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Phenol Removal from Refinery Wastewater by Electrochemical Oxidizing Using Catalytic Basket Electrochemical Reactor with Rotating Anode

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Abstract

Phenol is highly toxic even if it is present in very low concentrations. Industrial wastes from petroleum refineries generally contain phenol which must be treated. In situations when conventional techniques are unable to control phenol degradation, the electrochemical oxidation process has emerged as a solution to this problem. This study examines the effectiveness of a pilot electrochemical system that removes phenol from refinery wastewater using a rotating anode-induced basket electrochemical novel reactor technique. A rotary anode was used to increase the efficiency of the process and was also used (10%CuO/ γ- Al_2O_3) as a catalyst. The experimental results showed that by increasing the rotating speed and increasing the voltage of the power supply of the electrochemical reactor, the phenol removal increased. It was also found that when the concentration of phenol increased, the removal rate was decreased.

Keywords: Phenol, Electrochemical, Refinery waste water, Removal.

1-Introduction

Seventy percent of the surface of the earth is made of water, which is believed to be a singular source of life.As a result of the scarcity of freshwater resources, the amazing rise in population, and the vast expansion of metropolitan areas that have occurred in recent years, there is a pressing need for the development of innovative approaches that can enhance the proportion of freshwater [1].mVarious industrial wastes have resulted in the introduction of organic pollutants into the water . These organic pollutants are primarily recalcitrant stable chemical compounds that have a relatively high level of toxicity[2].nThe seriousness of the problem of water pollution caused by harmful organic pollutants has increased in tandem with the fast rise of businesses. phenolic compounds,all wastewaters produced by industrial processes.These wastewaters include those produced by the petrochemical, coke oven, textile, rubber, paper, glass, and paint fields[3].

Because of their mutagenicity and carcinogenicity, even at low concentrations, these chemicals constitute a significant threat to human health as well as to the entire ecosystem when they are present.It is important to note that the concentration of phenol at 2 mg·L−1 is considered harmful to aquatic creatures. However, concentrations ranging from 10 to 100 mg·L−1 result in death within 96 hours.The World Health Organization (

WHO) put out the idea of a concentration threshold of 0.001 mg·L−1 for phenol consumption in drinking water.In Table 1, the levels of phenol that are present in a variety of industries are presented [4].

Table1. shows the levels of phenol that are present in a variety of industries [4].

Industrial	Phenol concentration / (mg·L^{-1})
Petrochemical	200–1220
Coke ovens	600–3900
Textile	100–150
Rubber	3–10
Paper	20–80
Glass	40–2564
paint	1.1

The majority of wastewaters have concentrations that are higher than the toxicity limit. Since this is the case, the removal of phenolic compounds from wastewater from industrial processes before to discharge is an important and pressing matter that needs to be investigated.[5]. The removal or elimination of phenolic compounds from wastewaters has been accomplished by the application of a variety of treatment procedures, including chemical oxidation processes. [6]. Distillation technology[7]. adsorption and extraction method[8]. Biological degradation[5]. Chemical oxidation[9].electrochemical oxidation[10][11].Enzymatic treatment[12] .Membrane technology[13].The electrochemical oxidation process has been demonstrated to have a significant amount of potential for the treatment of wastewater that contains phenolic compounds, according to previous studies. [14]. The electro-oxidation processes as a cost-effective technology [15]. are great for treating aromatic pollutants, They can also be used as a pretreatment to increase biodegradability or even for the complete mineralization of organic pollutants into carbon dioxide and water [16]. Nowadays, scientists interested in wastewater treatment are drawn to electrochemical methods because they are safe, selective, capable of automating repetitive tasks, and environmentally friendly. As a result, these methods have grown more compact and efficient [17]. In order to remediate soil, sludge, and other materials, electrochemical technologies have been widely used. [18], have been utilized as corrosion inhibitors, yet they do not have any negative impact on the environment. [19].and for the determination of pharmaceuticals [20]. The significance of electrochemical techniques lies in their capacity to promptly furnish valuable insights into oxidation-reduction cycles, heterogeneous reaction kinetics, and adsorption mechanisms [21].

By using electrochemical oxidation, a variety of species, including hydroxyl radicals (OH·), O2·, HO_2·, and ROO· that are produced in situ in the reaction media, can be destroyed. Water and oxygen are byproducts of the breakdown of hydrogen peroxide by a hydroxyl radical. It can react with a wide range of organic compounds, regardless of their concentration, thanks to its strong reduction potential (E(OH·/H_2O) = 2,8 V vs. SVE), which also enables it to oxidize almost all organic compounds to CO_2 and H_2O [22],or to simpler compounds such as acetic acid, oxalic acid and maleic acid, acetone, and chloroform [23].One method of producing hydroxyl radicals is to directly electrolysis water on the anode. [24].They have the capability of removing contaminants from all three forms gas, liquid, and solid in the environment. The non-selectivity of OH radicals plays a crucial role in preventing the development of by-products that are not desirable. Anodic oxidation occurs close to the surface of the electrode due to the short life cycle of OH radicals, which allows the process to occur. However, due to their exceptionally high oxidation capacity, they act as a catalyst for the production of additional oxidants that are present in the wastewater, which are comprised of a wide variety of chemical compounds. [23].

In this work, the progress that has been made to establish a better knowledge of the oxidation of phenol in refinery wastewater in a basket electrochemical reactor with a rotating anode, the rotating anode, and a cathode composed of stainless steel (ASTM 316) is presented. It enables the creation of hydroxyl radicals on-site through the electrolysis of water for the purpose of phenol oxidation. The scope of our evaluation comprised a wide variety of operating parameters, including applied voltages, beginning concentrations of phenol, revolutions per minute, and the amount of time required to achieve optimal removal.

Table 2: Summary of some previous studies.

pollutant	Cathode material	Anode material	Removal efficiency	Ref.
phenol	C-PTFE	platinum plate	100%	[25]
phenol	C-PTFE	Ti/IrO2/RuO2	100%	[26]
phenol	stainless steel	stainless steel	80%	[27]
phenol	graphite	TiO_2 / ACF	83.26%	[28]
phenol	Pt	Pt	93%	[29]

2.1 Chemicals and Analytical Procedure.

Phenol (C_6H_5OH, 99%) and KOH (85%) was supplied from Thomas Baker Company, India. It was used as an electrolyte. Prior to use, all compounds were diluted in high-purity deionized water and were of the reagent grade. Analyzing the removal of phenol by electrochemical oxidation involved the use of a UV-VIS spectrophotometer. The UV-VIS spectrophotometer functioned at a wavelength of 269 nm, enabling accurate measurement and analysis in the electrochemical process.

Table 3: Properties of phenol and potassium chloride

	phenol	**Potassium chloride**
Molecular formula	C_6H_5OH	KOH
Molecular Weight(gm/gmol)	94.11	56.11
Physical form	solid	Solid
PH	4.8 _ 6	14
Density (kg/m^3)	1070	2120
Solidification point (C^o)	40.5	40.5
Boiling point (C^o)	180	360
Melting Point (C^o)	40	1327

2.2 Catalyst Preparation

Impregnation method is the procedure that is utilized in the process of preparing the catalyst The purchased of Sky Spring Nanomaterials, Inc. company, United States of America. After soaking in diluted H_2SO_4 for 12 hours, γ-Al_2O_3 was neutralized by washing with deionized water. Deionized water was used to disseminate the processed γ-Al_2O_3 powder. Slowly, a specific volume of $Cu(NO_3)_2 \cdot 3H_2O$ was added to it. In order to eliminate moisture and create a homogenous solid mixture, the solution was then heated to 80 °C while being stirred. The mixture was put in a tube furnace and pyrolyzed for four hours at 400 °C in a N_2 atmosphere. After that, it was washed for twelve hours at 80 °C using deionized water. 10%CuO/γ- Al_2O_3 was the label applied to the sample that was prepared using this basic nitrate decomposition procedure.

2.2 Catalyst characterization

2.2.1 X-ray Diffraction Spectroscopy (XRD)

In figure 1, the XRD graph for our catalyst, sharp peaks at 2θ of 35.5 and 38.7 for the copper oxide crystals in tenorite and monoclinic phases. Moreover, peaks at 2θ 37.7, 46.1 and 66.84 for the gamma alumina (γ-Al_2O_3) can be easily noticed. This confirms our catalyst preparation and structure [30][31].

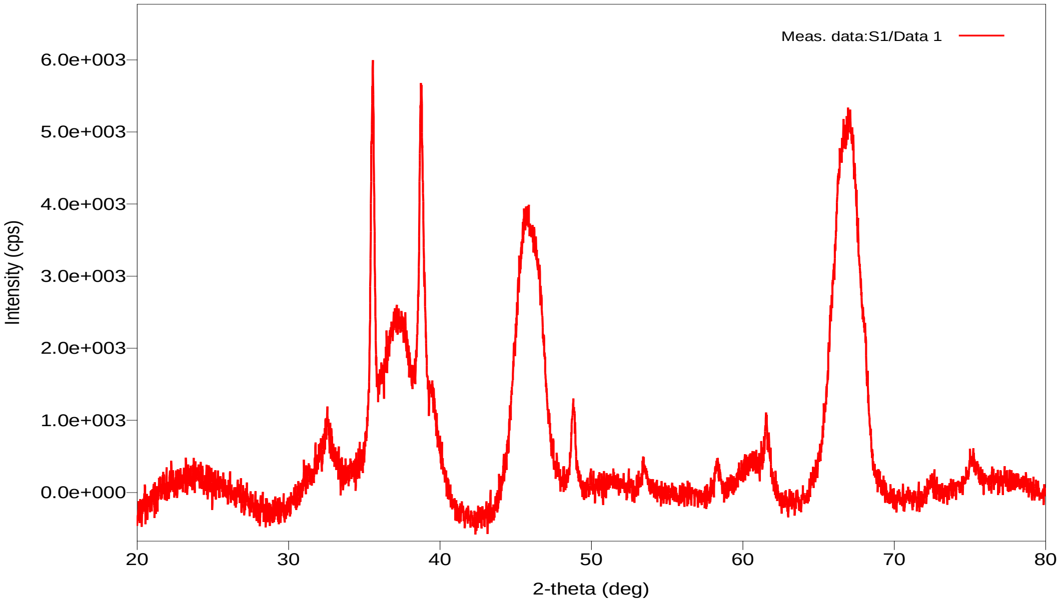


Figure (1): XRD graph for nanocomposites catalyst.

2.2.2 Fourier-Transform Infrared Spectroscopy (FTIR)

In Figure 2, the FTIR spectrum, we can confirm the catalyst structure from bands assigned to the following vibrations of aluminum bonds. Small bands at 740 and 620 cm^{-1}, symmetric (νs Al–O) and asymmetric (νas Al–O) bending vibrations of aluminum-oxygen bond, respectively. While bands for CuO vibrations, absorption bands (598, 520, 483, 440 cm^{-1}) is assigned to stretching Cu−O vibrations, were slightly out of the instruments' sensitivity [32].

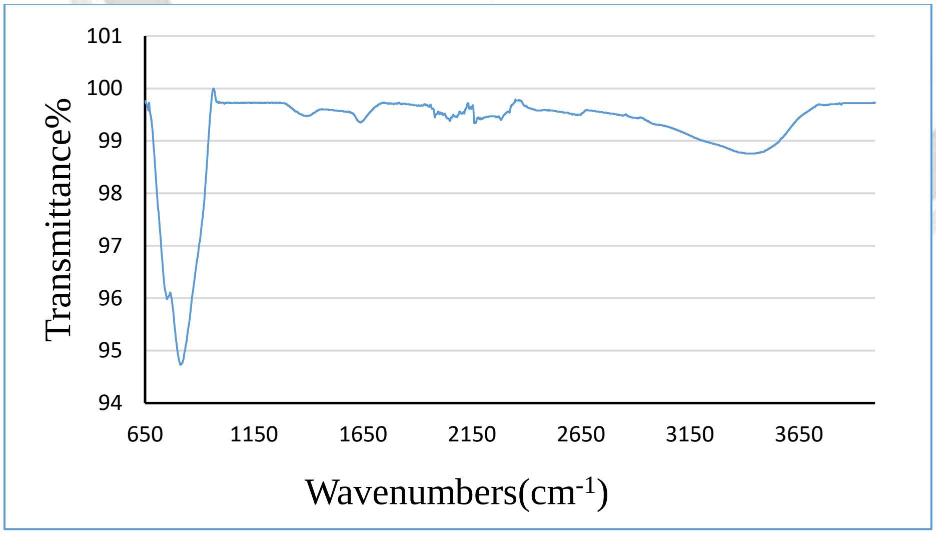


Figure (2): FTIR graph for the as prepared catalyst.

2.2.3 Surface Area Analysis Using BET

The surface area analysis of the catalyst was determined by nitrogen physisorption, using both BET and BJH theories. The usage of single point BET approximation was also used. By setting the intercept to 0 and ignoring the value of C. It might not be the most accurate surface area calculation but around the most appropriate relative pressure range for the BET, we can use it with very acceptable precision. We saw that the surface area from this estimation was in the range of 72.24 m^2/g to around 124.85 m^2/g in relation to a relative pressure range of 3.05 $x10^{-3}$ to almost 5 $x10^{-1}$ (P/P_o). This also matches the real surface area calculation from the Multi-point BET plot [33].

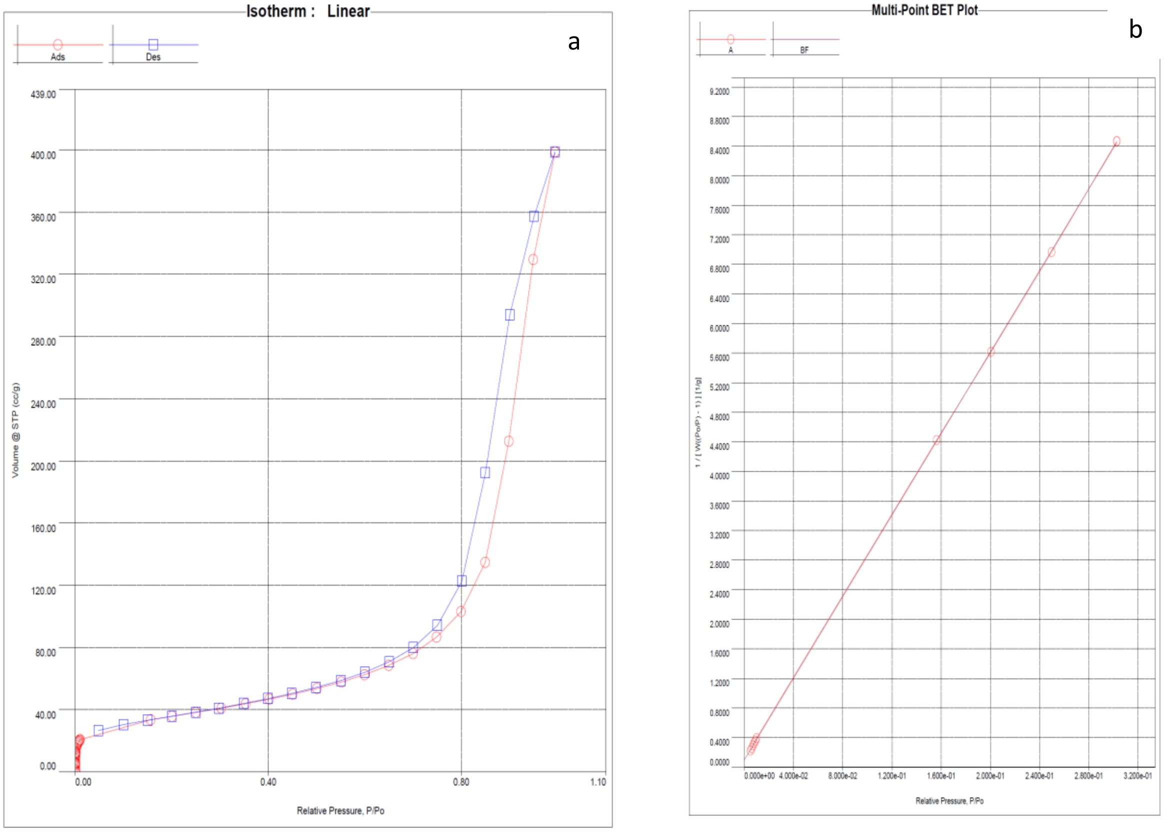
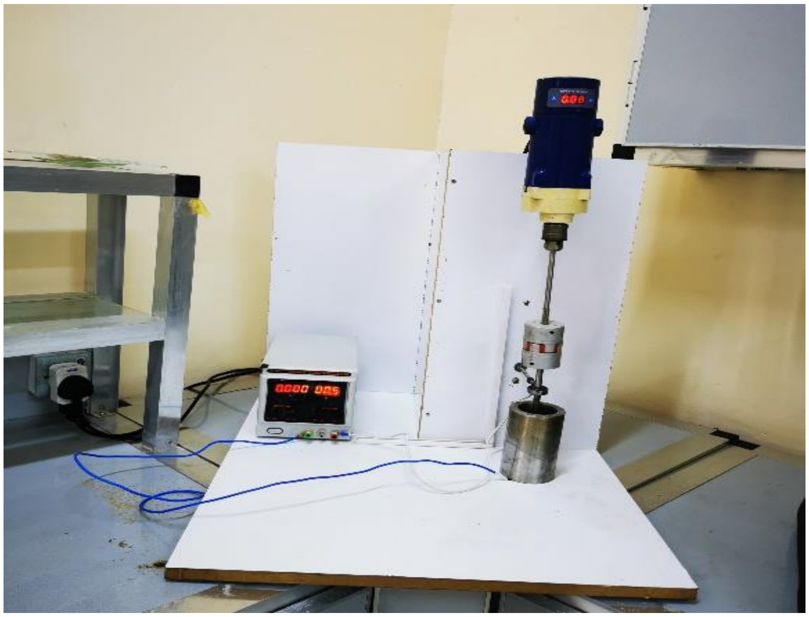


figure (3): a. Adsorption isotherm for the prepared nanocomposite. b Multi-point BET plot for the prepared nanocomposite.

3. Experimental Procedure and Setup

3.1 Electrochemical Framework and Mechanism

The electrochemical experimental setup involved an electrochemical basket reactor Figure 1, consisting of various components, including an anode who consist of A stainless steel (ASTM 316L) rotating basket reactor with four flat blades that has good capabilities to withstand corrosion and erosion of the electrolyte utilized. The basket serves as both the catalyst and anode electrode in this configuration, The cylindrical reactor shell material, stainless steel (A 316 L), was also used to make the cathode electrode also Non-conductive for electric, used to connect between the basket and agitator to ensure electrical separation. Phenol was dissolved in deionized water within a glass flask, then put in ultrasonic bath to completely dissolve phenol. For phenol elimination we put the specified amount of simulation refinery waste water in the shell of basket reactor.



Figure(4):Setup for an Experiment (real picture).

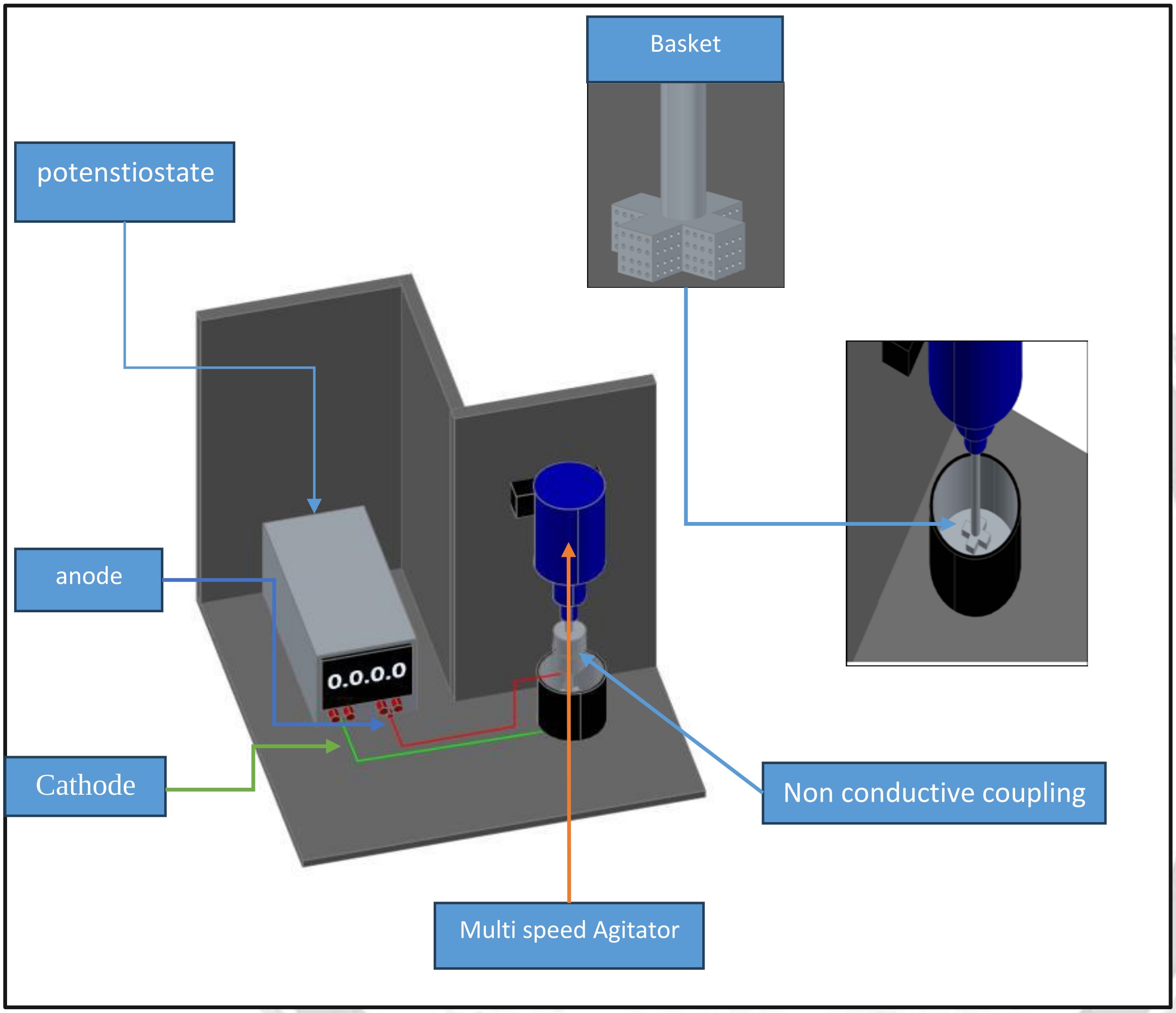


Figure (5): Experimental setup; 3D sketch.

According to previous research[34] [13], we can find that the known proposed mechanism for the electrochemical oxidation of organic substances using metal oxides, equation1, where the water is first oxidized where it forms a hydroxide free radical is formed beside our metal oxide which in turn oxidizes our organic matter present. In equation 2, we can also see the oxidation and degradation pathway for phenol, in waste water, and its side products and their pathways.

$$MO_x + H_2O \longrightarrow MO_x(OH\cdot) + H^+ + e^- \quad \ldots\ldots(1)$$

$$R + MOx(OH\cdot)z \longrightarrow z/2\, CO2 + zH^+ + ze^- + MOx \ \ldots..(2)$$

Equations (1) and (2) The general proposed mechanism for the electrochemical oxidation .

$$C_6H_5OH + H_2O \rightarrow C_6H_4O_2 + 4H^+ + 4e^-$$

$$C_6H_4O_2 + 6\,H_2O \rightarrow C_4H_4O_2 + 12\,H^+ + 2C0_2 + 12e^-$$

$$C_4H_4O_2 + 11\,H_2O \rightarrow 4CO_2 + 12\,H^+ + 12e^-$$

…………………………………………………………..

$$C_6H_5OH + 11\,H_2O \rightarrow 6CO_2 + 28\,H^+ + 28e^- \quad \ldots\ldots (3)$$

Equation (3) The proposed mechanisms and oxidation pathway for phenol.

Regarding our data, using the percentage product concentration formed (X%) at both 15 minutes it appears that higher voltages lead to higher removal efficiencies, suggesting that the catalyst is indeed more effective at higher voltages, though a higher voltage may come with more drawbacks such as, side reaction/products and inefficient water splitting, as we can see at 6 volts.

However, there could be an optimal voltage beyond which the efficiency does not increase or might even decrease due to factors such as excessive energy input leading to unwanted side reactions or damage to the catalyst or the equipment.

3.Effect of the Process Variables

4.1 Effect of the Initial Concentration

Figure (6) displayed the phenol degradation rates at various initial phenol concentrations of (10, 25, 50, 75, and 100 mg/l). The graphic demonstrates that the rate of phenol removal steadily increases as the concentration of phenol decreases. This is something that can be observed. The removal percentages were 94.66%, 85.11%, 81.34%, 74.7% and 60.52% at 15 min, respectively. The fact that this is the case shows that when the concentration is low, it is conducive to the practically full mineralization of phenol.

The initial concentration of a pollutant is a critical factor in any wastewater treatment process for several reasons. Firstly, the reaction rate, as the rate of many chemical reactions, often depends on the concentration of the reactants. In this case, a higher initial concentration of phenol might lead to a faster reaction rate, at least initially. Also, it affects the removal efficiency, for the removal efficiency is calculated based on the initial and final concentrations. The capacity of the catalyst might also be affected, as the effectiveness of a catalyst can depend on the pollutant concentration. If the concentration is too high, the catalyst might become saturated and less effective [35][34].

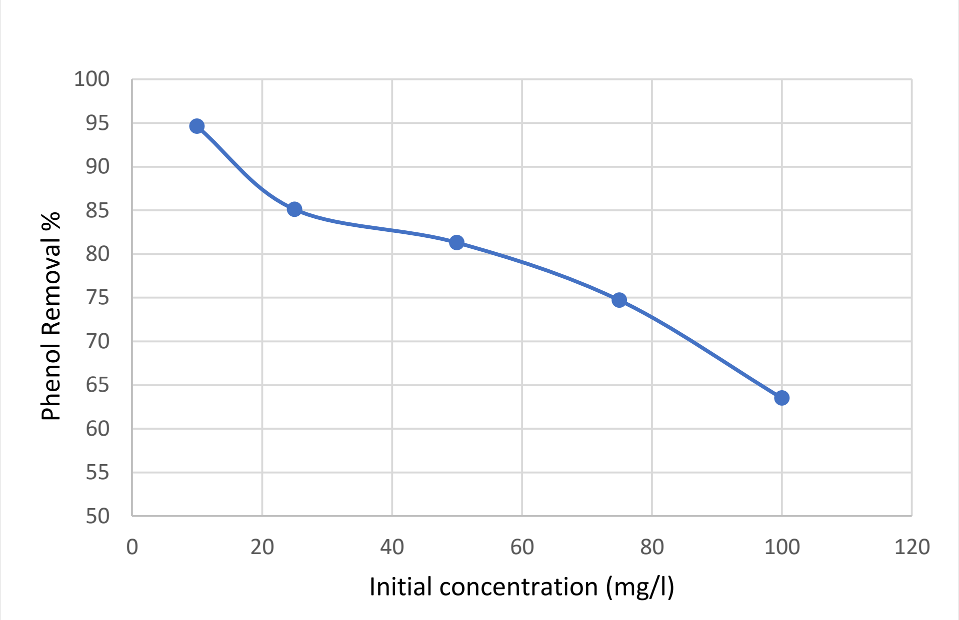


Figure (6): The effect of initial phenol concentration on the degradation rate of phenol at optimal condition (time:15 min, 5 V and rpm: 100)

4.2 Effect of the Applied Potential

The purpose of this study is to investigate the impact that the applied potential has on the effectiveness of the electrochemical oxidation process for the elimination of phenol, At a constant room temperature and working pressure, multiple experimental runs are carried out at varying voltages (3, 4, 5, and 6 volts) respectively, beginning amounts of phenol that are distinct (10,25,50,75 and 100 mg/l), four stirring speeds (25, 50, 75 and 100 rpm) and interval for time 15 min.

Here the voltage might play a role in multiple processes as, any electrochemical reactions can benefit from the higher voltages and will be driven more effectively. The catalyst being involved in the electrochemical reaction for the removal of phenol, then a higher voltage could increase the rate of that reaction. On the other hand, the generation of reactive species, electrochemical processes can generate reactive species that can react with and break down pollutants. A higher voltage can lead to the generation of more of these reactive species. In addition, the energy input, as the voltage represents the energy input into the system. A higher voltage means more energy is being supplied, which could enhance the reaction rate [36][37][38].

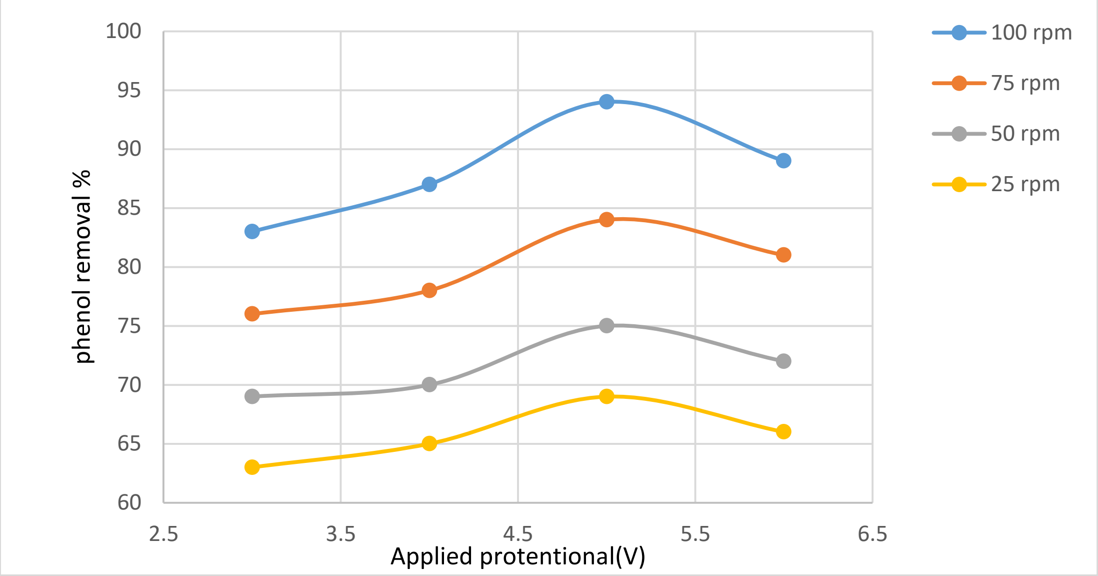


Figure (7): Effect of applied potential on phenol removal at different stirring speed (25,50,75 and 100 rpm), and reaction time of 15 minutes at initial Concentration (10 mg/l).

4.3 Effect of the rotation speed (RPM)

In most experiment, RPM (Revolutions Per Minute) likely refers to the speed at which the catalyst or the wastewater is being stirred or agitated. The RPM can have a significant impact on the effectiveness of the catalyst in a few ways. Initially, contact time, as higher RPMs can lead to more collisions between the phenol molecules and the catalyst, increasing the chances of the reaction occurring. The distribution of catalyst, if the catalyst is being stirred into the wastewater, a higher RPM will distribute the catalyst more evenly, increasing the surface area over which reactions can occur. However, this is usually a minor effect compared to the others [39].

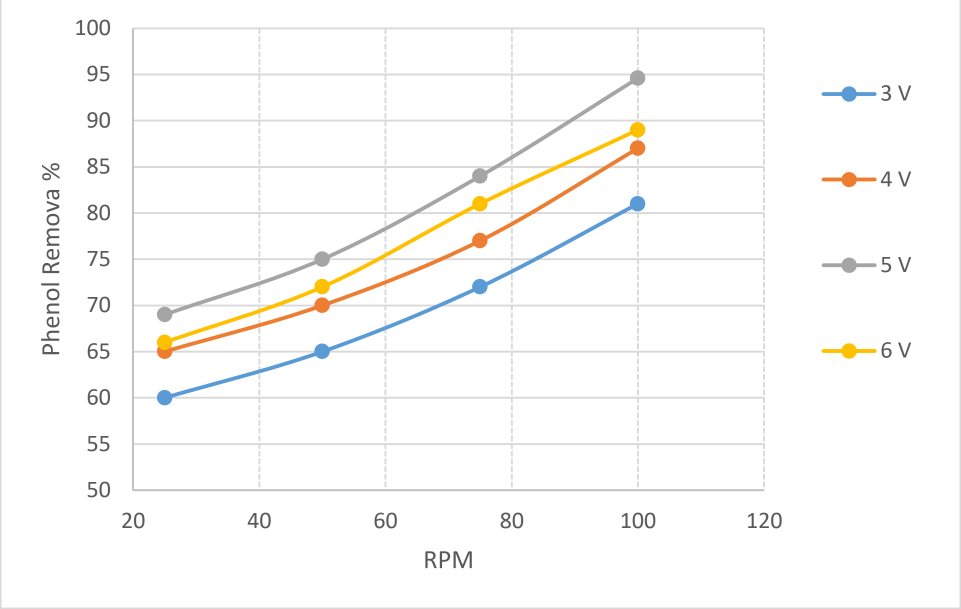


Figure (8): Effect of rotating speed on phenol removal at different applied potential (3,4,5 and 6 volt), and reaction time of 15 minutes at initial Concentration (10 mg/l).

In Figure (8) using the percentage product concentration formed (X%) at 15 minutes, it appears that higher RPMs generally lead to a higher removal efficiency, suggesting that the catalyst is indeed more effective at higher RPMs. This is consistent with the points above.

When the stirring speed is increased, there is a corresponding increase in the mass transfer in the bulk, which gives rise to the increased phenol conversion. When it comes to a multiphase process, the impact of mass transfer is extremely important. Adjusting the interface and lowering the resistance to mass transfer can be accomplished with relative ease through the use of stirring [40].

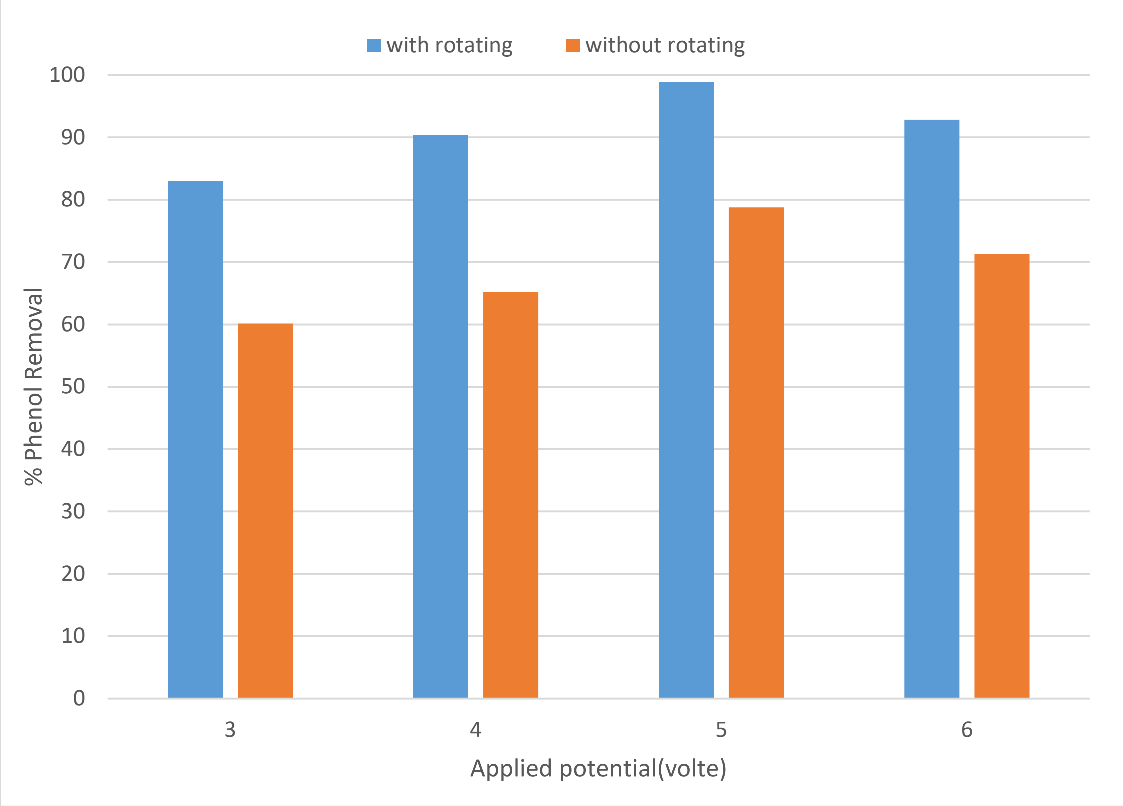


Figure (9): Effect of rotating anode at different applied potential (3,4,5 and 6) V, initial concentration 10 mg/l and 100 rpm.

Conclusion

Phenolic compound treatment is one of the major challenges in wastewater treatment. EO is an effective method for phenol treatment. This manuscript introduces an innovative approach to address the degradation of phenol using an advanced in-situ oxidation technique within a specially designed basket reactor. a stainless steel (ASTM 316L) rotating basket reactor with four flat blades that has good capabilities to withstand corrosion and erosion of the electrolyte utilized. The hydroxyl free radicals that were generated as a result of the electrolysis of water were able to successfully oxidize phenol without the need for any additional oxidants to be added.

The study methodically assesses the influence of several operational aspects such as applied voltage, initial concentration, and rotating speed, (reaction temperature and pressure) remaining constant, to achieve maximum removal by the electrochemical process within the Basket reactor. The results indicate that the in-situ oxidation process conducted in the customized reactor holds significant potential for improving phenol elimination. Under optimal conditions, including a potential of 5 V, a rotating speed of 100 rpm, an initial concentration of 10mg/l, and a reaction time of 30 minutes, the highest achieved phenol removal rate was an impressive 98.893%.

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إزالة الفينول من مياه الصرف الصحي للمصفاة عن طريق الاكسدة الكهروكيميائية باستخدام مفاعل كهروكيميائي ذو سلة تحفيزية مع انود دوار

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الخلاصة

الفينول شديد السمية حتى لو كان موجودًا بتراكيز منخفضة جدًا. تحتوي النفايات الصناعية الناتجة عن مصافي البترول بشكل عام على الفينول الذي يجب معالجته. في الحالات التي تكون فيها التقنيات التقليدية غير قادرة على التحكم في تحلل الفينول، ظهرت عملية الأكسدة الكهروكيميائية كحل لهذه المشكلة. تبحث هذه الدراسة في مدى فعالية النظام الكهروكيميائي التجريبي الذي يعمل على ازالة الفينول من مياه الصرف الصحي في المصافي باستخدام تقنية جديدة هي مفاعل كهروكيميائي ذو انود دوار. تم استخدام الأنود الدوار لزيادة كفاءة العملية وكذلك تم استخدم $(10\%\ CuO/\gamma-Al_2O_3)$ كعامل مساعد. أظهرت النتائج التجريبية أنه بزيادة سرعة الدوران للانود وزيادة جهد مصدر الطاقة للمفاعل الكهروكيميائي، زادت نسبة إزالة الفينول. وقد وجد أيضاً أنه بزيادة نركيز الفينول تنخفض نسبة الإزالة.

الكلمات الدالة: الفينول، الكهروكيميائية، مياه الصرف الصحي، الإزالة.