30th International Conference Ecological Truth & Environmental Research 2023

Proceedings

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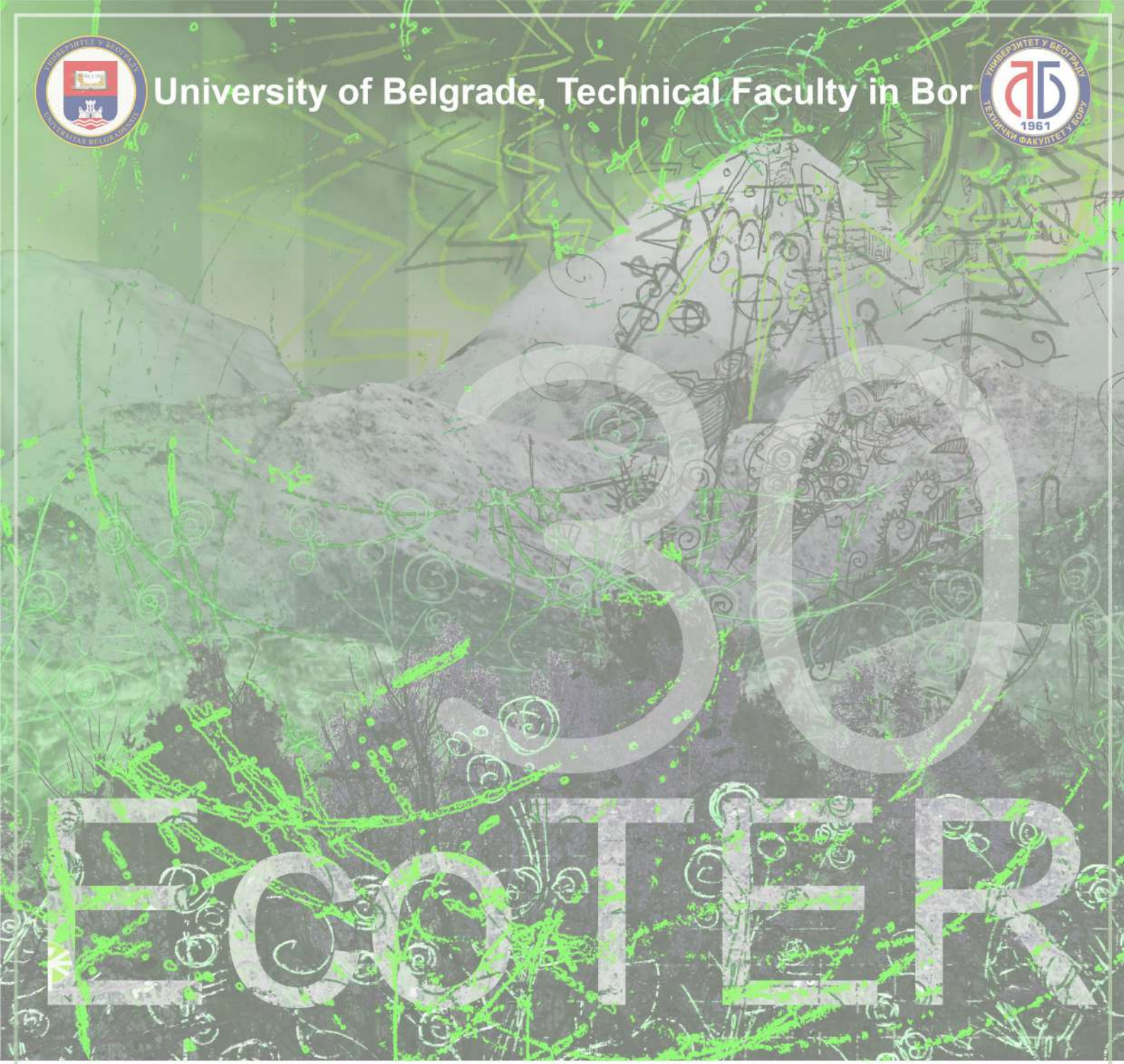

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PROCEEDINGS

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ECOLOGICAL TRUTH AND ENVIRONMENTAL RESEARCH – EcoTER'23

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PREFACE

The 30th international conference Ecological Truth & Environmental Research – EcoTER'23 kept three areas in focus: ecology, environmental protection and sustainable development. The conference will be held on Mt Stara Planina in hotel Stara Planina, Serbia, 20–23 June 2023. The monograph is published on the occasion of the 30th anniversary of the conference. On behalf of the scientific and organizing committee, it is a great honor and pleasure to wish all the participants a warm welcome to the conference.

The monograph is published on the occasion of the 30th anniversary of the conference.

We hope to convey the message of the conference, which is that a transformation of attitudes and behavior would bring the necessary changes. This is also an opportunity for the participants who are experts in this field to exchange their experiences, expertise and ideas, and also to consider the possibilities for their collaborative research.

The 30th international conference Ecological Truth & Environmental Research – EcoTER'23 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology, the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica, the University of Zagreb, Faculty of Metallurgy – Sisak, the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Association of Young Researchers, Bor.

These Proceedings 103 papers from the authors coming from the universities, research institutes and industries in 11 countries: Australia, USA, Brazil, Spain, Portugal, Libya, Italy, Bulgaria, Bosnia and Herzegovina, North Macedonia, and Serbia.

As a part of this year's conference, the 5th Student Session – EcoTERS'23 is being held. We appreciate the contribution of the students and their mentors who have also participated in the conference.

The support of the Gold donor and their willingness and ability to cooperate has been of great importance for the success of the EcoTER'23. The organizing committee would like to extend their appreciation and gratitude to the Gold donor of the conference for their donation and support.

We appreciate the effort of all the authors who have contributed to these Proceedings. We would also like to express our gratitude to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the conference participants for their support to the EcoTER'23. Sincere thanks go to all the people who have contributed to the successful organization of the EcoTER'23.

Prof. Snežana Šerbula,

President of the scientific and organizing committee

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MECHANISM AND KINETICS OF ELECTROCATALYTIC OXIDATION OF PHENOL

Sonja Stanković[1*], Vladan Nedelkovski[1], Milan Radovanović[1], Snežana Milić[1]
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Abstract

Phenols are toxic, carcinogenic, organic compounds present in the wastewater of numerous industrial facilities. Improper disposal of industrial wastewater poses a risk to the environment and human health, as the presence of phenolic compounds, even in low concentrations, can cause a range of harmful effects. In order to protect the environment and preserve human health, it is necessary to remove phenol and its derivatives from industrial wastewater before their release into the environment. Since the use of conventional methods in the treatment of phenolic wastewater is limited by low efficiency and high costs, in recent years there has been an increasing interest in the application of advanced oxidation processes in wastewater treatment. Among the numerous advanced oxidation processes, electrocatalytic oxidation has attracted a lot of attention due to its practicality and high efficiency. This paper presents the mechanism of electrocatalytic oxidation of phenol in aqueous solutions, as well as a literature review of the influence of process parameters on phenol oxidation.

Keywords: phenol, electrooxidation, applied potential, pH, initial concentration.

INTRODUCTION

Phenol is one of the most dangerous organic pollutants, which by releasing industrial wastewater, ends up in aquatic ecosystems [1–3]. The maximum allowable concentration of phenolic compounds in industrial wastewater is 2 mg/dm^3. However, the wastewater from numerous industrial facilities contains phenolic compounds in significantly higher concentrations than allowed (50-2000 mg/dm^3) [4]. 80% of wastewater, of which 28% is industrial wastewater, are discharged into the environment without any kind of prior treatment. Because of that, contamination of aquatic ecosystems represents a growing problem worldwide [5–7].

The presence of phenolic compounds in water is harmful to the environment and human health and can lead to a range of unwanted symptoms and health problems [4,8,9]. According to the Environmental Protection Agency (EPA), short-term exposure to water containing phenols, can cause skin, eye and mucous membrane irritation, while long-term exposure could eventually lead to bleeding and liver damage [10]. In addition, phenol can cause damage to the central nervous system, kidney disease, and the formation of various tumors. Therefore, the removal of phenol and its derivatives is significant in protecting the environment and preserving human health [11].

Conventional methods such as biological degradation, chemical oxidation, and adsorption can be used for purifying phenolic wastewater [7,12]. However, wastewaters containing high concentrations of phenol (>5 mg/dm^3) cannot be treated by biological methods due to their toxicity towards microorganisms [13]. The use of traditional methods such as adsorption on activated carbon and chemical oxidation can achieve phenol degradation, but their application in the process of purifying industrial wastewater is limited by high costs [2,13].

Due to their practicality and high efficiency, Advanced Oxidation Processes (AOPs) are attracting lots of attention from researchers. Unlike conventional methods, use AOPs can achieve complete degradation of phenol and its derivatives. In addition, the use of advanced oxidation processes in the treatment of wastewater does not result in secondary pollution [14]. Advanced oxidation processes include electrocatalytic oxidation, photocatalysis, ozonation, Fenton reaction and enzymatic treatment.

Electrocatalytic oxidation is an environmentally friendly and efficient method, which is based on redox reactions of organic compound and strong oxidants [9,12]. By using this method, complete oxidation of organic matter to CO_2 and H_2O can be achieved [9]. This paper presents the mechanism and kinetics of electrocatalytic oxidation of phenol.

MECHANISM OF ELECTROCATALYTIC OXIDATION OF PHENOL

A large number of research teams have investigated the mechanism of electrochemical oxidation of phenol and its derivatives [3,11,15–18]. The mechanism of phenol degradation is shown in Figure 1. The oxidation of phenol begins with electron transfer, which leads to radical reactions [18]. Free-radical reactions result in the formation of benzoquinone. It is assumed that benzoquinone is the main intermediate compound in the process of phenol oxidation [17–19]. Hydroxyl radicals generated by the electrolysis of water react with benzoquinone, leading to the opening of the aromatic ring and the formation of maleic acid and ethene. The formed maleic acid is reduced to succinic acid (reaction pathway IIIa) at the cathode, followed by the oxidation of succinic acid to malonic acid, further to acetic acid, and finally to CO_2. In parallel with the reduction of maleic acid to succinic acid, the oxidation of maleic acid to oxalic acid via hydroxyl radicals takes place (reaction pathway IIIb). The formed oxalic acid is easily oxidized to CO_2 [18]. Tasić *et al.* [3] emphasize that the reaction of phenol oxidation is controlled by the diffusion of phenol molecules on the surface of the anode. However, Li *et al.* [18] emphasize that the rate of phenol degradation is controlled by the rate of opening of the aromatic ring and the oxidation of maleic acid.

In the absence of strong oxidizing free radicals, the accumulation of hydroquinone and benzoquinone occurs, leading to the formation of a layer of polymeric compounds (reaction pathway II) that blocks active sites on the surface of the anode, preventing further oxidation of phenol [3,18]. Polymerization and aromatic ring opening reactions often occur simultaneously. If the aromatic ring opening reaction and formation of carboxylic acids are faster than polymerization, a small amount of benzoquinone and polymeric compounds will be formed on the surface of the anode. Nevertheless, if polymerization is faster than the aromatic ring opening reaction and formation of carboxylic acids on the surface of the anode, a significant amount of polymeric compounds will be formed [18].

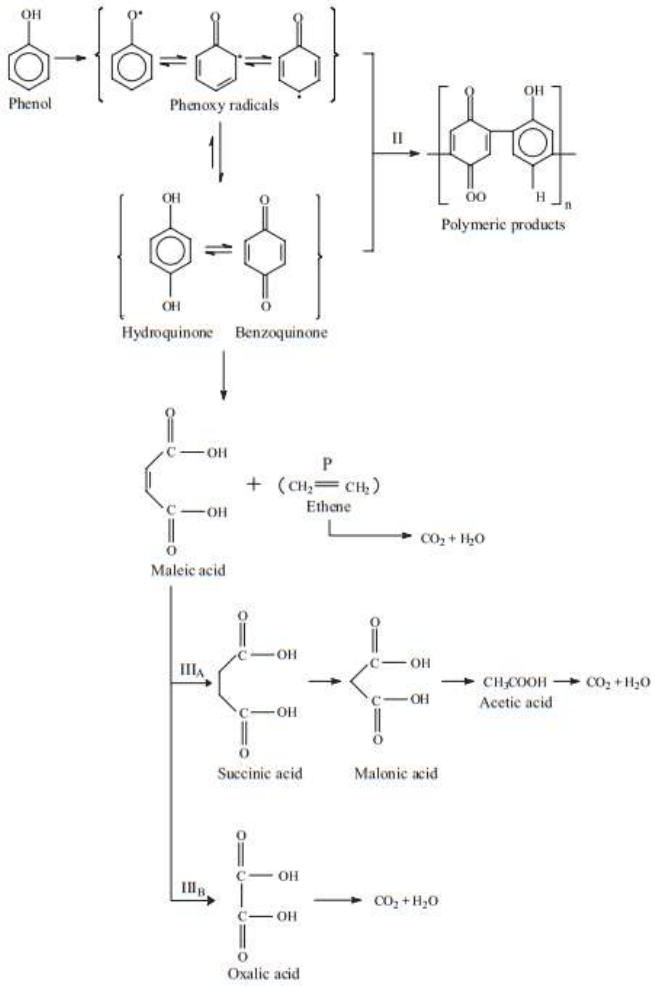

Figure 1 *Mechanism of electrocatalytic phenol degradation [18]*

KINETIC PROCESS PARAMETERS

Understanding the impact of process parameters is of great importance in selecting a sustainable and efficient method for treating phenolic wastewater. The mechanism and kinetics of phenol oxidation depend on a number of variables such as electrolyte pH, initial phenol concentration, current density, anodic material, electrolyte flow rate through the electrochemical cell, etc. [6].

Current density

Current density is the most important operating parameter that affects the efficiency and speed of electrocatalytic oxidation of phenol [11,17,19–23].

Zhu *et al.* [17] investigated the effect of current density (10–50 mA/cm^2) on the electrocatalytic degradation of phenol using a Ti/Sb_2O_3-SnO_2 anode and a stainless-steel cathode. Experimental results indicate that the efficiency of electrocatalytic phenol degradation in acidic, neutral, and basic media increases with increasing current density. After 12 hours of electrolysis, nearly 100% TOC removal was achieved at a current density of 40 mA/cm^2. Loloi *et al.* [19] confirmed in their work that increasing current density from 10 to 40 mA/cm^2 leads to an increase in phenol degradation efficiency. Ghanim and Juda [11]

investigated the effect of current density (30, 50, and 70 mA/cm^2) on the electrocatalytic degradation of phenol using a graphite and β-PbO2 anode. The highest degree of phenol degradation of 74.4% for graphite and 83.7% for β-PbO_2 were achieved at a current density of 70 mA/cm^2, an initial phenol concentration of 100 mg/dm^3, and a flow rate of 12 L/h.

Gao *et al.* [21] studied the impact of higher current densities (100-700 mA/cm^2) on the rate and efficiency of phenol degradation (500 mg/dm^3) using a Ti/IrO_2-Ta_2O_5 anode. Increasing the current density promotes electron transfer and •OH generation on the electrode surface, resulting in increased efficiency degradation of phenol and intermediates. However, higher current density leads to greater energy consumption. Gao *et al.* [21] point out that the optimal current density for electrocatalytic phenol degradation using a Ti/IrO_2-Ta_2O_5 anode, would be 500 mA/cm^2.

Initial concentration

In terms of practical application, of core research importance is to understand the influence of initial phenol concentration on removal efficiency and electrocatalytic oxidation kinetics [24]. The literature review of the impact of the initial concentration on the efficiency of electrocatalytic phenol degradation is provided in Table 1.

Table 1 *The impact of initial phenol concentration on the efficiency of electrocatalytic degradation*

Electrode	Current density (mA/cm^2)	Initial concentration (mg/dm^3)	Removal efficiency (%)	Reference
Ti/SnO_2-Sb_2O_5	20	50	96	[19]
		200	72	
Graphite	70	100	74.7	[11]
		500	58.9	
PbO_2	70	100	83.7	[11]
		500	74.3	
Ti/TiO_2NTs/NiO/Ce-PbO_2	40	50	98.1	[24]
		250	70	
SnO_2-Sb_2O_3/Ti	40	460	98.7	[17]
		550	97.2	
		620	95.4	
		710	94.3	

Based on the reviewed literature [11,17,19,23,24] it can be concluded that as the initial concentration of phenol increases, the efficiency of degradation decreases. As the initial concentration of phenol increases, the need for strong oxidizing agents (•OH) also increases. However, under constant testing conditions, the number of (•OH) formed on the surface of the electrode remains constant. In that case, the number of formed (•OH) is not sufficient to oxidize phenol at higher concentrations. Therefore, with the increase in pollutant concentration, at a constant current density, the efficiency of electrocatalytic phenol degradation decreases [19].

Gui *et al.* [25] investigated the effect of initial phenol concentration on the kinetics of electrocatalytic phenol oxidation. The results of the study are presented in Table 2 and indicate that the value of the reaction rate constant decreases with increasing concentration and that the phenol degradation reaction follows pseudo-first-order kinetics. Wang *et al.* [24], Kornienko *et al.* [26], Park *et al.* [27], Makgae *et al.* [28] and Zhu *et al.* [17] have also confirmed in their works that the electrocatalytic oxidation of phenol follows pseudo-first-order kinetics.

Table 2 *The effect of initial phenol concentration on the rate constant of phenol degradation [25]*

Electrode	Initial concentration (mg/dm^3)	k (min^{-1})
ZnO/Co(II)-PbO_2	50	$7.78 \cdot 10^{-3}$
	100	$7.69 \cdot 10^{-3}$
ZnO/PEG-Co(II)-PbO_2	50	$9.49 \cdot 10^{-3}$
	100	$9.17 \cdot 10^{-3}$
	460	$2.91 \cdot 10^{-3}$
	550	$1.67 \cdot 10^{-3}$
	620	$8.30 \cdot 10^{-4}$
	710	$6.67 \cdot 10^{-4}$

pH

Numerous research teams have investigated the effect of electrolyte pH on the efficiency of electrocatalytic oxidation of phenol [19,23,28–30].

Wu *et al.* [29] investigated the effect of electrolyte pH (4.68, 7.2, and 11) on the electrocatalytic degradation of phenol using a Ti/SnO_2-Sb_2O_5/PbO_2 anode. After 12 hours, complete phenol degradation was achieved only in an alkaline environment (pH 11). The results are consistent with those obtained by Loloi *et al.* [19] and Makgae *et al.* [28] and suggest that phenol degradation is favored in an alkaline environment. The high degree of phenol degradation in an alkaline environment can be attributed to the large amount of hydroxyl radicals formed according to reactions (1) and (2) [29]. The formed hydroxyl radicals participate in the oxidation of phenol.

$$2H_2O - 2e^- \rightarrow 2^{\cdot}OH + 2H^+ \quad (1)$$

$$OH^- - e^- \rightarrow {}^{\cdot}OH \quad (2)$$

Kornienko *et al.* [23] investigated the kinetics of the indirect oxidation of phenol mediated by reactive oxygen species on a Pb/PbO_2 anode in acidic and neutral media. The results of the study are presented in Table 3 and indicate that a higher rate and efficiency of phenol degradation are achieved in acidic than in neutral media. The oxidation reaction of phenol follows pseudo-first-order kinetics.

Table 3 *The effect of pH on the rate and efficiency of electrochemical oxidation of phenol (Co=200 mg/*dm^3*) [23]*

pH	Current density (A/m^2)	Oxidation efficiency (%)	Rate reaction constant k (h^{-1})
2–3	500	84	1.4
	1000	92	1.8
7	500	48	0.66
	1000	77	1.47

The positive effect of low pH on the rate of phenol degradation was confirmed by Li *et al.* [31], Gao *et al.* [21] and Loloi *et al.* [19]. Li *et al.* [31] point out that H^+ ions promote the formation of H_2O_2 and •OH and inhibit the release of oxygen. In an acidic environment, phenol is adsorbed on the surface of the electrode and donates an electron to the anode, forming a phenoxy radical, which is oxidized in the presence of hydroxyl radicals (Loloi et al., 2016).

CONCLUSION

Electrocatalytic oxidation is an efficient and environmentally friendly technology suitable for treating phenolic wastewater. Using this method, complete oxidation of phenolic compounds to CO_2 and H_2O can be achieved. The efficiency and rate of electrocatalytic oxidation of phenol depend on the nature of the anodic material, current density, pH value of the electrolyte, initial phenol concentration, and flow rate of the electrolyte through the electrochemical cell.

- Current density is one of the most important factors that affect the efficiency and rate of electrocatalytic degradation of phenol. Increasing the current density increases the rate of electrocatalytic degradation of phenol. However, it is necessary to consider the economic feasibility of the process, as the use of high current densities requires additional costs.
- Increasing the initial concentration of phenol can negatively affect the efficiency of electrocatalytic oxidation.
- The pH value of wastewater can greatly affect the efficiency and rate of electrocatalytic oxidation of phenol. The highest degree of phenol degradation is achieved in an alkaline, then acidic and neutral environment.

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