristics, it has found extensive applications across various high-technology fields. Indium is a crystalline, highly malleable, and very soft metal, notable for retaining its plasticity even at cryogenic temperatures [2]. Indium and its compounds exhibit semiconducting or optoelectronic properties that are used in the production of liquid crystal displays, semiconductors, low-pressure sodium lamps, and infrared photodetectors [3]. More than half of the world's indium consumption is accounted for by indium tin oxide (ITO) coatings, which are excellent, transparent, and conductive materials widely used in the manufacture of thin-film transistor liquid-crystal displays for television screens, laptop screens, and cell phone displays.

The electrodeposition and electroreduction of indium have been extensively investigated using various electrolytes and electrode materials [4-10].

The electroreduction of indium on a high-purity indium electrode (99.98 %) in perchlorate–chloride electrolytes was investigated using linear and cyclic voltammetry, electrochemical impedance spectroscopy, and chronoamperometry. Electroreduction of indium is diffusion-limited, and the reaction rate constant at an indium salt concentration of 0.1 M is 1.3 μm s^{-1}. The apparent rate constants of the charge transfer stage found by linear and cyclic voltammetry and impedance spectroscopy are 23.7, 36.2 and 30.6 μm s^{-1}, respectively. The diffusion coefficients of indium(III) ions, calculated from the Cottrell equation using chronoamperometric measurements and from the Warburg impedance based on impedance spectroscopy, are in good agreement. It has been established that a Gerischer impedance is observed, indicating a homogeneous reaction involving indium chloride complex formation and pointing to a chemical-electrochemical reaction mechanism [11-13].

The authors of [14] investigated the mechanism of indium electrodeposition on gold electrodes from acidic KBr solutions using a rotating ring–disk electrode technique. The measurements showed that the total electrode reaction $\mathrm{In(III)} + 3e^- \rightarrow \mathrm{In}$ consists of two consecutive charge transfer stages, *i.e.* $\mathrm{In(III)} + 2e^- \rightarrow \mathrm{In(I)}$ and $\mathrm{In(I)} + e^- \rightarrow \mathrm{In}$, with soluble monovalent indium as an intermediate product. The current efficiency of In(I) generation under steady-state conditions is 100 % at low disk current densities. Disproportionation of In(I) in the bulk solution occurs in lightly acidified solutions, pH 2, in addition to the electrochemical reduction of In(I). Based on the transient processes of the ring current, it has been established that the formation of a thin passive film of metallic indium precedes the stage of transfer of two electrons.

According to the studies of the authors [15], the dependence of the indium electrode potential on the logarithm of the indium(III) concentration is linear, and the determined slopes are comparable with the slopes calculated using the Nernst equation, assuming $n = 3$. The potential of In(III)/In) was determined as 0.805±0.003 V *vs.* Pt/$PtCl_2$ (1 M). At 693 K, the free energy of formation of indium(III) chloride in 1 M solution is -325,398 ± 500 J mol^{-1}. A single chronopotentiometric wave is observed for the reduction of $InCl_3$ at -0.81 V *vs.* Pt(II)/Pt reference electrode. The diffusion coefficient was calculated as 1.81×10^{-5} cm^2 s^{-1} based on the analytical data for $n = 3$. Chronopotentiometry showed a single-step reduction of In_2O_3. It was found that the quarter-wave potential is -0.7 V relative to the Pt(II)/Pt reference electrode. It was established that the reduction is not diffusion-controlled over the time interval studied [15].

In [15], *in-situ* scanning tunneling microscopy (STM) was used to study the electrodeposition of indium (In) on a copper (Cu) thin film electrode in 0.1 M K_2SO_4 + 1 mM H_2SO_4 + 1 mM $In_2(SO_4)_3$ (pH 3) electrolyte solution with and without 1 mM choline chloride. The Cu thin film pre-deposited on platinum (111) consisted of oriented Cu(111) layers supported on a Pt(111) substrate. This highly ordered Cu(111)-like substrate provided a detailed characterization of underpotential and overpotential deposition (UPD and OPD) at positive and negative potentials of -0.60 V (*vs*. Ag/AgCl), respectively. The electrodeposition of indium from 0.05 mol L^{-1} $InCl_3$ dissolved in a mixture of choline chloride ($ChCl_3$) and ethylene glycol (EG) in a molar ratio of 1:2 was controlled by diffusion, and the activation energy of In^{3+} was found to be 32.50 kJ mol^{-1}. The analysis of current *vs.* time curves based on the Scharifker-Hills model revealed that deposition occurred through progressive nucleation and three-dimensional growth. The morphology of the electrodeposits obtained at temperatures ranging from 25 to 65 °C was characterized by randomly distributed grains and rods on the micrometer scale [16,17].

From previous studies, however, it became clear that the kinetics and mechanism of the electrochemical reduction of indium ions are studied differently. Based on the literature analysis, we conclude that, unlike at an indium electrode, the electrochemical reduction of indium ions proceeds differently on various electrode materials, as does the mechanism of indium deposition. On the indium electrode, the electrochemical reduction occurs in a single step, in which In(III) is directly converted to In(0). Therefore, to clarify this, the main goal of our work is to identify the potential region and to study the kinetics and mechanism of the electrochemical reduction of indium ions in the selected electrolyte for future co-deposition of In-Sb thin films.

Experimental

Cyclic and linear polarization curves were recorded using an IVIUMSTAT Electrochemical Interface potentiostat to determine the potential region and investigate the mechanism of the electroreduction of indium ions. A three-electrode glass electrochemical cell with a volume of 100 ml was used. Pt wire with an area of 0.35 cm^2, Ni plate with an area of 2 cm^2, Pt/In wire with an area of 0.35 cm^2 and a platinum plate with an area of 4 cm^2 were used as the working electrodes. For preparing a Pt/In electrode via the potentiostatic method, a layer of In is first deposited on the Pt wire. A fully saturated silver chloride electrode served as the reference electrode.

Kinetic studies were performed using a rotating disk electrode (RDE-710, Gamry Instruments, USA). A three-electrode glass electrochemical cell with a volume of 100 mL was employed. The platinum electrode (surface area: 0.2 cm^2) served as the working electrode, while a carbon electrode and a silver/silver chloride electrode were used as the auxiliary and reference electrodes, respectively.

The electrolyte was prepared using indium(III) oxide (chemically pure, CAS: 1312-43-2, Sigma-Aldrich, Germany). Indium oxide was first dissolved in 5 to 10 mL of concentrated HCl (37 %, pH 2.8) at 50 °C. Subsequently, 50 mL of bi-distilled water was added, and the solution was evaporated to dryness. The residue was then redissolved in 50 mL of bi-distilled water with continuous stirring. The resulting electrolyte contained indium chloride in the concentration range of 0.06 to 0.28 M.

Prior to each experiment, the platinum electrode surface was cleaned electrochemically. The electrode was immersed in pure H_2SO_4 and polarized at 1.2 V for several minutes to oxidize and desorb organic impurities. It was then held at 0.0 V to reduce adsorbed oxygen, followed by polarization at 0.3 V to remove residual adsorbed hydrogen. This procedure ensured complete cleaning of the Pt electrode surface [18].

Before the experiments, the Ni electrodes were mechanically ground, treated with dilute HNO_3 for 30 seconds to remove oxide layers, immersed in alcohol or acetone, and finally washed with bi-distilled water. Electrochemical polishing of Ni electrodes was carried out in a solution consisting of 55 ml H_2SO_4, 55 ml H_3PO_4, 50 ml H_2O (T = 293 to 303 K, j = 0.5 A cm^2, τ = 180 s), and then washed with bi-distilled water [19].

The electrolyte temperature during electrolysis was maintained at a stable level using a UTU-4 thermostat.

Results and discussion

The kinetics and mechanism of the electrochemical reduction of indium ions in aqueous electrolyte were studied using the potentiodynamic method. For this purpose, polarization curves were recorded on various substrates, including Pt, Ni and Pt/In electrodes.

Figure 1 shows the cathode polarization curve of the electrochemical reduction of indium ions from an aqueous electrolyte on Pt substrates. Indium in the electrolyte is in the form of In^{3+}.

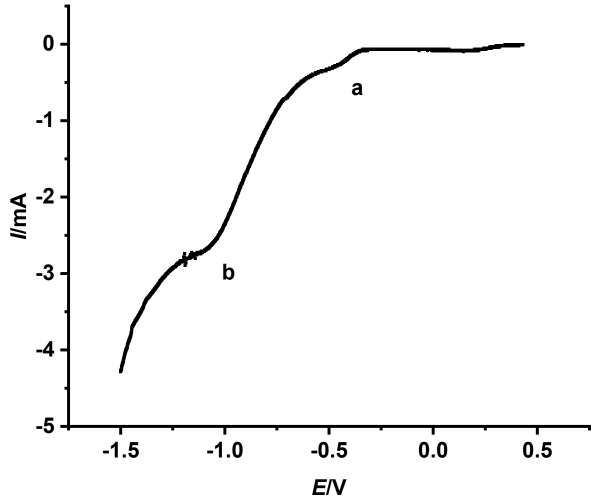

Figure 1. *Cathodic polarization curve of electroreduction of indium ions from aqueous electrolyte on Pt electrode. Electrolyte: 0.2 mol* L^{-1} $InCl_3$*, pH 2.8,* T *= 298 K, scan rate = 20 mV* s^{-1}

As is seen from Figure 1, starting from a potential of -0.37 V with a potential interval of -0.37 to -0.6 V (peak a), indium ions are reduced according to the following reaction: $In^{3+} + 2e^{-} = In^{+}$.

After -0.6 V (peak b in Fig. 1), the process accelerates due to the increase in current, which promotes the deposition of indium nucleus on the electrode surface. Then, the current spent on the process stabilizes due to the passivation of the electrode surface (here, the electrode surface is completely covered with an indium film). There is also an increase in current with increasing potential, starting at -1.15 V. At -1.1 V, hydrogen evolution is also observed.

Comparison of Figures 1 and 2 shows that the background electrolyte typically does not participate in the main electrochemical reaction. In Figure 1, hydrogen evolution occurs after -1.1 V. This is because the electroreduction activity of indium is higher than that of hydrogen evolution. In the absence of indium ions, hydrogen evolution occurs after -0.26 V (Figure 2).

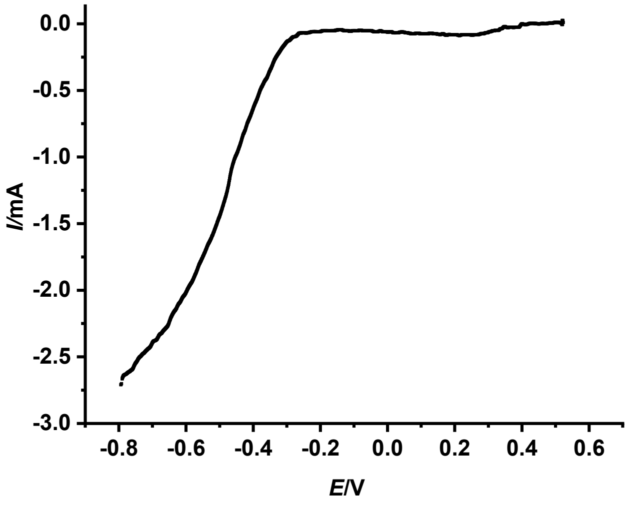

Figure 2. *Cathodic polarization curve of background electrolyte on Pt electrode. Electrolyte: 1.6 mM HCl, pH 2.8,* T *= 298 K, scan rate = 20 mV* s^{-1}

The experimental results indicate that at the Pt electrode, reduction of In ions occur in two stages, whereas at Ni and In/Pt, it occurs in only one.

Figure 3 shows the cathode polarization curve of the electroreduction of indium ions from an aqueous electrolyte on Ni electrodes. The comparison of the two figures shows that on Ni electrodes, the reduction of indium ions occurs within the potential range of -0.75 to -1.16 V (peak a), as shown by reaction (1). Starting from -0.8 V, the process accelerates as indium deposits on the electrode surface. After -0.95 V, the current required for the process stabilizes due to passivation of the electrode surface (here, the electrode is completely covered with an indium film). Then, at a potential of -1.18 V, the current increases from 50 to 350 mA.

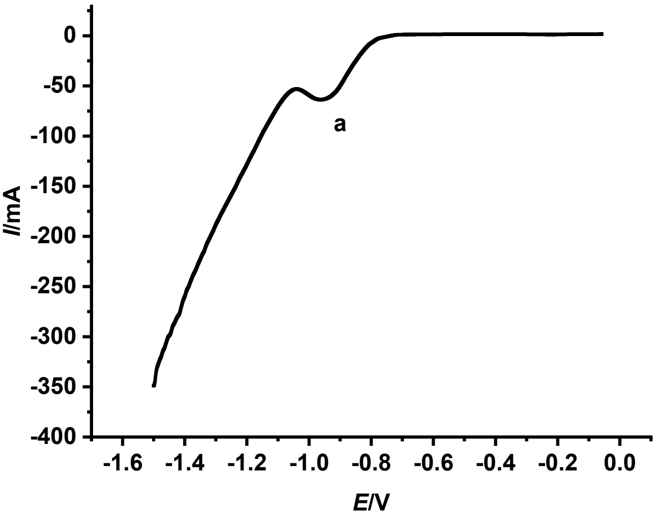

Figure 3. *Cathodic polarization curve of electroreduction of indium ions from aqueous electrolyte on Ni electrode. Electrolyte: 0.2 mol* L^{-1} *$InCl_3$, pH 2.8,* T *= 298 K, scan rate = 20 mV* s^{-1}

Electrochemical reduction of indium ions was also studied on a Pt/In electrode. As is seen from Figure 4, the process occurs in one stage at a potential range of -0.63 to -1.15 V (peak a) (in comparison with Figure 1). Here, the reduction and deposition of metallic indium on the substrate begins, starting from a potential of -0.63 V according to the reaction $In^{3+} + 3e^- = In$. The process accelerates with the deposition of indium on the electrode surface. And after a potential of -1.0 V, the current required for the process decreases from -50 to -40 mA. Beyond a potential of -1.2 V, the current associated with this process increases progressively with further cathodic polarization.

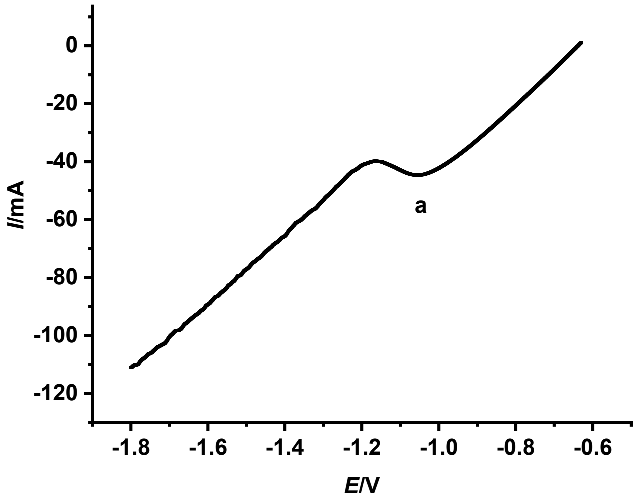

Figure 4. *Cathode polarization curve of electroreduction of indium ions from aqueous electrolyte on Pt/In electrode. Electrolyte: 0.2 mol* L^{-1} *$InCl_3$, pH 2.8,* T *= 298K, scan rate = 20 mV* s^{-1}

As is seen from Figures 1 to 4, differences in current values for Pt, Ni and Pt/In are explained by the fact that, as shown in the experimental methodology of the article, the visible surface area of the nickel electrode is 5.7 times larger than that of the platinum electrode. This also hypothesizes that the rate of indium deposition on the nickel electrode is higher than on the platinum electrode. This is due to their crystalline structures. That is, on other electrodes, indium reduction proceeds in multiple stages due to complex adsorption processes, interfacial barriers, and the stabilization of intermediate valence states. In contrast, on an indium electrode, the process occurs in a single step because indium readily deposits onto its own surface without forming intermediate energy barriers.

A rotating disk electrode (Pt) was used to determine the kinetics of the electroreduction process of indium ions in aqueous solutions.

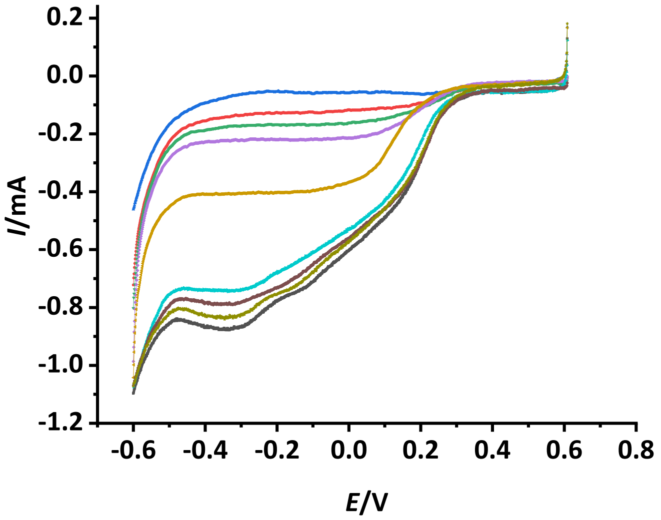

Figure 5. *Cathodic polarization curves of indium reduction on a rotating platinum electrode. Electrolyte composition: 0.2 M $InCl_3$, pH 2.8, scan rate = 20 mV s^{-1},* T *= 298 K. Electrode rotation speed: blue - 0; red - 100; green - 200; purple - 400; yellow - 800; turquoise - 1600; brown - 3200; swamp - 4500 and grey - 5600 rpm*

As shown in Figure 5, increasing the rotation intensity has a negligible effect on the cathode process rate.

Based on Figure 5, the dependence of the peak current (I_p) on the square root of rotation speed, *i.e.* angular velocity (ω = rpm×2π/60 rad), of the platinum disk electrode is plotted in Figure 6. As shown in Figure 6, with the disk electrode angular velocity increased to 335 rad s^{-1}, the current required for the process increases. After this, an imperceptible increase in current is observed, which indicates the limiting current [20].

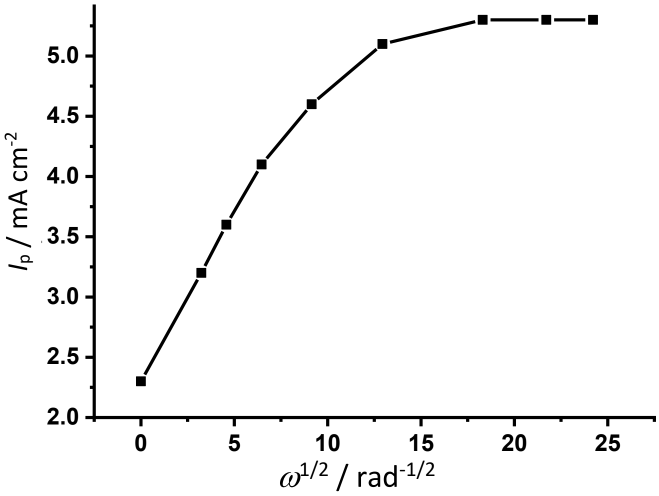

Figure 6. *Dependence of* i_p *on the square root of Pt electrode rotation speed*

The dependence of the current density on the disk electrode's rotation speed at different potentials, as shown in Figure 5, is plotted in Figure 7. As shown in Figure 7, increasing the cathode rotation speed from -0.5 to -0.6 V has little effect on the cathode process rate. The weak effect of mixing on the process rate indicates that at potentials up to -0.6 V, the transfer of discharged particles to the cathode surface is the limiting stage of the process, *i.e.* concentration polarization takes place in this case. In addition, the current decreases across all potential studied, within a mixing range of 400 to 900 rpm.

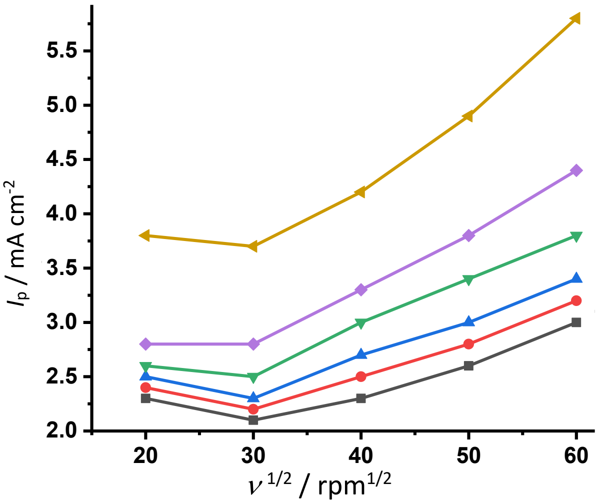

***Figure 7**. Current density as a function of disk electrode rotation speed. Electrolyte composition 0.2 M $InCl_3$ at potentials: black -0.50; red -0.55; blue -0.6; green -0.65; violet -0.7 and yellow -0.75 V*

This is explained by the formation of indium nuclei at these potentials. The diffusion rate increases after 900 rpm, and indium ions deposit rapidly on the electrode surface. If we project these lines onto the vertical axis, we observe that they intersect the axis near zero at cathode potentials from -0.5 to -0.65 V, which is typical for processes in which the limiting stage is the diffusion of the deposited ions to the cathode surface.

The effects of temperature, indium ion concentration, and other parameters on the electroreduction of indium ions from aqueous solutions were investigated to determine the optimal electrolysis conditions and electrolyte composition. The influence of temperature was studied using the potentiodynamic method over the range 298 to 328 K. The results are shown in Figure 8.

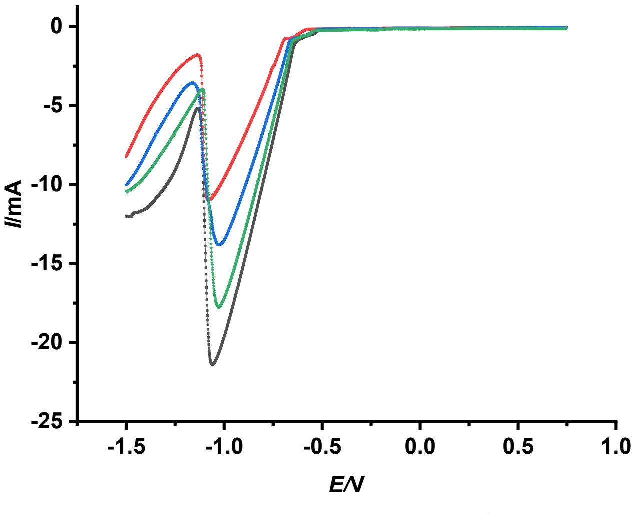

***Figure 8.** Influence of temperature on the electroreduction process of indium ions from aqueous solution on the Pt substrates. Electrolyte: 0.2 mol L^{-1} $InCl_3$. pH 2.8, scan rate 20 mV s^{-1}. T: red - 298; blue - 308; green - 31 and black - 328 K*

From the recorded cathodic polarization curves, it is evident that the temperature has a positive effect on the process. At the same time, the stationary potential shifts toward the anodic side. As shown in Figure 8, at 298 K the electrochemical reduction of indium ions begins at -0.7 V, whereas at 328 K it begins at -0.5 V.

Figure 9 shows the linear polarization curves for the effect of indium ion concentration on the electroreduction of indium. From the cathodic polarization curves, it is evident that an increase in the concentration of indium ions in the electrolyte accelerates the process, and, at the same time, the reduction potential shifts to a more cathodic region by ≈ -0.11 V. That is, the electroreduction of indium ions in high concentrations begins at a more anodic potential.

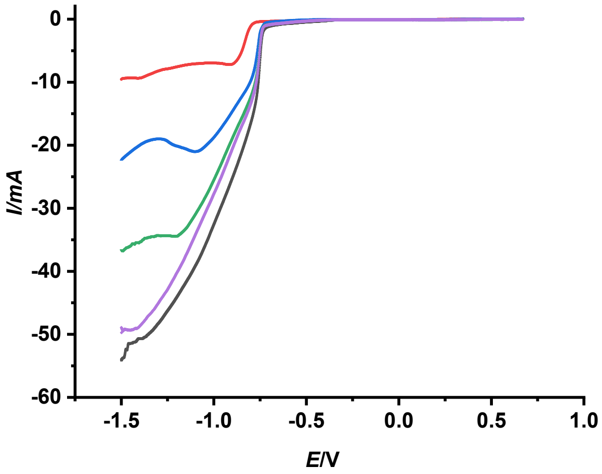

Figure 9. *Effect of indium ion concentration on the electroreduction process on Pt substrates.* $InCl_3$ *concentration: red - 0.06; blue - 0.14; green - 0.2; purple - 0.24; grey - 0.28 mol/L.* T = 298 *K, scan rate 20 mV* s^{-1}

The effect of the scan rate on the electroreduction process of indium ions was studied in the range 5 to 100 mV s^{-1}. Linear polarization curves in Figure 10 show a regular increase in current during the electroreduction of indium ions with increasing scan rate.

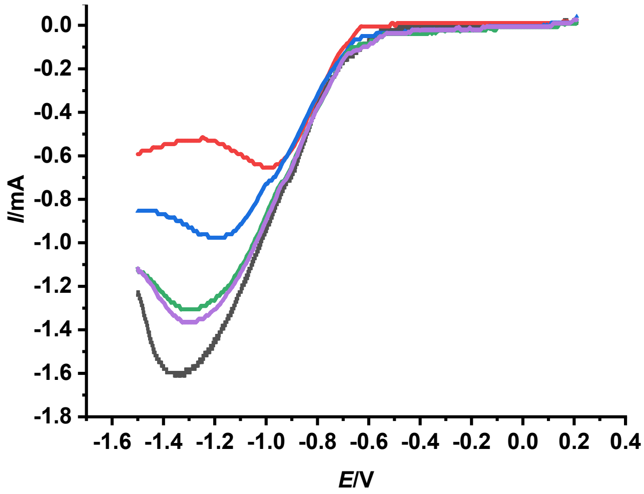

Figure 10. *Effect of the scan rate on the electroreduction process of indium ions on the Pt substrate: 0.2 M* $InCl_3$*, pH 2.8,* T = 298 *K. Scan rate: red - 5; blue - 20; green - 40; purple - 60; grey - 100 mV* s^{-1}

Conclusions

The electrochemical reduction of indium ions at Pt, Ni, and Pt/In electrodes in aqueous electrolyte was studied using cyclic voltammetry. It was determined that at the Pt electrode, reduction of In ions occurs in two stages, while at Ni and In/Pt by only one.

The results of all experiments show that indium ion concentration and temperature affect the electroreduction of indium ions in an aqueous electrolyte. With the study of the influence of these factors, the optimal electrolysis mode and electrolyte composition for the electroreduction process of indium ions were selected as: 0.2 mol L^{-1} $InCl_3$, T = 298 K and scan rate of 20 mV s^{-1}.

When studying the process kinetics and mechanism, it was determined that electroreduction proceeds via diffusion-limited kinetics. To confirm this conclusion, polarization curves were recorded on a rotating disk electrode, and the dependence i_p *vs.* $\nu^{1/2}$ was plotted, which has a linear character, which is also inherent in processes controlled by diffusion. By plotting the dependence of disk electrode rotation speed on the current density at different potentials, it was concluded that the process is accompanied by concentration polarization in the potential range of -0.5 to -0.65 V.

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Conflicts of Interest: *The authors declare that they have no conflict of interest.*

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