

Electrodeposition and characterization of Pt coatings on Ti for electrooxidation of cyanide

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Cyanide solutions used in industry are harmful not only to humans but also to other living organisms. The development of low cost and simple in preparation electrodes for cyanide electrochemical destruction is an important requirement. Ti electrodes galvanostatically covered with a Pt layer of 120, 300 and 600 nm average thickness were chosen as the anodes for electrochemical oxidation of high concentration (0.1 M) cyanide solutions. The morphology and microstructure of Pt coatings were observed by using scanning electron microscopy (SEM) and X-ray diffraction (XRD) methods. It was found from XRD data that the average Pt crystallite size is equal to 9.3 ± 0.5 nm regardless of whether the electrode has been exploited for cyanide electrochemical destruction. Electrocatalytic activity of platinized Ti electrodes was tested by determining the current efficiencies of cyanide electrooxidation that were found to be equal to 60% thus showing high effectiveness of the process. The obtained results indicate that electrocatalytic properties of the electrodes change over time as the oxidation of cyanide is carried out. Nevertheless, a Ti electrode galvanostatically covered with a Pt layer of 600 nm average thickness shows higher stability, making it applicable for effective anodic utilization of cyanide ions.

Key words: platinized titanium, cyanide, electrooxidation, Pt crystallite size

INTRODUCTION

Cyanide containing wastewater may not be disposed to the environment, especially at high cyanide concentration levels due to high toxicity to people and wildlife. Technologies for destruction of cyanide by chemical, biological and photochemical methods have been developed to reduce the concentration of cyanide and cyanide compounds to below the regulated limits in wastewaters [1]. However, all these methods are typically expensive or harmful to the environment and have limitations. In part, widely used chemical treatment methods have become limited due to the toxic nature of intermediate and by-products [2]. Effectiveness of cyanide biodegradation is limited to cyanide concentrations and to very slow detoxification and susceptible to temperature upsets [3]. UV oxidation is limited to relatively clear solutions [4, 5].

The method of electrochemical oxidation is one of attractive alternatives since it is a cost-effective and environmentally friendly way to utilize cyanide solutions producing no toxic side products [6–9]. It is important to note that electrochemical oxidation can be used to destroy high concen-

tration cyanide wastes (e. g. 0.5–1 M) at the anode [10], and to recover dissolved metals at the cathode simultaneously [11–13]. However, it is pointed out in scientific literature [14–18] that the direct oxidation of cyanide is a fairly slow reaction. For this reason, the main effort of investigators is to develop a working electrode which must be the most efficient and as resistant as possible to a strong oxidizing agent such as oxygen, because intensive discharge of water proceeds near the anode during the process of electrochemical oxidation of cyanide [19]. Platinum, steel, lead and nickel [20–23] anodes have been used in many experiments for cyanide oxidation. Unfortunately, these electrodes exhibit poor current efficiency or show poor resistance to corrosion or both. Better results were obtained by using graphite [17, 24–26], but these anodes become slowly degraded during electrolysis. Tissot and Fragniere [27] described a three-dimensional electrode for the oxidation of cyanide by the electrodeposition of PbO_2 on a reticulated vitreous carbon substrate. The authors state that the stability of this electrode is excellent. Recently, the effective destruction of cyanide was demonstrated by using an electrochemical reactor equipped with $\text{Ti/SnO}_2\text{-Sb-Ce}$ anodes [28]. However, many of the proposed electrodes are too expensive or complicated in preparation.

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Since cyanide treatment may require up-front high capital costs, the investigations for development of low cost anodes remain topical. It is likely that platinized Ti electrodes are appropriate as they have been developed for various applications. It was observed that a Ti/Pt electrode itself has higher electroactivity to the reduction of nitrate [29]. Iniesta et al. [30] reported that the active surface area of the Ti/Pt electrode is 125 times larger than that of a polycrystalline Pt electrode of the same geometric area. Due to their electrocatalytic properties Ti/Pt anodes are suitable for treating wastewaters containing dyes (methylene blue) [31]. Platinized Ti electrodes were evaluated for urea oxidation activity [32], and it was found that Ti/Pt surfaces are 1 900 times more active for urea oxidation than smooth Pt surfaces. It was demonstrated [33] that platinized Ti electrodes may serve as anodes for effective electrochemical oxidation of high concentrated cyanide solutions reaching current efficiencies comparable to those obtained using pure Pt anodes. However, the lifetime of platinized Ti electrodes in cyanide destruction was not investigated elsewhere.

The main goal of this work was to characterize the Pt layer of platinized Ti electrodes before and after exploitation for cyanide electrooxidation, and to investigate possible catalytic property changes of Pt coatings during the process of cyanide destruction.

MATERIALS AND METHODS

All experiments were performed at 21 °C temperature, and all solutions were prepared using distilled water. Ti plates (99.2% pure, Alfa Aesar GmbH & Co, Germany) 2 cm² in area and 1 mm in thickness were used as substrates for Pt electrodeposition. Before electrochemical platinization, the plates of Ti were mechanically polished to remove any contaminants as well as to create a certain level of roughness. Then, the Ti plates were degreased and thoroughly washed with distilled water. Any special procedures, such as electrocleaning, acid etching etc., were not used to ensure a low cost and simple in preparation process. The polished and washed Ti electrodes were electroplated galvanostatically at 1.5 mA cm⁻² of current density in a platinum solution of such composition: 3.764 g L⁻¹ Pt (puriss p. a. Alfa Aesar GmbH & Co, Germany) and 250 g L⁻¹ HCl (puriss p. a., Standard, Poland). The time of electrolysis was calculated according to the thickness of Pt needed. However, these thicknesses are equivalent to a theoretical 100% yield, though real surface coverage is dependent on the practical current yield (aprox. 60%) and morphology of electrodeposits. In consideration of this fact the average thicknesses of 120, 300 and 600 nm of Pt were used throughout the paper.

Scanning electron microscopy (SEM) was carried out with a Hitachi TM 3000 Tabletop Microscope using accelerating voltage of 15 kV. Platinized Ti electrodes with Pt coatings of different average thickness were used for the SEM analysis before and after electrooxidation of cyanide to observe the morphology changes of the surface.

The XRD patterns were recorded using a MiniFlex 2 Rigaku X-ray diffractometer (Cu-K_α radiation, Bragg-Brentano geometry, a high-speed semiconductor element one-dimensional x-ray detector D/teX Ultra). The X-ray generator was operated at 30 kW and 15 mA. XRD measurements were performed on different samples of the platinized Ti electrodes before and after the electrooxidation of cyanide to observe possible crystalline phase changes of Pt coatings. The Program PDXL 1.8.1.0 was used to process the results and to evaluate average Pt crystallite sizes.

In order to evaluate the rate of cyanide degradation and to obtain the current efficiencies of the anodic process, the electrooxidation of 0.1 M KCN solution was carried out using the Ti/Pt anode and stainless steel plates as counter electrodes. The applied current density was 25 mA cm⁻². The information about the electrode catalytic property changes over time was detected by extending the cyanide electrolysis time. The procedure for electrooxidation of cyanide and the calculation of the current efficiencies and concentration of cyanide ions were described in our previous work in greater detail [33].

RESULTS AND DISCUSSION

Characterization of Ti/Pt electrodes before and after cyanide electrooxidation

SEM analysis

The morphology of Ti electrodes with 120, 300 and 600 nm average thickness Pt coatings was investigated by using SEM. The parameters of the surface of platinized Ti electrodes are presented in Table 1. Figure 1 shows the distribution and structure of Pt microparticles (white spots) on Ti surfaces before using the electrodes for cyanide electrooxidation. It was determined that the spherical shape platinum microparticles form on the Ti electrode surface and their density increases with the thickness of the Pt layer. The elemental composition in the chosen areas revealed that the electrode surface structure consists of 20–30% Pt microparticles when an average coating thickness is 120 nm (Fig. 1a) and 50–70% when an average coating thickness is 300 nm (Fig. 1b). At 600 nm of the calculated average thickness, more than 90% of the electrode surface is covered by Pt microparticles (Fig. 1c). Figures 1a, b clearly demonstrate that the microparticles of Pt are spatially distributed in a random fashion, ranging in a diameter from 1 to 2 μm leaving large portions of the electrode surface uncovered. Upon deposition of 600 nm of the calculated average thickness Pt layer (Fig. 1c), overlap of spherical Pt microparticles was observed and the surface of Ti became almost completely covered by the Pt layer.

The surfaces of electrochemically platinized Ti electrodes were once again interrogated by the SEM method after using them for the anodic oxidation of cyanide ions. It was found that the Pt coating remains on the Ti electrode surface after 5 h of anodic oxidation of cyanide ions (this is equivalent to 900 C of charge passing on 2 cm² electrode at 25 mA cm⁻²

Table 1. The parameters of Pt coatings obtained by using the SEM method before and after exploitation for cyanide electrooxidation

Average thickness of Pt layer, nm	Before CN ⁻ oxidation			After 50 h CN ⁻ oxidation		After 100 h CN ⁻ oxidation
	120	300	600	300	600	600
Diameter of Pt microparticles, μm	1.0–2.0	1.0–1.5	1.5–2.0	1.0–1.5	1.0–2.0	0.5–1.0
Percentage of Pt microparticles, %	20–30	50–70	90–95	30–40	60–70	50–60

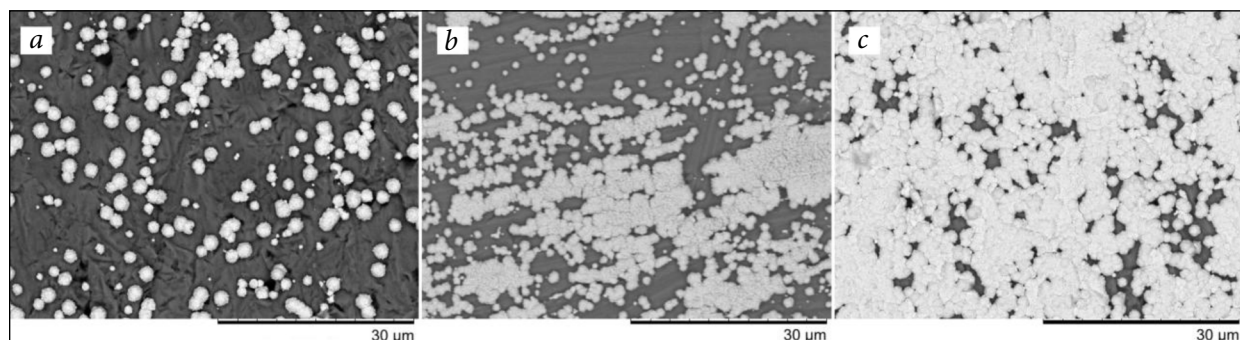


Fig. 1. SEM images of the surface of electrochemically platinized Ti electrodes before exploitation for cyanide electrooxidation. The average thickness of Pt layer: *a* – 120 nm, *b* – 300 nm, *c* – 600 nm

current density), however, the percentage of Pt particles on the electrode surface decreases by about 5–10% (data not shown). Then, cyanide electrolysis time was extended up to 50 h, and the SEM analysis of the used electrodes allowed us to see unequal changes in Pt coatings (Fig. 2). In particular, for the 120 nm average thickness of the Pt layer electrode (data not shown), it was found that the Pt coating was almost completely lost from the electrode surface (only 1–3% left). Meanwhile, for the Ti electrodes with the 300 nm and 600 nm average thickness Pt layer, the remaining percentage of Pt particles on the electrode surface decreased by about 10–20% (Fig. 2a, b and Table 1), but a big portion of the electrode surface was still covered by Pt clusters. The Ti electrode with the initial 600 nm average thickness of the Pt layer was further used for cyanide electrooxidation for another 50 h, extending the total electrolysis time to 100 h. After the experiment, it was found that Pt microparticles decreased in diameter to 0.5–1 μm (Table 1). However, even ~55% of the Pt

coating remained on the surface of the Ti electrode (Fig. 2c), making it applicable for further anodic cyanide oxidation.

XRD analysis

Platinized Ti electrodes with the 600 nm average thickness Pt coating were investigated by using the XRD method. The obtained diffractograms are shown in Fig. 3a. According to Swanson and Tatge [34], the polycrystalline Pt metal has main XRD peaks due to the crystal planes of 111, 200, 220 and 311 at 2θ of 39.76°, 46.24°, 67.46° and 81.29°, respectively. The obtained diffraction peaks (Table 2) clearly indicate that the investigated Pt coatings are polycrystalline. Some deviations of 2θ values from those of the pure Pt metal were obtained for all platinized Ti electrodes before and after using them for cyanide electrooxidation. A shift in the diffraction peaks to higher 2θ values can be caused by tensile stress or by strain (crystal defects), e. g. crack, dislocation. Also, it is known that the XRD patterns of platinized

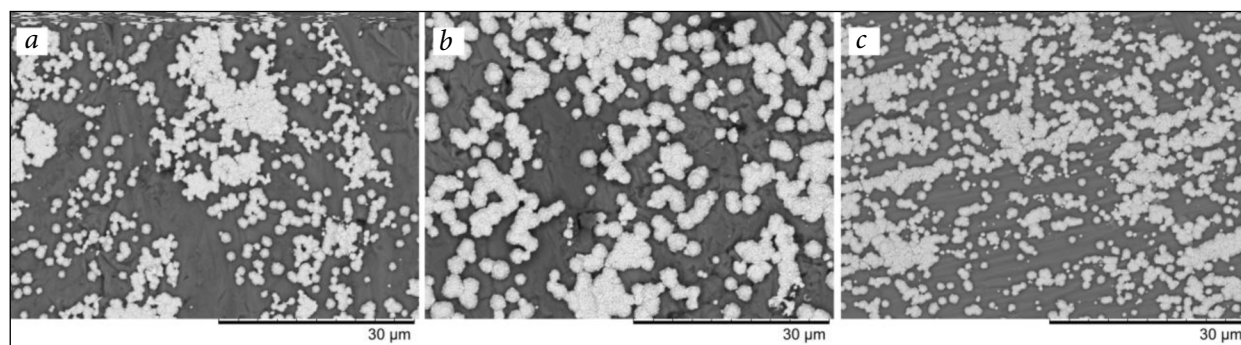


Fig. 2. SEM images of the surface of electrochemically platinized Ti electrodes after exploitation for (a, b) 50 h, (c) 100 h of 0.1 M KCN electrooxidation. The average thickness of the Pt layer: *a* – 300 nm, *b* and *c* – 600 nm

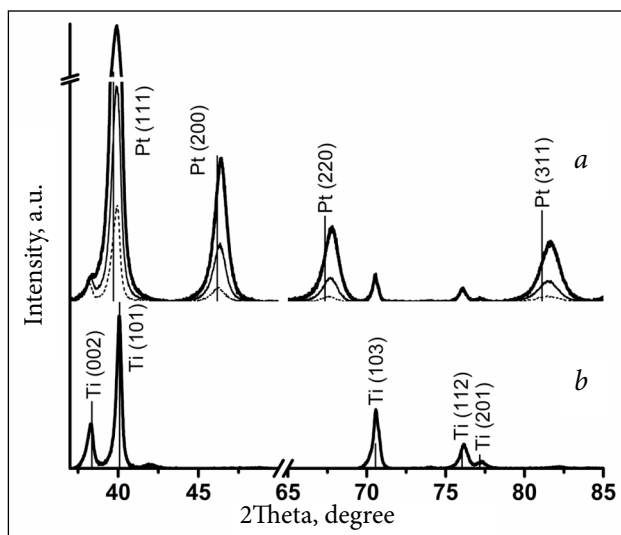


Fig. 3. XRD patterns of (a) Ti electrodes covered with 600 nm average thickness of Pt: — before 0.1 M KCN electrooxidation, — after 50 h of 0.1 M KCN electrooxidation, after 100 h of 0.1 M KCN electrooxidation; (b) pure Ti electrode before platinization

Ti electrodes are affected by electrode treatment, such as electrocleaning [35].

One can see in Fig. 3a that the full width of the diffraction peaks of Pt coatings and the intensities at each plane decreased after applying platinized Ti electrodes for cyanide oxidation depending on the electrolysis time. These results indicate that the Pt coating diminishes during the process of cyanide destruction. Meanwhile, no changes of the XRD parameters were determined for Ti. Thus, it is possible to put all patterns of Pt coatings on a normalized intensity of Ti (Fig. 3b) and to calculate the average Pt crystallite size by using the Scherrer equation [36]. The corresponding values for Ti/Pt electrodes before and after cyanide electrooxidation are listed in Table 2. The average Pt crystallite size was found to be equal to 8.8–9.8 nm, and hardly shows any dependence on exploitation for cyanide electrooxidation. Thus, it can be concluded that the process of electrochemical oxidation of cyanides has no significant influence on the crystallinity of the Pt coatings.

Electrocatalytic activity of Ti/Pt electrodes

Electrocatalytic activity of the platinized Ti electrodes was investigated by using them for electrooxidation of a 0.1 M KCN solution. During the process of electrolysis, the concentration of cyanide ions was measured every 1 hour and current efficiencies of cyanide electrooxidation were calculated respectively (Figs. 4, 5). It was found that cyanide electrooxidation proceeds slowly on the Ti anode covered with the 120 nm average thickness Pt layer (Fig. 4a, grey columns). If the average thickness of the Pt layer is 300 or 600 nm, the concentration of cyanide ions decays much faster. In particular, for the electrode with the 600 nm average thickness Pt layer (Fig. 4a, black columns), the cyanide concentration after 5 h of electrolysis was found to be close to zero, i. e. the amount of cyanide ions for the investigated solution decreased to less than 1%. From the data in Fig. 5 it can be seen that the highest current efficiencies of 50–60% are reached during the first 2–3 hours of electrolysis, and after 900 C of charge passing (equivalent to 69 kC L⁻¹ of charge) the current efficiencies of approx. 20–25% are still obtained. It is important that by applying electrochemically platinized Ti electrodes the process of cyanide electrooxidation effectively proceeds without using any chemical additives reaching low levels of the residual cyanide concentration.

The electrocatalytic activity of platinized titanium electrodes was investigated once again extending the electrolysis time of 0.1 M KCN solution up to 50 and 100 h (Figs. 4b, 5b). It was found that the anode covered with the Pt layer of 120 nm average thickness is already unsuitable for oxidation of cyanide ions after only 20 h (data not shown). The oxidation of cyanide ions is still taking place after 50 h of electrolysis if the Ti anodes covered with the 300 or 600 nm average thickness Pt layer are used. However, for the Ti anode covered with the Pt layer of 300 nm average thickness (Figs. 4b, 5b, white columns), the rate of cyanide electrooxidation is much slower and the obtained current efficiencies are rather small compared to those obtained during the first 5 h of electrolysis. Nevertheless, by using the Ti electrode covered with the 600 nm average thickness Pt layer the current efficiencies of cyanide electrooxidation still keep high values (Fig. 5b, black columns) reaching over 60%. These results

Table 2. The parameters of the XRD patterns of platinized Ti electrodes with the 600 nm average thickness of the Pt coating before and after exploitation for cyanide electrooxidation

Pt crystal plane, hkl	2θ, deg. [34]	Before cyanide oxidation			After 50 h cyanide oxidation			After 100 h cyanide oxidation		
		2θ, deg.	Pt crystallite size, nm	Pt crystallite size average, nm	2θ, deg.	Pt crystallite size, nm	Pt crystallite size average, nm	2θ, deg.	Pt crystallite size, nm	Pt crystallite size average (nm)
111	39.76	39.90	—*		39.99	—		40.03	—	
200	46.24	46.42	10.2	9.4 ± 0.5	46.37	10.0	8.8 ± 0.9	46.28	10.8	9.8 ± 1.7
220	67.46	67.83	9.3		67.75	9.0		67.50	11.3	
311	81.29	81.76	8.7		81.59	7.4		81.61	7.3	

* Pt crystallite size for Pt(111) was not calculated because XRD peaks of Pt(111) overlap with Ti(101) and Ti(002).

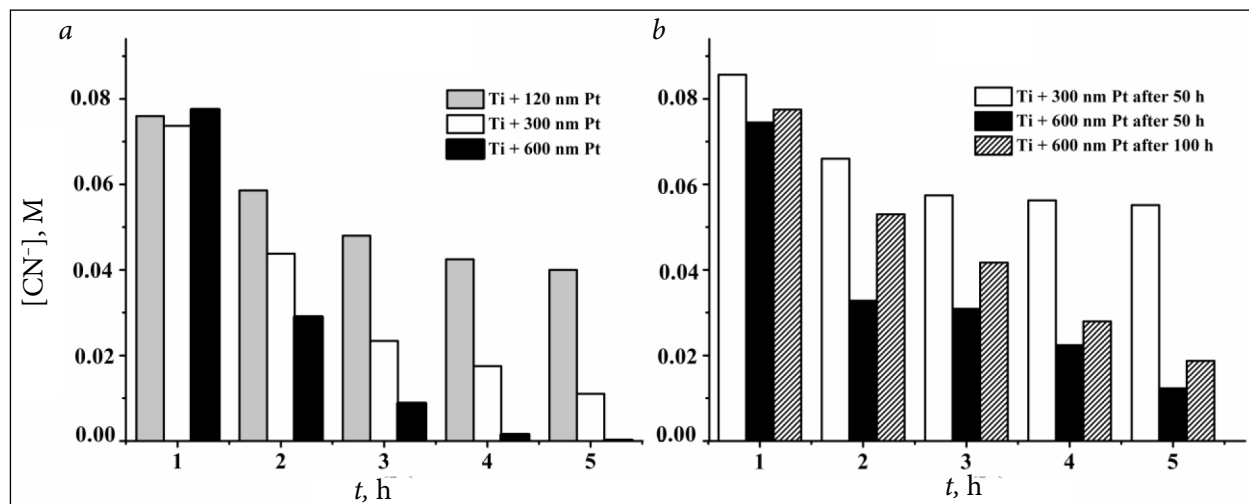


Fig. 4. Dependencies of cyanide ions concentration on the time of electrolysis using platinized Ti electrodes (a) freshly prepared; (b) after exploitation for 50 h and 100 h of 0.1 M KCN electrooxidation. Applied current density: 25 mA cm^{-2}

show that the investigated electrode can be used yet again for another 50 h of cyanide electrolysis. One can see in Figs. 4b and 5b (striped columns) that the effectiveness of the cyanide electrooxidation only slightly decreased, nevertheless, the Ti electrode covered with the 600 nm average thickness Pt layer remains suitable to be used even after 100 h of electrolysis. Finally, it can be stated that the electrochemical oxidation of cyanide diminishes the electrocatalytic activity of Ti electrodes covered with the 120 or 300 nm average thickness Pt layer, i. e. when a large portion of the Ti surface remains uncovered by Pt microparticles. It could be expected that during anodic polarization of the electrode, growth of the TiO_2 film

layer on the uncovered areas of the Ti surface inevitably occurs, causing degradation of the Pt coating. However, if the Pt coating is equal to 600 nm, i. e. almost all surface area of Ti is covered by the Pt layer, the electrode shows high stability and high electrocatalytic activity for electrochemical cyanide destruction. Taking into account that galvanostatic platinization is a cheap and simply applicable method, it can be concluded that Ti electrodes, covered only by a submicroscopic layer of the 600 nm average thickness Pt, could enable to economise the natural resources of Pt, and could be proposed as an attractive alternative for expensive and complicated in preparation electrodes used for utilization of cyanide wastes.

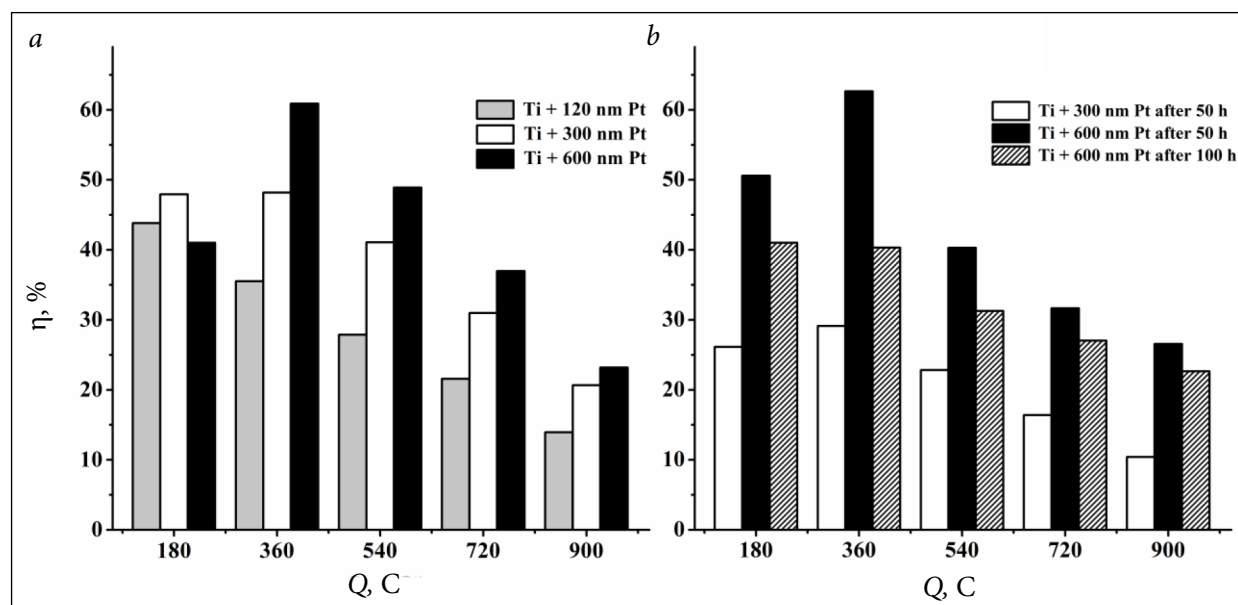


Fig. 5. The plot of current efficiency of cyanide electrooxidation vs the charge passed through 0.1 M KCN electrolyte solution using platinized Ti electrodes (a) freshly prepared; (b) after exploitation for 50 h and 100 h of cyanide electrooxidation. Applied current density: 25 mA cm^{-2}

CONCLUSIONS

Ti electrodes galvanostatically covered with the 600 nm average thickness layer Pt are suitable for oxidation of a high concentration cyanide solution showing high stability and high electrocatalytic activity for electrochemical cyanide destruction. However, the Ti electrodes covered with the 120 nm or 300 nm average thickness Pt layer considerably degrade during the cyanide destruction process, causing decrease in the diameter and percentage of Pt microparticles. Taking into account that platinized Ti electrodes covered only by a sub-microscopic layer of Pt could replace expensive Pt anodes, and that utilization of high concentration cyanide solutions is inevitably required in chemical laboratories and institutions of chemical industry, we state that our findings are important for developing a cheap and simple in exploitation method for destruction of cyanide ions.

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TI ELEKTRODŲ ELEKTROCHEMINIS PLATINAVIMAS IR Pt DANGŲ TINKAMUMO CIANIDŲ ELEKTROOKSIDACIJAI TYRIMAS

Santrauka

Ti elektrodai, galvanostatiškai padengti 120, 300 ir 600 nm storio Pt dangomis, buvo naudojami 0,1 M KCN tirpalo elektrooksidacijai. Elektrodų elektrokatalizinis aktyvumas įvertintas nustatant cianido jonų elektrooksidacijos srovines išeigas, atliekant skirtingos trukmės KCN tirpalo elektrolizę. Pt dangų morfologija ir kristališkumas buvo tiriami naudojant skenuojančios elektroninės mikroskopijos bei rentgeno spindulių difrakcijos metodus. Nustatyta, kad 120 ir 300 nm storio Pt sluoksniu dengtų Ti elektrodų elektrokatalizinis aktyvumas cianido jonų elektrooksidacijos metu mažėja, tačiau 600 nm storio Pt sluoksniu padengti Ti elektrodai pasižymi aukštu stabilumu ir gali būti efektyviai naudojami cianido jonams utilizuoti.