Treatment of Industrial Wastewater Containing Phenol Using the Electro-Fenton Technique in Gas Sparged Cell

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Abstract

The present study is concerned with evaluating the performance of Electro-Fenton process using iron electrodes as an alternative wastewater treatment technique. In this method, hydrogen peroxide is externally added into the system while a sacrificial iron anode is used as Fe^{+2} source so Fenton reaction was formed in the reactor. The batch reactor was stirred by introducing nitrogen gas through G4 type sintered glass and a synthetic solution of phenol was used to simulate wastewater. The effect of various process parameters such as: pH, gas superficial velocity, current density, H_2O_2 concentration, initial phenol concentration, and NaCl concentration on the electro-Fenton reaction was investigated to achieve maximum degradation efficiency. The batch experimental results revealed that the overall COD reduction reached 97% for initial phenol concentration 50 mg/l. The optimum pH, superficial gas velocity, current density was 3, 1.18 cm/s, 1.7 mA/cm² and H_2O_2 concentration was 1500 mg/l, respectively. The kinetic study revealed that the rate of COD reduction followed a pseudo first order reaction. Energy consumption and iron electrode consumption were calculated per gram of COD removed.

Keywords

Electro-Fenton, Phenol, Wastewater, Iron Electrode, Gas Sparged Cell

1. Introduction

Nowadays, the treatment of industrial wastewater has become a necessity to avoid environmental damage and to meet the strict environmental guidelines imposed by governments to control pollutants from industries. Among these pollutants, Phenol is a toxic, carcinogenic, and nonbiodegradable chemical. Phenol and its compounds add toxicity in water streams and are harmful to both human and aquatic animals [1]. Presence of phenolic compounds even at low concentration in the industrial wastewater adversely affects aquatic as well as human life directly or indirectly when disposed off to public sewage, river or surface water. Sometimes these form complex compounds with metal ions, discharged from other industries, which are more carcinogenic in nature than the phenolic compounds [2].

Phenols are present in wastewater of various industries, such as refineries, coking operations, coal processing and manufacture of petrochemicals. Phenols are also the main organic constituents present in condensate streams in coal gasification and liquefaction processes. Other sources of waste stream water containing phenols are pharmaceutical, plastics, wood products, paint, pulp and paper industries and chemical production facilities [3,4].

There are different methods for the removal of phenol from wastewater such as: steam distillation, separation by extraction, adsorption, separation by membrane processes, wet air oxidation, polymerization, biological treatment, electro-coagulation and chemical oxidation. Many problems associated with the previous mentioned methods have been reported such as: high cost, low efficiency and generation of toxic products. For example, toxicity of phenolic compounds inhibits biological treatment or even eliminates sensitive micro-organisms from biological wastewater treatment process and significantly reduces the biodegradation of the other components. This is why the degradation of phenol is such a difficult and crucial biological process [5].

As a result, the use of alternative treatment technologies, aiming to mineralize or transform refractory molecules into others which could be further biodegraded, is a matter of great concern. Among them, advanced oxidation processes (AOPs) have already been used for the treatment of wastewater containing recalcitrant organic compounds such pesticides. surfactants. colouring as matters. pharmaceuticals and endocrine disrupting chemicals. Moreover, they have been successfully used as pretreatment methods in order to reduce the concentrations of toxic organic compounds that inhibit biological wastewater treatment processes [6]. One of the most effective AOPs consists of the use of Fenton's reagent, a combination of H_2O_2 and Fe^{+2} . In this process, H_2O_2 decomposes catalytically by means of Fe⁺² at acid pH, giving rise to hydroxyl radicals [7].

The objective of the present work is to investigate the Elector-Fenton process in gas sparged cell using a sacrificial iron electrodes to remove phenol from wastewater by studying variables such as: pH, superficial gas velocity, current density, hydrogen peroxide concentration, initial phenol concentration, and electrolyte concentration (NaCl). Gas sparging rather than mechanical stirring was used in the present work to improve the mixing condition in the cell in view of the low cost of gas sparging compared to mechanical stirring [8]. The use of gas sparging would reduce the operating costs of the process. The removal efficiency of the treatment was determined by monitoring the reduction in COD.

2. Theoretical Considerations of the Electro-Fenton Process

The Fenton oxidation process is one of the oldest AOPs which are being increasingly used in the treatment of industrial wastewater. Fenton's reagent is able to destroy toxic compounds in wastewater such as phenols and herbicides. The Fenton's system consists of ferrous salts combined with hydrogen peroxide under acidic conditions. This reaction allows the generation of hydroxyl radicals as shown in (Eq.1) [9]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\bullet}$$
(1)

This reaction is propagated by ferrous ion regeneration (Eq.2), which is mainly due to the reduction of the produced ferric species with hydrogen peroxide where hydroperoxyl radical (HO₂[•]), with much lower oxidant power than OH, is formed. In addition, reduction of Fe³⁺ with HO₂[•] and/or organic radical intermediates is also possible (Eq. 3&4)

$$\mathrm{Fe}^{3+} + \mathrm{H}_2\mathrm{O}_2 \rightarrow \mathrm{Fe}^{2+} + \mathrm{HO}_2^{\bullet} + \mathrm{H}^+$$
(2)

$$\operatorname{Fe}^{3+} + \operatorname{HO}_{2} \xrightarrow{\bullet} \operatorname{Fe}^{2+} + \operatorname{O}_{2} + \operatorname{H}^{+}$$
(3)

$$\operatorname{Fe}^{3^+} + \operatorname{R}^{\bullet} \to \operatorname{Fe}^{2^+} + \operatorname{R}^+$$
 (4)

In Electro-Fenton, hydrogen peroxide is externally added into the system while a sacrificial iron anode is used as ferrous ion source (Eq.5), so the Fenton reaction is formed in the system.

$$Fe \rightarrow Fe^{2+} + 2e^{-}$$
 (5)

At the cathode, the hydrogen evolution (Eq.6) is the main reaction and the ferric ions can be directly reduced as (Eq.7):

$$2\mathrm{H}^{+} + 2\mathrm{e}^{-} \to \mathrm{H}_{2} \tag{6}$$

$$Fe^{3+} + e^{-} \rightarrow Fe^{2+}$$
(7)

This process was applied to detoxifying herbicide, pesticide, and insecticide containing wastewater and polishing biological effluent of a petrochemical wastewater as well [10].

3. Experimental Part

3.1. Chemicals

All chemicals used were analytical grade. The main chemical used for Electro-Fenton process is hydrogen peroxide solution H_2O_2 (30 wt%). Phenol was purified by distillation and dissolved in distilled water as a stock solution. Experimental solutions of the desired concentrations were obtained by successive dilution with distilled water. The initial pH of the solution was adjusted to the desired value using sodium hydroxide or hydrochloric acid solution.

3.2. Analytical Method

Chemical oxygen demand (COD) of samples was measured by closed reflux method according to ASTM D1252-95 [11]. The samples were tested using COD digestion vials (Hach), where the sample is placed within digestion vials which in turn are heated at 150°C for 2 hours. Digestion vials were then allowed to cool naturally to ambient temperature before having the COD measured.

3.3. Experimental Setup

Electro-Fenton experiments were carried out in a batch reactor system that schematically illustrated in Figure (1). The reactor consists of a plexi-glass cylinder of 11.5 cm diameter and 21 cm height, with a G4 type sintered glass attached to its bottom to maintain a uniform gas flow into the cell. A nitrogen cylinder was used to supply needed nitrogen to the bottom of the cell through the sintered glass.

A cylindrical iron sheet lining the vessel wall with a height of 15.8 cm and a diameter of 11.2 cm, isolated with epoxy from its back, was used as cathode, while an iron rod with a height of 13 cm and a diameter of 2 cm was used as anode, The flat end of the cylinder anode was isolated with epoxy resin. The iron rod anode was placed in the center of the reactor at 2.8 cm from the reactor bottom.

The electrical circuit consisted of power supply (20V, 10A) with a voltage regulator and multi range ammeter, all connected in series with the cell, a voltmeter was connected in parallel with the cell to measure its voltage. All experiments were performed at room temperature ($25\pm1^{\circ}C$). Temperature changes were measured by a thermometer and were not significant.



1. Power supply 2. Anode 3. Cathode 4. Sintered glass 5. Control valve 6. Gasflow meter. Nitrogen cylinder

Figure (1). Experimental setup.

3.4. Electro-Fenton Procedures

At the beginning of a run, the phenol solution was fed into the electrolytic cylindrical cell and the pH and conductivity were adjusted to the desired values. The pH was adjusted using either NaOH or HCl as necessary. The conductivity of the solution was raised by adding the appropriate amount of NaCl into the phenol solution. Hydrogen peroxide was added at the desired concentrations (500, 1000, 1500, 2000, 2500 mg/l). The reaction was timed starting when the D.C. power supply was switched on. Direct current from the D.C power supplier was passed through the solution via the two electrodes during 60 minutes of electrolysis run; samples from the solution were drawn at every 15 minute interval for one hour then COD was measured. The location of the drawn samples was kept constant for each run.

The electrodes were washed with HCl solution (15% w/v) before each run in order to remove any adhering scales or oxides. Following each run, the electrodes were washed with distilled water, dried until they are used again. The percentage reduction in COD was calculated as:

% Reduction in COD =($(C_i - C_f)/C_i$)*100

Where; C_i is the initial chemical oxygen demand (mg/l) and C_f is the final chemical oxygen demand (mg/l).

4. Results and Discussion

4.1. Effect of pH

The pH is one of the most important factors for the electro-Fenton process. It has been confirmed that the optimal value of pH is $2\sim4$ [12]. The effect of change of initial pH values on the removal efficiency is illustrated in Fig. (2). Therefore, the initial pH value has to be in the acidic range to generate the maximum amount of OH[•] and oxidize the organic compounds. In particular the optimal value of pH is 3, which is in agreement with previous results obtained in other studies using Fenton's reagent [13-17].

At pH > 3, Hydrogen peroxide decomposes to oxygen and water and does not produce hydroxyl radical [18] as well as the iron ions especially the Fe⁺³ precipitate. Therefore, the efficiency of Electro-Fenton decreases rapidly above pH 3. On the other hand, when pH< 3, H₂O₂ can not be discomposed to OH[•] by Fe²⁺. In this case, H₂O₂ turns into H₃O₂⁺ by capturing one proton. H₃O₂⁺ is electrophilic, leading to the decreasing rate of reaction between H₂O₂ and Fe²⁺ [12].

$$H_2O_2 + H^+ \rightarrow H_3O_2^+$$
(8)



Figure (2). Effect of pH on the percentage reduction of COD (initial phenol conc. = 50 mg/l, hydrogen peroxide conc. = 1500 mg/l, NaCl conc. = 2 g/l, C.D= 1.7 mA/cm^2 , superficial gas velocity = 1.18 cm/s and time= 60 min).

4.2. Effect of Superficial Gas Velocity

The effect of change of superficial gas flow velocity on the COD removal efficiency is illustrated in Fig. (3). Within the range from 0.36 cm/s to 1.9 cm/s it was found out that the superficial gas velocity has a slight effect on COD removal then it remains constant probably because the reaction is chemically controlled where diffusion plays a minor role, the

optimum superficial gas flow velocity is 1.18 cm/s. The role of N₂ stirring is to improve the mixing conditions at the anode surface to decrease concentration polarization arising on the accumulation of dissolved Fe^{++} at the anode surface, decrease of concentration polarization not only eliminate anode passivation but also decrease cell voltage and electrical energy consumption.



Figure (3). Effect of gas flow velocity on the percentage reduction in COD (initial phenol conc. = 50 mg/l, hydrogen peroxide conc. = 1500 mg/l, NaCl conc. = 1 g/l, C.D = 1.7 mA/cm^2 , pH = 3 and time = 60 min).

4.3. Effect of Current Density

Current density is very important parameter in the Electro-Fenton processes because it is responsible for the generation of Fe^{++} ions within the electrochemical cell. The main goal of ferrous ion as a catalyst is to release hydroxyl radical from hydrogen peroxide.

As seen in Fig. (4) at constant initial phenol concentration, hydrogen peroxide, pH, superficial gas velocity and NaCl

concentration; it was found that the % COD reduction has increased from 78.8% to 97% by increasing current density from 0.7 mA/cm² to 1.7 mA/cm² then it decreased to 93% by increasing current density to 2.2 mA/cm².

This is ascribed to the fact that at higher current densities the dissolution of the anode increases according to Faraday's law. Generally the efficiency of the Electro-Fenton process increases with Fe²⁺ concentration because the concentration of hydroxyl radicals, which is the main oxidizing agent in the Electro-Fenton process increases with the increase in Fe²⁺ concentration. However, ferrous ions in the electrolyte solution when present in excess could consume the hydroxyl radicals and affect the extent of degradation [19, 20].



Figure (4). Effect of current density on the percentage reduction in COD (Initial phenol conc. = 50 mg/l, Hydrogen peroxide conc. = 1500 mg/l, NaCl conc. = 3 g/l, pH = 3, superficial gas velocity = 1.18 cm/s and time = 60 min.

The effect of current density on the % reduction in COD at different initial phenol concentrations was presented in Figure (5).



Figure (5). Effect of current density on the percentage reduction of COD removal at different initial concentration of phenol (Hydrogen peroxide conc. = 1500 mg/l, NaCl conc. = 2 g/l, pH = 3, Superficial gas velocity = 1.18 cm/s and time= 60 min).

4.4. Effect of H₂O₂Concentration

As seen in Figure (6) at constant current density, initial phenol concentration, pH, superficial gas velocity and NaCl

concentration, it was found that the % COD reduction has increased from 33% to 96% by increasing H_2O_2 from 0 mg/l to 1500 mg/l then it decreased to 82% by increasing H_2O_2 to 2500 mg/l.

(9)

The initial concentration of H_2O_2 plays an important role in the Electro-Fenton process. Removal of pollutants increases with increase in H_2O_2 concentration. The increase in the removal efficiency was due to the increase in hydroxyl radical concentration as a result of the addition of H_2O_2 . At a high dosage of H_2O_2 , the decrease in removal efficiency was due to the hydroxyl radical scavenging effect of H_2O_2 and the recombination of the hydroxyl radical [19].

$$\mathrm{H}_{2}\mathrm{O}_{2} + \mathrm{OH}^{\bullet} \rightarrow \mathrm{HO}_{2}^{\bullet} + \mathrm{H}_{2}\mathrm{O}$$
 (10)

This reaction result in the consumption of hydrogen peroxide as well hydroxyl radical and the production of hydroperoxyl radical, a species with much weaker oxidizing power compared with hydroxyl radical.



Figure (6). Effect of Hydrogen peroxide conc. on the %reduction in COD (Initial phenol conc. = 50 mg/l, C.D = 1.7mA/cm^2 , NaCl conc. = 2 g/l, pH = 3, gas velocity = 1.18 cm/s and time = 60 min).

The effect of H_2O_2 concentration on the percentage reduction in COD at different initial phenol concentrations was presented in Figure (7).



Figure (7). Effect of hydrogen peroxide conc. on the %reduction in COD at different initial concentration of phenol (C. $D = 1.7 \text{ mA/cm}^2$, NaCl conc. = 2 g/l, pH = 3, gas velocity = 1.18 cm/s and time = 60 min).

4.5. Effect of Initial Phenol Concentration

A series of batch experiments with initial phenol concentrations at a constant current density were performed to derive the influence of phenol initial concentrations on %COD reduction. Figure (8) shows the effect of initial phenol concentration on the variation of the COD removal during the Electro-Fenton process. The percentage reduction in COD was gradually decreased from 89.7% to 78.5% as the

phenol concentration increased from 50 to 250 mg/l. This is possibly due to the formation of insufficient number of hydroxyl radicals produced for a given hydrogen peroxide concentration and applied current density to oxidize the excessive amount of phenol at higher concentrations. It is, therefore, quite clear that under the present experimental conditions the lower is the phenol concentration, the better is the removal efficiency [10].



Figure (8). Effect of time on the %reduction inCOD at different initial phenol concentration (Hydrogen peroxide conc. = 1500 mg/l, NaCl conc. = 1 g/l, C.D = 1.7 mA/cm^2 , pH = 3, gas velocity = 1.18 cm/s and time = 60 min).

The effect of initial phenol concentrations on the percentage reduction in COD at different hydrogen peroxide concentrations was presented in Figure (9).



Figure (9). Effect of initial phenol concentration on the % reduction in COD at different H_2O_2 concentration (C. $D = 1.7 \text{ mA/cm}^2$, NaCl conc. = 1 g/l, pH = 3, superficial gas velocity = 1.18 cm/s and time = 60 min).

4.6. Effect of NaCl Concentration

Different doses of sodium chloride were added as a supporting electrolyte to enhance the conductivity of the solution and to overcome anode passivity. Figures (10,11) showed that the increase in the amount of NaCl results in increasing removal efficiency.

The amount of iron generated increased rapidly as the sodium chloride dose varies from 1 to 3 g/l. This may be explained by the fact that the higher chloride ion concentration, the higher the ability of CI^{-} to destroy any passive oxide film, which tends to be formed on the anode

and limit anode dissolution, Hence it increases the availability of iron in the solution and improves the efficiency of phenol removal. The addition of NaCl would also lead to the decrease in power consumption due to the increase in conductivity. The higher ionic strength will generally cause an increase in current density at the same cell voltage or, equivalently, the cell voltage decreases with increasing wastewater conductivity at constant current density. Consequently, the necessary voltage for attaining to a certain current density will be diminished and the consumed electrical energy will decrease [10].



Figure (10). Effect of NaCl concentration on the % reduction in COD (initial phenol concentration = 50 mg/l, C. $D = 1.7 \text{ mA/cm}^2$, $H_2O_2 = 1500 \text{ mg/l}$, pH = 3, superficial gas velocity = 1.18 cm/s and time = 60 min).



Figure (11). Effect of NaCl concentration on the % reduction in COD at different initial phenol concentration (C. $D = 1.7 \text{ mA/cm}^2$, $H_2O_2 = 1500 \text{ mg/l}$, pH = 3, gas velocity = 1.18 cm/s and time = 60 min).

4.7. Electrical Energy Consumption and Electrode Consumption

In order to assist in assessing the economic feasibility of Electro-Fenton in comparison with other techniques, the energy consumption and Fe metal consumption were calculated as follows [21]:

(i) Energy consumption (kWh/g COD removed) =

 $\frac{1}{(C_{\circ} - C_{t})V}$

Where:

E is the cell voltage (Volt),

I is the current (A),

t is the Electro-Fenton time (h),

C_o is the initial compound concentration (mg/l),

 C_t is the concentration at time t (mg/l),

V is the treated volume (L).

(ii) The amount of Fe metal consumed in Electro-Fenton was calculated using Faraday's law:

Fe consumption (g Fe/g phenol removed) =
$$\frac{\text{ItM}}{\text{ZFV}(C_{\circ} - C_{t})}$$

Where:

M is the molecular weight of iron (55.85 g/mol)

Z is the number of electron transfer ($Z_{Fe} = 2$).

F is Faraday's constant (96,500 C/mol).

The variation of electrical energy consumption and electrode consumption with current density, initial hydrogen peroxide concentration, initial phenol concentration and initial electrolyte concentration were presented in Figures (12 - 15).



Figure (12). Effect of current density on the energy consumption and Fe consumption, (initial phenol conc. = 50 mg/l, H_2O_2 conc. = 1500 mg/l, NaCl conc. = 2 g/l, pH = 3 and superficial gas velocity = 1.18 cm/sec).



Figure (13). Effect of H_2O_2 conc. on the energy consumption and Fe consumption, (initial phenol conc. = 50 mg/l, $C.D = 1.7 \text{ mA/cm}^2$, NaCl conc. = 2 g/l, pH = 3 and superficial gas velocity = 1.18 cm/sec).



Figure (14). Effect of initial phenol conc. on the energy consumption and Fe consumption, $(H_2O_2 \text{ conc.} = 1500 \text{ mg/l}, \text{ C.D} = 1.7 \text{ mA/cm}^2, \text{ NaCl conc.} = 2 \text{ g/l}, pH = 3 \text{ and superficial gas velocity} = 1.18 \text{ cm/sec}$).



Figure (15). Effect of initial NaCl conc. on the energy consumption and Fe consumption, (initial phenol conc. = 50 mg/l, H_2O_2 conc. = 1500 mg/l, C.D = 1.7 mA/cm², pH = 3 and superficial gas velocity = 1.18 cm/sec).

4.8. Kinetics of Electro-Fenton

The kinetic study is an important issue in order to examine the order of the reaction. In an attempt to throw some light on the kinetics of the present process which is difficult to quantify owing to its complexity and it is being controlled by many interacting variables. It was assumed that the process takes place according to pseudo-first order mechanism [22, 23], which can be expressed by:

 $\ln\left(\frac{C_o}{C_i}\right) = Kt$

- C_o is the initial concentration, (mg/l),
- C_t is the concentration at time t, (mg/l),
- K is the rate constant, (min⁻¹) and
- t is the time, (min.).

To investigate the kinetics of COD Reduction, electrolytes were performed with different parameters effect. A pseudofirst order kinetics model was applied to investigate the kinetics of degradation. Figures (16) to (19) show the effect of current density, NaCl concentration, pH value and initial concentration of phenol on the kinetics of the reaction



Figure (16). Effect of Hydrogen peroxide conc. concentration on the kinetics of the reaction, (initial phenol concentration = $50 \text{ mg/l C.D} = 1.7 \text{ mA/cm}^2$, NaCl conc. = 2 g/L, pH = 3, gas velocity = 1.18 cm/s, Time= 60 min).



Figure (17). Effect of NaClconcentration on the kinetics of the reaction, (initial phenol concentration = $150 \text{ mg/l C.D} = 1.7 \text{ mA/cm}^2$, pH= 3, gas velocity = 1.18 cm/s, Time= 60 min).



Figure (18). Effect initial concentration of phenol on the kinetics of the reaction, $(H_2O_2 \text{ conc.} = 150 \text{ mg/l C.}D= 1.7 \text{ mA/cm}^2, \text{ NaCl conc.} = 2g/l, pH= 3, gas velocity = 1.18 cm/s, Time = 60min).$



Figure (19). Effect current density on the kinetics of the reaction, (initial conc. of phenol = 150 mg/l, $H_2O_2 \text{ conc.} = 1500 \text{ mg/l}$, $C.D = 1.7 \text{ mA/cm}^2$, NaCl conc. = 2g/l, pH = 3, gas velocity = 1.18 cm/s, Time = 60 min).

5. Conclusions

The efficiency of the Electro-Fenton process for removal of phenol from wastewater depends on many operational parameters, such as: pH, superficial gas velocity, current density, H_2O_2 concentration, initial phenol concentration, and electrolyte concentration (NaCl).

The following conclusions have been withdrawn:

- 1. The optimal value of pH is 3. The initial pH at highly acidic or alkaline conditions, the percentage of COD reduction was lower.
- 2. The optimal value of superficial gas velocity is 1.18 cm/sec. Superficial gas velocity does not have a significant effect on % COD reduction as the reaction is chemically controlled.
- 3. The percentage of COD reduction increases with increasing current density. But beyond the optimum current density, the %COD reduction starts to decrease again
- 4. The percentage of COD reduction increases with increasing H_2O_2 concentration. But beyond the optimum H_2O_2 concentration, the %COD reduction starts to decrease again
- 5. At the highest initial phenol concentration, the %COD reduction was decreased.
- 6. The percentage of COD reduction increases with increasing NaCl concentration.
- 7. Energy and iron consumption decrease with increasing initial concentration of phenol, NaCl Concentration and increases with increasing the current density.
- 8. For the purpose of reactor design, study of the kinetics revealed that the processes of phenol removal take place through a pseudo-first order reaction.

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