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ELECTROCHEMICAL BEHAVIOR OF PHOSPHOROUS ACID ON A PLATINUM ELECTRODE AND ITS GALVANOSTATIC ANODIC OXIDATION USING A LEAD ANODE

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Abstract. Anodic oxidation of reduced phosphorus forms - hypophosphorous and phosphorous acids is an attractive approach from both fundamental science and applied applications (purification and phosphorus recycling). The aim of this research is to investigate the anodic oxidation of phosphorous acid under galvanostatic conditions and to comprehensively evaluate the influence of key parameters on the process efficiency and reaction mechanism. Electrochemical property of phosphorous acid was carried out by recording cyclic voltammetry using a Potentiostat/galvanostat Electrochemical Workstation Corrtest Instruments and electrolysis under galvanostatic condition. The influence of the current density, electrolyte concentration, and temperature on the current efficiency of phosphorous acid oxidation has been studied. The obtained results show that the two distinct anodic peaks observed at a potential of approximately +0,7 V and a potential of approximately +0,95 V upon the addition of H_3PO_3 can be confidently assigned to the stepwise electro-oxidation of phosphorous acid. The results demonstrate that under electrolysis conditions, the anodic oxidation of phosphorous

acid yields phosphate ions. Based on the electrochemical data, it was hypothesized that the formed lead oxide layer exhibits an electrocatalytic effect toward this process, significantly enhancing the anodic currents. The practical significance of this work lies in the scientific substantiation and selection of optimal process parameters for the anodic oxidation of phosphorous acid. Replacing expensive noble metals with a lead anode—which is both cost-effective and corrosion-resistant in sulfuric acid media - ensures a high current efficiency for the target product. Based on the obtained scientific results, the proposed electrochemical method for purifying thermal phosphoric acid was tested at the «NDFZ» LLP production facility.

Keywords: phosphorous acid, electrooxidation, cyclic voltammetry, electrolysis, current efficiency, phosphate-ions

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ФОСФОРЛЫ ҚЫШҚЫЛДЫҢ ПЛАТИНА ЭЛЕКТРОДЫНДАҒЫ ЭЛЕКТРОХИМИЯЛЫҚ ҚАСИЕТІ ЖӘНЕ ОНЫҢ ҚОРҒАСЫН АНОДЫН ҚОЛДАНЫП ГАЛЬВАНОСТАТИКАЛЫҚ АНОДТЫ ТОТЫҒУЫ

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Аннотация. Фосфордың тотықсызданған түрлерінің, атап айтқанда гипофосфорлы және фосфорлы қышқылдардың анодтық тотығуы іргелі зерттеулер тұрғысынан да, фосфорды тазарту мен қайта қалпына келтіру сияқты қолданбалы міндеттер үшін де маңызды бағыт болып табылады. Зерттеудің мақсаты — фосфорлы қышқылдың гальваностатикалық жағдайдағы анодтық тотығуын зерттеу және процестің тиімділігі мен реакция механизміне негізгі параметрлердің әсерін кешенді бағалау. Фосфорлы қышқылдың электрохимиялық қасиеттері Corrtest потенциостат-гальваностаты көмегімен циклдік вольтамперометрия әдісімен зерттеліп, кейін гальваностатикалық электролиз жүргізілді. Ток тығыздығының, электролит концентрациясының және температураның фосфорлы қышқылдың тотығу тоғы бойынша шығымына әсері анықталды. H_3PO_3 қосылғаннан кейін шамамен +0,70 және +0,95 В потенциалдарында байқалған екі айқын анодтық шың фосфорлы қышқылдың сатылы электрототығуына жатқызылды. Электролиз жағдайында анодтық тотығу нәтижесінде фосфат-иондары түзілетіні анықталды. Электрохимиялық деректер электрод бетінде түзілген қорғасын оксиді қабатының электрокаталитикалық әсер көрсетіп, анодтық токтарды арттыратынын болжауға мүмкіндік береді. Зерттеудің практикалық маңыздылығы фосфорлы қышқылдың анодтық тотығуының оңтайлы технологиялық параметрлерін ғылыми негіздеу және таңдаумен анықталады. Қымбат асыл металдардың орнына күкірт қышқылы ортасында коррозияға төзімді және экономикалық тиімді қорғасын анодын пайдалану мақсатты өнімнің ток бойынша жоғары шығымын қамтамасыз етеді. Алынған нәтижелер негізінде термиялық фосфор қышқылын тазартудың ұсынылған электрохимиялық әдісі «НДФЗ» ЖШС өндірістік жағдайында сынақтан өткізілді.

Түйін сөздер: фосфорлы қышқыл, электрототығу, циклді вольтамперометрия, электролиз, ток бойынша шығым, фосфат-ионы

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ЭЛЕКТРОХИМИЧЕСКОЕ ПОВЕДЕНИЕ ФОСФОРИСТОЙ КИСЛОТЫ НА ПЛАТИНОВОМ ЭЛЕКТРОДЕ И ЕЕ ГАЛЬВАНОСТАТИЧЕСКОЕ АНОДНОЕ ОКИСЛЕНИЕ С ИСПОЛЬЗОВАНИЕМ СВИНЦОВОГО АНОДА

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Аннотация. Анодное окисление восстановленных форм фосфора, в частности фосфорноватистой и фосфористой кислот, представляет интерес как с точки зрения фундаментальных исследований, так и для решения прикладных задач, связанных с очисткой и рекуперацией фосфора. Цель исследования заключалась в изучении анодного окисления фосфористой кислоты в гальваностатических условиях и комплексной оценке влияния основных технологических параметров на эффективность процесса и механизм реакции. Электрохимическое поведение фосфористой кислоты исследовали методом циклической вольтамперометрии с использованием потенциостата-гальваностата Corrtest, после чего проводили гальваностатический электролиз. Изучено влияние плотности тока, концентрации электролита и температуры на выход по току при окислении фосфористой кислоты. После добавления H_3PO_3 на вольтамперограммах наблюдались два выраженных анодных пика при потенциалах около +0,70 и +0,95 В, которые были отнесены к последовательным стадиям электроокисления фосфористой кислоты. Установлено, что в условиях электролиза анодное окисление приводит к образованию фосфат-ионов. На основании электрохимических данных выдвинуто предположение, что образующийся на поверхности электрода слой оксида свинца проявляет электрокаталитическое действие и способствует увеличению анодных токов. Практическая значимость работы заключается в научном обосновании и выборе оптимальных параметров анодного окисления фосфористой кислоты. Замена дорогостоящих благородных металлов свинцовым анодом, характеризующимся экономичностью и коррозионной устойчивостью в сернокислой среде, обеспечивает высокий выход целевого продукта по току. Разработанный электрохимический способ очистки термической фосфорной кислоты был апробирован в производственных условиях ТОО «НДФЗ».

Ключевые слова: фосфористая кислота, электроокисление, циклическая вольтамперометрия, электролиз, выход по току, фосфат-ионы

Introduction. Kazakhstan is one of the largest producers of yellow phosphorus. The main man-made waste product of phosphorus production with a high content of phosphorus and its compounds is phosphorus-containing sludge, which is formed at the stage of yellow phosphorus condensation as a result of incomplete purification of waste dust and gas components.

Thermal sludge phosphoric acid is a product of the thermal production of orthophosphoric acid. It contains phosphorus from the raw materials and suspended solids (sludge) resulting from air or equipment contamination.

Underoxidized forms of phosphorus exist as elemental phosphorus (P_4), hypophosphorous acid (H_3PO_2) and phosphorous acid (H_3PO_3) (Bayeshov et al., 1987).

Purification of thermal slurry phosphoric acid is a critical task for ensuring the production of high-quality phosphoric acid and compliance with environmental standards. Various purification methods, such as flotation, precipitation, ion exchange, sorbent use, and chemical oxidation, effectively remove impurities and improve the quality of the final product. However, further improvement of purification processes requires continued research into new technologies and materials.

The electrochemical oxidation of lower phosphorus oxides has both theoretical and practical significance. Theoretical significance is due to the lack of research on the subject and the contradictory nature of the available data. From a practical point of view, the oxidation of under-oxidized forms of phosphorus allows the purification of thermal sludge phosphoric acid obtained during the processing of phosphorus sludge.

Literary review

The purification of thermal slurry phosphoric acid derived from industrial wastes represents a major technological and environmental challenge due to its highly complex multi-component chemical composition and aggressive acidic matrix. The primary problem is that the target product - orthophosphoric acid (H_3PO_4 , oxidation state +5) - is accompanied by substantial amounts of dissolved lower-valence, underoxidized phosphorus species, predominantly represented by phosphorous (H_3PO_3) and hypophosphorous (H_3PO_2) acids. Additionally, the raw technical medium is heavily contaminated with colloidal particles, polyphosphates, organic surfactants, and heavy metal ions such as Fe^{3+} , Al^{3+} , and Ca^{2+} (Petrov et al., 2016; Raut et al., 2021). This severely limits the direct industrial application of the acid and requires deep, selective purification.

Traditional industrial approaches rely primarily on chemical precipitation and coagulation methods. These techniques involve the addition of various external reagents, including iron sulfate, lime, and organic polyelectrolytes, to induce the aggregation and precipitation of colloidal impurities and heavy metals (Mhedhbi et al., 2023; Kumar et al., 2019). Sorption techniques utilizing activated carbons and surface-modified materials are also considered highly promising for removing organic components (Raut et al., 2021). However, despite their localized efficiency, almost all conventional chemical and sorption methods possess significant drawbacks: they are inevitably accompanied by the formation of massive volumes of secondary hazardous chemical sludges, require a continuous consumption of costly reagents, and demand multiple additional processing and filtration stages.

The electrochemical oxidation of lower phosphorus acids has emerged as a much more attractive approach from both fundamental science and applied engineering. Converting underoxidized forms of phosphorus directly solves the core problem of purifying thermal slurry phosphoric acid while enabling efficient phosphorus recycling (Bayeshov et al., 2001; Zhang et al., 2020). Early reports highlighted that lower phosphorus oxoacids could be effectively converted under specific conditions. For instance, A.P. Tomilov (Tomilov et al., 1996) reported that H_3PO_3 oxidation is efficient

on a graphite electrode in the presence of 0,5 N hydrochloric acid. The literature also demonstrates that H_3PO_3 oxidizes effectively even on a nickel anode when a specific ratio between disodium phosphite and caustic soda is maintained in alkaline solutions, with the degree of conversion reaching up to 99%. However, research regarding these alternative environments is still in its early stages, and a significant number of questions remain unanswered regarding efficient operation in highly acidic solutions.

To understand the fundamental kinetics and reaction pathways of lower phosphorus oxoacids, platinum (Pt) has traditionally served as the primary model catalyst. Extensive research has been dedicated to studying the electrochemical behavior of phosphorus-containing acids on Pt electrodes (Prokop et al., 2015; Prokop et al., 2016; Gomes et al., 2022). According to Lunyatskas (Lunyatskas, 1965), phosphites in an alkaline solution can be oxidized on a platinum anode with a current and substance efficiency of roughly 40%. A key finding in this domain is that H_3PO_3 and its reduction products exhibit exceptionally strong adsorption behavior on the platinum surface. This severe adsorption leads to the complete blocking of active catalytic sites, which drastically reduces the overall efficiency of electrochemical reactions, including the oxygen reduction reaction (ORR) in high-temperature proton-exchange membrane fuel cells (HT-PEMFC) (Prokop et al., 2015).

Furthermore, recent cutting-edge investigations (Wibowo et al., 2023; Wibowo et al., 2024) have revealed that Pt is capable of catalyzing the oxidation of an aqueous solution of H_3PO_3 to H_3PO_4 even under room conditions without any external current application. Utilizing advanced surface-sensitive analytical techniques, it was demonstrated that while H_3PO_3 remains entirely stable in the bulk solution, it undergoes unique spontaneous chemical transformations at the Pt/ H_3PO_3 interface. At this boundary, the acid is oxidized by water molecules, simultaneously releasing molecular hydrogen (H_2). The authors suggested that the strong adsorption of H_3PO_3 on the platinum surface strictly in its reactive pyramidal tautomeric form is the key driving force behind this interfacial catalytic phenomenon.

While platinum provides deep insights into the fundamental interfacial kinetics and adsorption phenomena of H_3PO_3 , its prohibitive cost restricts its use in large-scale technological applications. Consequently, researchers have extensively sought alternative, industrial-grade anode materials, focusing heavily on lead (Pb), lead dioxide (PbO_2), and carbonaceous substrates. However, historical data regarding these materials remain highly contradictory.

Early literature explicitly demonstrates that lower oxoacids of phosphorus can exhibit completely different reactivities depending on the specific nature of the alternative anode surface. In a report by Baudler and Schellenberg (Osadchenko, 2001), it was stated that phosphorous acid is readily and effectively oxidized to phosphoric acid on a conventional lead anode, though no quantitative kinetic metrics were provided. In sharp contrast to these findings, Hickling and Johnson (Osadchenko, 2001) strongly asserted that no noticeable oxidation of H_3PO_3 is practically observed on platinum, graphite, lead, and lead dioxide anodes up to the potentials of intense oxygen evolution. According to their results, the process is kinetically blocked on these surfaces, and only the addition

of potassium iodide (KI) as a homogeneous catalyst to the solution allows a reasonable current efficiency of 65% to be achieved.

The mechanism and the reasons behind these discrepancies were partially addressed by Trasatti and Alberti (Tomilov et al., 1996), who concluded that the oxidation of H_3PO_3 is heavily dictated by the catalytic properties of the anode surface and the specific tautomeric form of the acid. They also expressed the pioneering opinion that hypophosphorous acid is oxidized via a highly similar pathway to that of phosphoric and phosphorous acids. To expand the understanding of elemental phosphorus conversions, in our early studies, the electrochemical properties of red phosphorus were thoroughly investigated using voltammetric measurements with a novel phosphorus-graphite composite electrode developed by our group (Bayeshov, 2025).

Nevertheless, our comprehensive literature review reveals a very limited number of scientific publications specifically dedicated to the anodic electrochemical conversion of H_3PO_3 in highly acidic media. A significant portion of modern studies still focuses on either complex chemical or sluggish biological oxidation pathways. The available electrochemical data and fundamental insights are largely based on historical results obtained in previous decades, and alternative electrode materials - except for platinum - have not been fully or systematically explored. Crucially, a systematic and rigorous comparison of stable base-metal electrodes operating in highly acidic, industrial-like media remains absent, and the long-term electrochemical stability, selectivity, and kinetic parameters of base-metal anodes (such as lead) remain inadequately discussed.

Accordingly, the *aim of this research* is to investigate the anodic oxidation of phosphorous acid under galvanostatic conditions and to comprehensively evaluate the influence of key operational parameters - namely current density, temperature, and supporting electrolyte concentration - on the process efficiency and reaction mechanism.

The *scientific novelty* of this work lies in establishing that phosphorous acid can be effectively oxidized via a direct electrochemical pathway on a cost-effective lead anode in strongly acidic solutions, bypassing the requirement for expensive noble-metal catalysts.

Methods and materials. The electrochemical behaviour of phosphorous acid was studied by recording cyclic anode-cathodic and cathode-anodic potentiodynamic polarization curves using a CS350 Potentiostat/galvanostat (Electrochemical Workstation Corrtest Instruments, China) equipped with Corrtest software for real-time data acquisition and three-electrode electrochemical cell. The CS Studio6 version software was installed on the instrument that was used to perform data capture and analyses during the studies. The main polarization curves were recorded at the potential sweep rate of 50 mV/sec. Electrochemical measurements were performed in a 4-port glass electrochemical cell with a total solution volume of 100 mL at the room temperature without stirring.

All chemical reagents used in this study were of analytical grade and used as received without further purification. Phosphorous acid (H_3PO_3 , purity $\geq 98,0\%$) was purchased from Sigma-Aldrich, and sulfuric acid (H_2SO_4 , 95–98%, density 1,84 g/cm³) was

obtained from Merck. High-purity distilled water (with a specific conductivity of less than $2,0\ \mu\text{S}/\text{cm}$) was utilized for the preparation of all aqueous solutions.

Electrodes Preparation. Saturated silver chloride Ag/AgCl ($E = +0,203\ \text{V}$) and graphite ($\varnothing 3\text{mm}$) were used as reference and counter electrodes, respectively. The working platinum anode was prepared immediately before each experiment: its surface was mechanically cleaned with P1200 grit sandpaper to remove natural oxide films, then degreased in ethanol and rinsed with distilled water. The counter graphite electrode was rinsed with distilled water before each experiment. The potentials were measured against a silver/silver chloride (Ag/AgCl) reference electrode filled with a 3 M KCl solution. The potential of the reference electrode was $+0,203\ \text{V}$ vs. SHE at a temperature of $25\ ^\circ\text{C}$. The reference electrode was maintained in a standard working condition and did not undergo any mechanical treatment. The interelectrode distance between the anode and cathode was strictly fixed at 15 mm.

A platinum (Pt) disk electrode with a geometric surface area of $4\ \text{mm}^2$ was employed as the working electrode. A high-purity graphite rod with a surface area of $4\ \text{cm}^2$ served as the counter electrode. The interelectrode distance between the working and reference electrodes was fixed at 4 mm.

Lead of grade C0 (99,992% purity) with a working surface area of $6\ \text{cm}^2$ was used as the anode during electrolysis. A graphite rod with a diameter of 4 mm and a working section length of 20 mm served as the cathode. The interelectrode distance was set at 3.5 cm. Before each experiment, the surface of the lead anode was mechanically cleaned using P1200-grit abrasive paper, degreased in ethanol, and rinsed with distilled water. The graphite cathode was cleaned and rinsed prior to use.

The use of two different electrode materials and experimental setups (a model platinum anode for voltammetry and a lead anode for preparative galvanostatic electrolysis) stems from the fact that platinum is an ideal model catalyst for studying the mechanism, while the lead electrode represents the applied part of the work, where the current density, temperature, and sulfuric acid concentration are optimized to achieve a high current efficiency on a practical lead electrode.

After studying the mechanism of oxidation by voltammetric method, we studied the electrochemical behavior of phosphorous acid by electrolysis. An electrolyte solution was prepared from a specific amount of phosphorous acid, and electrolysis was performed under specified conditions. Phosphorous acid oxidation on solid electrodes was carried out in a thermostatted 100-mL glass electrolyzer without separating the anodic and cathodic spaces under galvanostatic conditions (Figure 1).

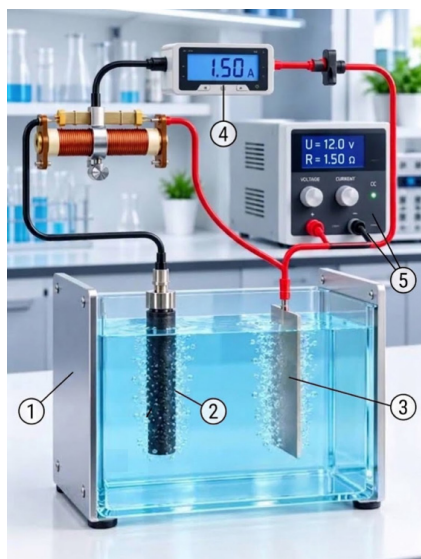


Figure 1 – Schematic diagram of the electrochemical oxidation of phosphorous acid: 1 – electrolyzer body, 2 – cathode (graphite rod), 3 – anode (lead electrode), 4 – ammeter, 5 – current source.

To isolate the fundamental kinetic features of the anodic process and avoid the masking effects of heavy metals and organic impurities typical for real industrial sludges, synthetic model solutions of H_3PO_3 were prepared in a sulfuric acid (H_2SO_4) supporting electrolyte for the laboratory-scale experiments. To validate the scalability and applicability of the developed approach under industrial conditions, pilot-scale trials were subsequently performed using real thermal sludge phosphoric acid directly at the production site of “NDFZ” LLP (Novodzhambul Phosphorous Plant). In the latter case, the real thermal phosphoric acid itself acted as the multi-component technological electrolyte undergoing anodic self-purification.

After electrolysis, the phosphate-ions formed were determined by using the known photocolorimetric method (Moyzhes, 1973). Orthophosphates react with vanadium and molybdenum salts to form yellow compounds with the composition $\text{P}_2\text{O}_5 \cdot \text{V}_2\text{O}_5 \cdot 22\text{MoO}_3 \cdot n\text{H}_2\text{O}$. Photocolorimetric measurements were performed on a KFK-3 device at a wavelength of 510-540 nm. Maximum light absorption coefficients of $E = 20,000$ ($\lambda = 315$ nm) and $E = 2,500$ ($\lambda = 400$ nm) indicate peak intensity in a 0,5–1,2 N nitric acid solution; the color develops and remains stable for several days. Small amounts of Si, NH_4 , Ca, Ba, Ni, Bi, Mo, and As do not affect the accuracy of the determinations.

Solution A. Dissolve 50 g of ammonium molybdate in 500 ml of water, heat to 50-60°C. (If vanadate turns yellow during dissolution, it is necessary to add a few drops of ammonia). Filter the solution, cool and add 250 ml of HNO_3 1:3.

Solution C. Molybdenovanadium reagent. Solution A is slowly poured into solution B with vigorous stirring, add another 50 ml of concentrated HNO_3 and mix thoroughly. The solution is stable.

Solution D. The standard solution of potassium dihydrogen phosphate 0,4794 g of KH_2PO_4 dried over sulfuric acid is placed in a volumetric flask (1L), dissolved in water, and the volume is brought up to the mark; (1 ml of solution contains 0.00025 g of P_2O_5).

Construction of calibration graph. 10, 14, 18, 22, 26, 30 or 20, 24, 28, 32, 36, 40 ml of standard solution are placed in measuring flasks with a volume of 100 ml. 5 ml of HNO_3 (concentrated), 70 ml of H_2O and 20 ml of solution B are poured into each flask, the volume is brought up to the mark with water, and after 20-30 minutes, colorimetry is carried out on FEK-N-57 with light filter No. 3, FEK-56 with CH-98 lamps at $\lambda=410\text{-}430$ nm in cuvettes with a length of 10 mm. Measurements were made relative to the reference solution for the first calibration graph containing 10 mm, for the second 20 mm of P_2O_5 solution. According to the obtained results, calibration graphs are built, which allow to determine 2,5-7,5 and 5-10 mg of P_2O_5 .

The analysis was carried out according to the linear dependence of the concentrations of phosphate-ions on the optical density of the solution ($D=f(C)$).

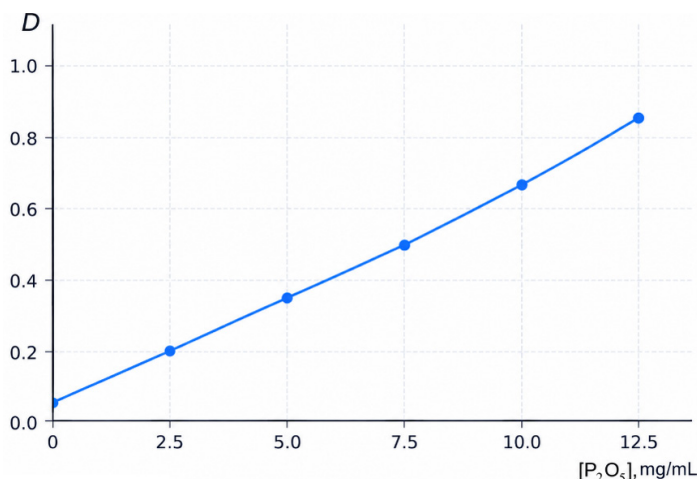


Figure 2 – Calibration graph in coordinates: optical density - concentration P_2O_5 (mg/ml).

The phosphate-ion content according to the calibration curve is determined by this formula:

$$C(\text{PO}_4^{-3}) = \frac{A(\text{PO}_4^{-3}) \cdot W}{1000 \cdot \alpha} \quad (1)$$

where, $A(\text{PO}_4^{-3})$ is the PO_4^{-3} fraction found from the calibration curve; W is the fraction of the main solution, ml; α is the aliquot part of the solution, ml.

Formula for calculating phosphine oxidation (n , %):

$$n = \frac{C(\text{PO}_4^{-3})}{C_{\text{total}}} \cdot 100\% \quad (2)$$

where $C(\text{PO}_4^{-3})$ is the phosphate-ion content formed in the solution after electrolysis, mg; C_{total} - total phosphine content calculated based on phosphate-ions, mg.

To determine the current efficiency, the theoretical mass was calculated using Faraday's law:

$$m_{\text{theor.}} = I * q * \tau \quad (3)$$

where I is the current, A; q is the electrochemical equivalent, g/A·h τ is time, h. The current efficiency was calculated using the following formula:

$$CE = m_{\text{pract}}/m_{\text{theor.}} * 100\% \quad (4)$$

The influence of the current density, electrolyte concentration, and duration of electrolysis on the current efficiency of phosphorous acid oxidation has been studied.

Statistical Data Processing. To ensure the reproducibility of the electrochemical results, all measurements (including current efficiency tests) were carried out at least three times under identical conditions. Statistical analysis was performed using the standard small-sample methodology. Data are presented as the mean (+) standard deviation (SD) or confidence interval (CI) calculated for a 95% confidence level ($P = 0,95$). Data visualization, curve fitting, and error bar calculations were performed using OriginPro 2024. The differences between the parameters were considered statistically significant if their 95% confidence intervals did not overlap.

Results. Phosphorous acid (H_3PO_3) is an important chemical compound with unique redox properties, making it an object of interest for electrochemical research.

The pioneering studies were conducted on a potentiostat-galvanostat with the ability to record cyclic voltammograms. A platinum electrode with a carefully prepared polished surface served as the working electrode. The auxiliary electrode was a graphite wire, and a silver chloride electrode was used as the reference electrode.

A solution of phosphorous acid with a concentration of 5 g/L was used for the experiments, prepared in a 0,5 N sulfuric acid solution, which provided an acidic environment and stable ionic strength. The polarization curves were recorded by cyclic voltammetry in the potential range from -0,5 V to +1,5 V at a potential sweep rate of 50 mV/sec.

To unambiguously differentiate the currents associated with (H_3PO_3) oxidation from the background processes on the platinum surface, a control experiment was performed in the pure supporting electrolyte (Fig. 3, curve 1). As can be seen, the blank voltammetric curve exhibits no peak features in the potential range of 0,5 to 1,2 V, indicating that platinum oxide formation contributes negligibly to the overall current in this region. Consequently, the two distinct anodic peaks observed at a potential of approximately +0.7 V and a potential of approximately +0,95 V upon the addition of H_3PO_3 (curve 2) can be confidently assigned to the stepwise electro-oxidation of phosphorous acid (Figure 3). The first peak appears at a potential of $E_{p1} = 0.70\text{V}$ with a corresponding

current density of $j_{p1} = -4.2 \cdot 10^{-5} \text{ A/cm}^2$. The second oxidation peak is located at $E_{p2} = 0,95\text{V}$ with a current density of $j_{p2} = -2.7 \cdot 10^{-5} \text{ A/cm}^2$. This quantitative separation confirms a two-step mechanism for the process.

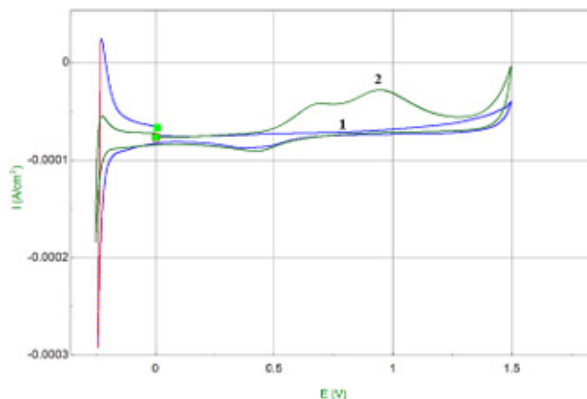


Figure 3 – Cyclic anodic-cathodic potentiodynamic polarization curves of a platinum electrode in a solution containing:
1–0,5 N sulfuric acid (blue line); 2–0,5 N sulfuric acid + 5 g/L phosphorous acid (green line); potential sweep rate: 50 mV/sec

The reduction peaks were less pronounced, indicating possible irreversible or partially reversible reaction behavior. The recorded cyclic voltammogram also showed reduction, indicating the occurrence of electrochemical reactions of phosphorous acid on the surface of the platinum electrode. An analysis of the reverse scan of the cyclic voltammogram (Fig. 3, curve 2) reveals a total absence of cathodic peaks in the potential range from 1,5 to 0 V, which would correspond to the reduction of the phosphorous acid oxidation products. The absence of a measurable cathodic peak current ($j_{pc} = 0$) precludes the calculation of standard reversibility criteria, such as the peak-to-peak separation (ΔE_p) and the peak current ratio ($|j_{pa}/j_{pc}|$). Therefore, the experimental data strictly demonstrate that the electrochemical oxidation of H_3PO_3 on the Pt electrode is a completely irreversible process.

The effect of the potential scan rate on the electrochemical behavior of phosphorous acid on the platinum electrode was systematically investigated (Figure 4). As can be seen from the cyclic voltammograms, an increase in the potential scan rate from 10 to 300 mV/sec leads to a continuous increase in the peak currents for both anodic oxidation steps of phosphorous acid. To identify the nature of the rate-determining step, diagnostic plots of the peak current density (j_p) as a function of the scan rate parameters were analyzed. The dependencies of j_p versus $\nu^{1/2}$ exhibited a strict linear correlation ($R^2 > 0,99$) for all six scan rates. Furthermore, the linear regression of $\log j_p$ vs. $\log \nu$ yielded slopes of 0,51 for Peak 1 and 0,49 for Peak 2. These values perfectly match the theoretical diagnostic criterion of 0,5, unambiguously demonstrating that the electro-oxidation of H_3PO_3 is entirely governed by diffusion control rather than surface kinetic limitations.

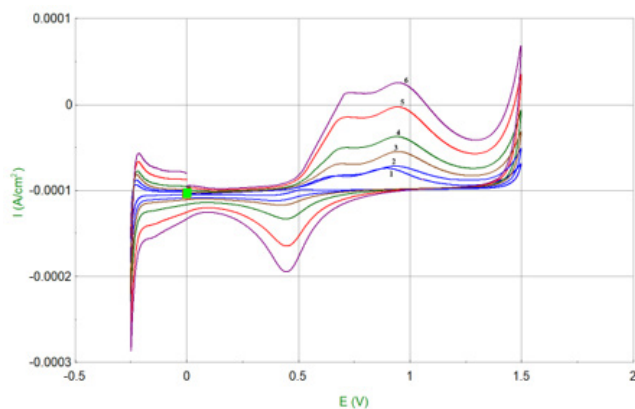
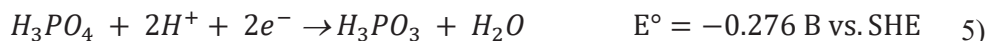


Figure 4 – Anodic-cathodic polarization curve of phosphorous acid in a sulfuric acid solution on a platinum electrode at different potential scan rates: 1–10, 2–30, 3– 50, 4–100, 5–200, 6–300 mV/sec.

After studying the mechanism of electrochemical oxidation by voltammetric method, we continue the research experiments by electrolysis. Galvanostatic electrolysis is characterized by the supply of direct current, which ensures stable and controllable reaction conditions. This allows for a detailed study of the kinetics of the process, determination of optimal parameters, and understanding of the mechanisms of the reactions taking place. The aim of this galvanostatic research is to study the anodic oxidation of phosphorous acid by electrolysis and to investigate the influence of the main factors such as current density, temperature, and electrolyte concentration on the rate of phosphorous acid oxidation.

Anodic oxidation of phosphorous acid is an important electrochemical process that converts H_3PO_3 into phosphoric acid (H_3PO_4). The electrochemical oxidation of phosphorous acid occurs on the surface of the lead anode and allows for focusing on the oxidation process (Tukibayeva, 2025).

During the anodic oxidation of phosphorous acid, the following reaction can occur on the electrode surface:



The influence of electrochemical factors such as current density, temperature, and electrolyte concentration on the current efficiency of phosphorous acid oxidation was studied.

Our experimental data show that as current density increases from 100 to 500 A/m², the oxidation rate of phosphite ions is initially low (Figure 5). All experimental studies to determine current efficiency were performed in triplicate (n = 3).

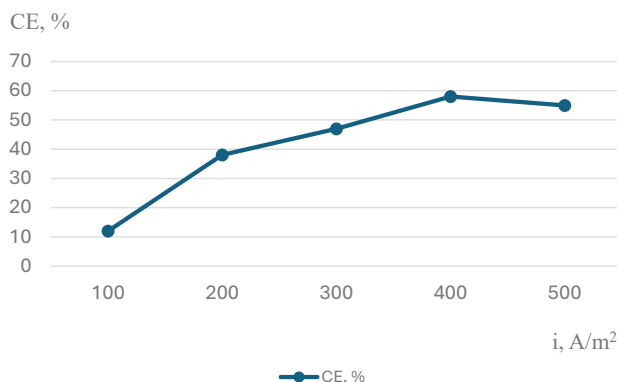


Figure 5 – Effect of current density on the current efficiency of phosphorous acid oxidation:
 $[\text{H}_2\text{SO}_4]$ -100 g/L, $[\text{H}_3\text{PO}_3]$ – 30 g/L; τ

Figure 5 presents the calculated values for standard deviations (SD) and confidence intervals (ΔX), computed using Student's distribution at a confidence level of $P = 0,95$ (standard for chemical research). Vertical error bars are plotted on the graphs. The statistical analysis fully confirms the validity of selecting the optimal operating mode (400 A/m²; current efficiency: 59,0 ± 2,5%). The confidence intervals for this point do not overlap with those of adjacent modes (300 A/m²: 49,3 ± 3,8%; 500 A/m²: 54,0 ± 2,5%), thereby mathematically demonstrating the reliability and statistical significance of the observed maximum and ruling out the influence of random factors.

Table 1 – Effect of current density on the current efficiency of phosphorous acid oxidation:
 $[\text{H}_2\text{SO}_4]$ -100 g/L, $[\text{H}_3\text{PO}_3]$ – 30 g/L; τ

$i, \text{A/m}^2$	100	200	300	400	500
$\text{CE, \%}$	11,0 ± 5,0%	39,3 ± 3,8%	49,3 ± 3,8%	59,0 ± 2,5%	54,0 ± 2,5%

We hypothesize that this is due to insufficient electrode potential for efficient anodic reaction. With increasing current density to 400 A/m², a sharp increase in the oxidation rate and, correspondingly, the current efficiency of phosphorous acid oxidation was observed. With further increases in current density, the reaction rate slows down, which is explained by competition with side reactions, such as oxygen evolution.

An investigation of the effect of temperature on current output revealed a monotonic increase in the target value across the entire range studied, from 20 to 60 °C (Table 2).

Table 2 – Effect of electrolyte temperature on the current efficiency of phosphorous acid oxidation:
 $[\text{H}_3\text{PO}_3]$ -30 g/L; $i = 400 \text{ A/m}^2$; $\tau = 0,5 \text{ h}$

$T, ^\circ$	20	30	40	50	60
$\text{CE, \%}$	21,0 ± 5%	24,7 ± 3,8%	34,0 ± 2,5%	50,3 ± 3,8%	60,7 ± 3,8%

Solution temperature directly influences the anodic oxidation process of phosphorous acid. Increasing the temperature accelerates the transport of reactants to the anode, reduces concentration polarization, and increases the rate of the main target reaction. In

our case, increasing the electrolyte temperature from 20 to 60 °C favorably affects the rate of the anodic process.

Statistical analysis of the data ($n = 3$, $P = 0.95$) confirmed the high reproducibility of the results. With the exception of the 20–30 °C range, where the confidence intervals partially overlap ($21,0 \pm 5,0\%$) and ($24,7 \pm 3,8\%$), respectively, a further increase in temperature leads to a statistically significant (reliable) increase in current output. The maximum value is reached at 60 °C and amounts to ($60,7 \pm 3,8\%$); the lower limit of this regime (56,9%) is strictly higher than the upper limit of the regime at 50 °C (54,1%), which mathematically confirms the selection of this temperature as the optimal one.

When studying the concentration of sulfuric acid solution, anodic oxidation of phosphorous acid initially proceeds at a high rate, and a decrease in the rate of the anodic process is observed when the sulfuric acid concentration reaches 150 g/L (Figure 6). According to the laws of electrochemistry, at high sulfuric acid concentrations, the solution viscosity may increase and ion mobility may decrease, which may impair the transport of phosphorous acid to the anode. Also, as the concentration of sulfate-ions in the electrolyte increases, a very dense layer of lead sulfate with a low solubility product ($SP(PbSO_4) = 2 \cdot 10^{-8}$) forms on the surface of the electrode (Dickerson et al., 1982), which reduces the amount of current passing through the circuit:

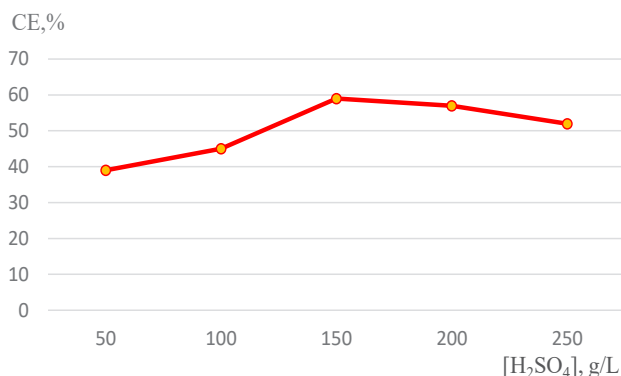


Figure 6 – Effect of sulfuric acid concentration on the current efficiency of phosphorous acid oxidation: $[H_3PO_3] - 30 \text{ g/L}$; $i = 400 \text{ A/m}^2$; $\tau - 1.0 \text{ h}$

At high current densities, a darkening of the lead anode surface to a brownish-purple color is also observed, indicating that the reaction proceeds through lead dioxide. From a technological perspective, when considering this electrochemical approach as a basis for phosphoric acid purification, the potential contamination of the final product with toxic heavy metal ions must be carefully evaluated. Although direct analytical determination of lead was beyond the scope of this fundamental study, a rigorous thermodynamic

estimation can be applied. In a strongly acidic medium containing an excess of sulfate ions from the supporting electrolyte, the accumulation of free Pb^{2+} is strictly prevented by the immediate precipitation of PbSO_4 ($K_{\text{sp}} = 1,6 \cdot 10^{-8} - 2,0 \cdot 10^{-8}$). Due to the common-ion effect, the residual concentration of dissolved Pb^{2+} is theoretically limited to less than $4,0 \cdot 10^{-8} \text{ M}$ ($\sim 6,6\text{-}8,3 \text{ ppb}$). This guarantees that the contamination of the purified acid remains far below the critical thresholds for industrial applications, confirming the viability of the proposed method.

Table 3 – Effect of sulfuric acid concentration on the current efficiency of phosphorous acid oxidation: $[\text{H}_3\text{PO}_3] = 30 \text{ g/L}$; $i = 400 \text{ A/m}^2$; $\tau = 1,0 \text{ h}$

$[\text{H}_2\text{SO}_4], \text{ g/L}$	50	100	150	200	250
CE, %	$40,0 \pm 2,5\%$	$42,0 \pm 2,5\%$	$59,7 \pm 3,8\%$	$58,7 \pm 3,8\%$	$52,3 \pm 3,8\%$

The dependence of current efficiency on sulfuric acid concentration exhibits an extremum, with a maximum at 150 g/L. Statistical analysis of the data ($n = 3$, $P = 0,95$) confirms that increasing the concentration from 50 to 150 g/L results in a significant rise in current efficiency to $(59,7 \pm 3,8\%)$. In the 150–200 g/L range, the values remain stable within the limits of experimental error. However, a further increase in concentration to 250 g/L leads to a statistically significant decrease in the value to $(52,3 \pm 3,8\%)$, thereby mathematically justifying the selection of 150 g/L as the optimal process parameter.

Discussion. The observed two pronounced oxidation peaks in the anodic direction on the cyclic voltammograms show the anodic oxidation proceeding, which corresponds to the oxidation of phosphorous acid with the probable formation of phosphoric acid or other oxidized forms. This research requires further detailed investigation.

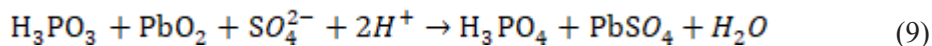
The obtained results show that anodic oxidation of phosphorous acid is electrocatalytic. The mechanism of anodic oxidation of phosphorous acid on a lead anode (Pb) has features that distinguish it from processes on inert anodes such as platinum or graphite. A lead anode is a passivated anode that can participate in reactions not only as a current conductor but also as a reactive surface, forming oxide layers (PbO_2 , etc.). Similar results were obtained during the study of anodic oxidation of hypophosphite-ions in neutral solutions (Tukibayeva et al, 2022).

Based on the observed characteristic dark-brown coloration of the lead anode surface during electrolysis, the formation of a lead dioxide (PbO_2) phase can be hypothesized. In strongly acidic media at high anodic potentials, the electrochemical transition of lead to (PbO_2) is thermodynamically favorable. Therefore, we propose a tentative mechanism where (PbO_2) acts as an in situ generated mediator that facilitates the oxidation of phosphite-ions.



The electrochemical oxidation of phosphorous acid occurs through chemical oxidation of the phosphorous acid by a layer of PbO_2 formed on the anode surface during anodic polarization and direct electrooxidation on the surface of the lead anode.

During the oxidation of phosphorous acid, chemical oxidation occurs on the lead anode surface:



It should be noted that while the electrochemical data point toward a probable catalytic role of the lead oxide surface layer, its precise phase composition, morphology, and chemical state remain to be confirmed. Detailed surface characterization using XRD, XPS, and SEM/EDS techniques will be the focus of our future studies to comprehensively unravel the reaction mechanism.

Thus, we have studied the anodic oxidation of phosphorous acid on a lead electrode in a sulfuric acid solution. The current efficiency of phosphorous acid oxidation in a sulfuric acid solution on a lead electrode under the preferred operating parameters ($[\text{H}_2\text{SO}_4] = 150 \text{ g/L}$, $i = 400 \text{ A/m}^2$, $t = 60^\circ\text{C}$) is 71%.

The industrial viability of the developed electrochemical oxidation method was successfully validated through pilot-scale trials on real thermal sludge phosphoric acid directly at the “NDFZ” LLP production site. The highly efficient performance of the lead anode under optimized regimes (400 A/m^2 , 60°C) and the obtained technical parameters were officially confirmed by the Testing Act issued by “NDFZ” LLP.

Conclusions

In this work, the electrochemical behavior of phosphorous acid was studied using voltammetric measurements. The cyclic voltammograms shows two pronounced oxidation peaks in the anodic direction at a potential of approximately +0,56 to +0,7 V on platinum electrode, which corresponds to the oxidation of phosphorous acid with the probable formation of phosphoric acid or other oxidized forms. These studies require further detailed investigation.

It was investigated the influence of the main factors such as current density, temperature, and electrolyte concentration on the rate of phosphorous acid oxidation under galvanostatic conditions. The results show that during anodic oxidation of phosphorous acid, oxidation occurs to phosphate-ions. The oxidation of phosphite-ions can be considered electrocatalytic, with the catalyst being the oxide layers formed on the electrode surface. It has been shown that the oxide layer formed on the surface of a lead electrode has a catalytic effect and positively influences the oxidation of phosphite ions. Electrode materials play a key role in ensuring high catalytic activity and efficient charge transfer during phosphorous acid oxidation.

The practical significance of this work lies in the scientific substantiation and selection of optimal process parameters for the anodic oxidation of phosphorous acid. Replacing expensive noble metals with a lead anode—which is both cost-effective and corrosion-resistant in sulfuric acid media - ensures a high current efficiency for the target product. The proposed electrochemical method for purifying thermal phosphoric acid was tested at the NDFZ LLP production facility (Taraz). The test results confirm the feasibility of electrochemically removing lower phosphorus acids from thermal phosphoric acid at ambient temperatures and demonstrate the applicability of the developed method.

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