



Research Journal of  
**Environmental  
Sciences**

ISSN 1819-3412



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## Degradation of Phenol in Aqueous Solutions Using Electro-Fenton Process

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### ABSTRACT

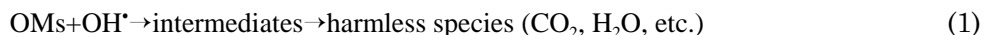
Electrochemical methods as one of the Advanced Oxidation Processes (AOPs), have been applied effectively to degradation of recalcitrant organic in aqueous solutions. In the present study, the performance of Electro-Fenton (EF) method using iron electrodes on the oxidation of phenol was studied. This experimental study was performed in batch reactor and the effect of operating parameters such as solution pH, operating time, applied current, H<sub>2</sub>O<sub>2</sub> and phenol concentration was investigated. In this study the H<sub>2</sub>O<sub>2</sub> is manually added while a disposal iron anode is applied as a ferrous ion source. Experimental data indicated that pH, H<sub>2</sub>O<sub>2</sub> and phenol concentration have principal role in the process. The results show that removal efficiency of phenol decreased with increasing pH, so that the maximum efficiency 26% was obtained at pH 10, also decreasing of H<sub>2</sub>O<sub>2</sub> concentration decreased the efficiency. Optimal condition for removing 100% of phenol was found after 30 min of reaction at pH = 3.0, H<sub>2</sub>O<sub>2</sub> concentration 100 mg L<sup>-1</sup> and 5 mA cm<sup>-2</sup>. Electro-Fenton process using iron electrodes for phenol degradation and remediation of wastewater is a promising process in terms of environmental and low-cost features.

**Key words:** Electro-Fenton, phenol, aqueous solution

### INTRODUCTION

Treatment of industrial wastewater is a problem of main concern at present. More stern regulations are being imposed, which persevere on the need to develop and apply treatment technologies effective to deal with the hazardous pollutants present-day in many industrial waste streams, wastewater compressing phenol and phenolic compounds presents a critical discharge problem due to their poor biodegradability, high toxicity and ecological features, phenols are large-scale distributed as environmental pollutants (Abdelwahab *et al.*, 2009). Currently, phenol is produced at a rate of about 6 million t in year global, with a critical increasing trend (Busca *et al.*, 2008). Phenol and phenolic compounds released into the surface water bodies in different from many industrial processes, including coking, synthetic rubber, pulp and paper, oil

refineries, plastics, petrochemical, steel, conversion processes, phenolic resin industries and by different other chemical works (Abdelwahab *et al.*, 2009; Babuponnusami and Muthukumar, 2011, 2012). Phenol has germane health effects for humans, this chemical compounds is quickly absorbed through the skin and can cause skin and eye burns upon touch, stupor, paroxysms, cyanosis and death can result from overexposure to it. Internally, phenol affects the liver, kidneys, lungs and vascular system. The ingestion of 1 g of phenol is fatal for man (Busca *et al.*, 2008). Since, the Environmental Protection Agency (EPA) calls for reducing phenol amount in the wastewater to less than 1 mg L<sup>-1</sup> (Abdelwahab *et al.*, 2009). Generally studied methods for the treatment of wastewaters containing phenols and phenolic compounds include biological treatment (Fang *et al.*, 2006), extraction (Kujawski *et al.*, 2004), wet oxidation (Santos *et al.*, 2006), electrocoagulation (Ugurlu *et al.*, 2008) and chemical oxidation (Babuponnusami and Muthukumar, 2012). Phenol due to high solubility and stability makes its removal using traditional biologic treatment reasonably unachievable (Babuponnusami and Muthukumar, 2011). Though, Advanced Oxidation Processes (AOPs) have been widely used for the removal of large range of toxic organic pollutants from wastewater (Babuponnusami and Muthukumar, 2011, 2012). The important of AOPs, are Fenton, photo-Fenton, sono-Fenton (Babuponnusami and Muthukumar, 2012; Ranjit *et al.*, 2008), electrochemical oxidation such as electro-Fenton (Jiang and Mao, 2012) methods. These process are based on the production of quickly and very reactive, non-selective, very powerful oxidizing agents such as hydroxyl radicals (OH<sup>•</sup>) in solution, which have a powerful oxidative potential (E<sup>0</sup> = 2.80 V vs. NHE) that is only lesser than fluorine (Brillas *et al.*, 2005; Ghoneim *et al.*, 2011). Due to the volatility of OH radical, it must be generated continuously “*in situ*” through chemical or photochemical reactions (Hao *et al.*, 2000; Khataee *et al.*, 2009). The Organic Matters (OMs) in the wastewater are attacked by hydroxyl radical, as explained in Eq. 1 (Khataee *et al.*, 2009):



Classic Fenton reaction (Fe<sup>2+</sup>/H<sub>2</sub>O<sub>2</sub>) is one of the most widely used AOPs for the degradation of a wide range of refractory organics such as dyes that cannot be oxidation biologically in effluents (Fan *et al.*, 2010; Ma *et al.*, 2005). In Fenton process the generation of hydroxyl radical is based on the electron transfer between hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and a metal catalyst such as (Fe<sup>2+</sup>) (Ali *et al.*, 1996). This process is explained in Eq. 2 (Babuponnusami and Muthukumar, 2012):



But Fenton process has two main problems for large-scale used in the wastewater treatment: The first is that H<sub>2</sub>O<sub>2</sub> expensive and the second is related to the operational pH, because Fe<sup>2+</sup> and Fe<sup>3+</sup> precipitated as a hydroxide at pH higher than 2-3 (Brillas *et al.*, 2000; Brillas and Casado, 2002). A cooperation of electrochemical process and Fenton reaction, namely Electro-Fenton (EF) process, are combined to improve the oxidation of refractory organic compounds in wastewater (Umar *et al.*, 2010). This method is divided into four groups: In type 1, H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> are electro generation on a sacrificial anode and a cathode, respectively (Wang *et al.*, 2010), in type 2, H<sub>2</sub>O<sub>2</sub> is manually added while a sacrificial iron anode is applied as a Fe<sup>2+</sup> source (Kurt *et al.*, 2007; Sengil and Ozacar, 2006), in type 3, Fe<sup>2+</sup> ion is manually added and H<sub>2</sub>O<sub>2</sub> are electro generation using an oxygen sparging on cathode (Badellino *et al.*, 2006) and in type 4,

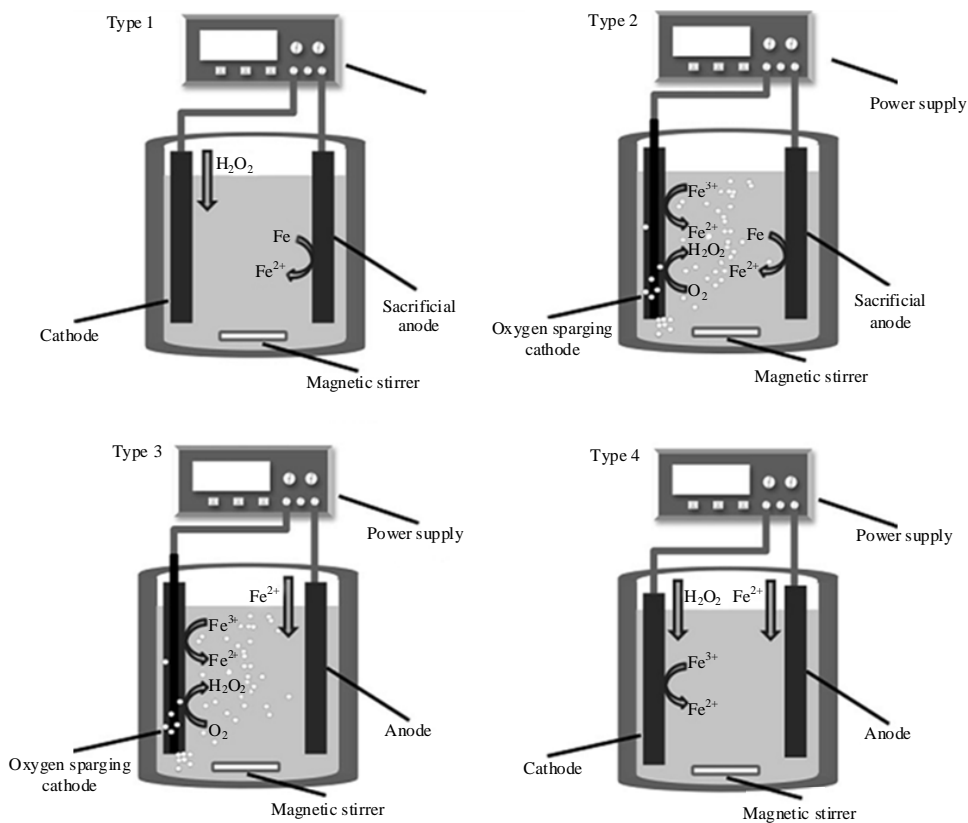


Fig. 1: Schematic of the electro-Fenton process and different groups of classification

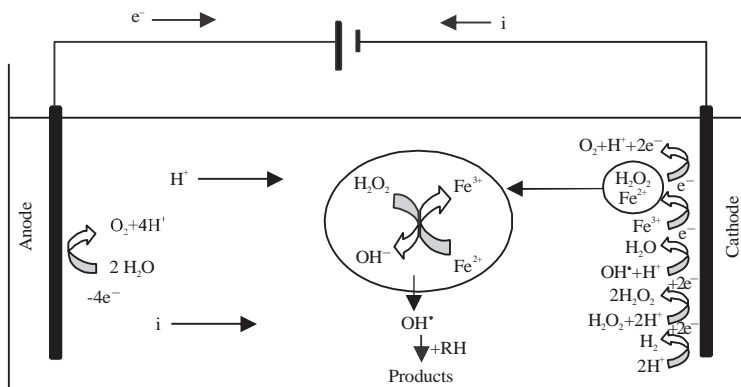
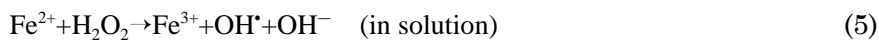


Fig. 2: Reaction mechanism of electro-Fenton process

Fenton's reagent is applied to generation  $\text{OH}^\bullet$  in an electrolytic cell and ferrous ion is regenerated via the reduction of ferric ion at the cathode (Babuponnusami and Muthukumar, 2012). Schematic of the EF process and different groups of classification showed in Fig. 1 (Rosales *et al.*, 2012).

Electro-Fenton (EF) process is not using any harmful and toxic materials, it is an environment friendly and low-cost method for treatment of water and wastewater (Rosales *et al.*, 2012). The main reaction of EF process explained in the Eq. 2-5 and Fig. 2 (Nidheesh and Gandhimathi, 2012):





The objective of this study was to investigate the efficiency of EF process using iron electrodes in oxidation of phenol. In this regard, the effects of  $\text{H}_2\text{O}_2$  and phenol concentration, pH, applied current and operating time were studied.

## MATERIALS AND METHODS

**Materials:** Phenol (99.5% pure), hydrogen peroxide (30 wt. %), 4-aminoantipyrine, potassium ferricyanide, hydroxide ammonium, phosphate buffer, sodium hydroxide, sulfuric acid, sodium sulfate and all other reagents were obtained from Merck (Germany). The solutions were prepared by dissolving the adequate quantities of the compounds in distilled water. Stock solution ( $1000 \text{ mg L}^{-1}$ ) of phenol was prepared by dissolving of 1 g phenol into distilled water. Experimental solutions of the desired concentrations of phenol were obtained by successive dilutions (50, 100, 150 and  $200 \text{ mg L}^{-1}$ ).

**Experiments:** Batch experiments were performed in the cylindrical glass cell (1250 mL) and filled with 1000 mL phenol solution of desired concentration for EF experiments. Electrochemical cell equipped with four electrodes that immersed into the solution, the iron cathode and anode of equal dimensions ( $2 \times 20 \times 200 \text{ mm}$ ) and the gap between the anode and cathode was 20 mm. A single-output adjustable DC power supply was used as the power supplier. Solution pH values were adjusted using reagent grade NaOH and/or sulfuric acid solutions with different molar concentrations. The pH of solutions was measured using a pH meter (Wagteck Mi. 151). Arrangements and experiments were realized at room temperature.

For improve conductivity and the ionic strength, added of  $100 \text{ mg L}^{-1}$  of  $\text{Na}_2\text{SO}_4$  as the electrolyte. The mixture was stirred using a magnetic stirrer for 60 min, samples were taken from the electrochemical cell for each 15 min in all the experiments and centrifuged for 3 min at 4000 rpm before analysis. The effect of  $\text{H}_2\text{O}_2$  concentration ( $25\text{-}100 \text{ mg L}^{-1}$ ), initial pH (3-10), initial concentration of phenol ( $50\text{-}200 \text{ mg L}^{-1}$ ), applied current ( $3.5\text{-}7 \text{ mA cm}^{-2}$ ). The schema of the experimental apparatus showed in Fig. 3.

**Analytical methods:** The concentration of phenol was measured using direct photometric method, according to procedure of the standard methods for the examination of water and wastewater (5530 D) via UV-vis spectrophotometer (DR5000, HACH) by measuring the absorption at a wavelength of 500 nm. The obtained data were analyzed by the Excel software.

## RESULTS AND DISCUSSION

**Effect of pH:** The effect of solution pH (3-10) on phenol degradation in EF process was studied and experimental data are shown in Fig. 4. This step of experiments was performed using initial phenol concentration of  $50 \text{ mg L}^{-1}$ ,  $\text{H}_2\text{O}_2$  concentration of  $100 \text{ mg L}^{-1}$  and current density  $5 \text{ mA cm}^{-2}$ . The results show that the solution pH in the phenol degradation was meaningful. The maximum phenol removal (>99.99%) was observed at pH 3. Increasing the pH from 3-10 decreased the efficiency from 100-26%. Also with increasing electrolysis time from 0-60 min, increase removal efficiency. Undoubtedly, the pH is one of the most important factors in the EF process, commonly Fenton and EF process was controlled in acidic solution, controls the electro generation of the hydroxyl

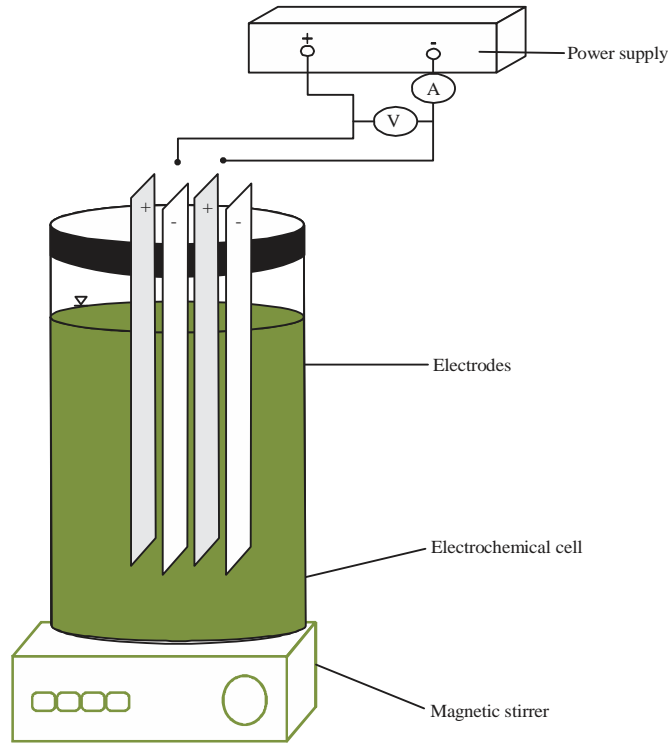


Fig. 3: Experimental setup of electro-Fenton process

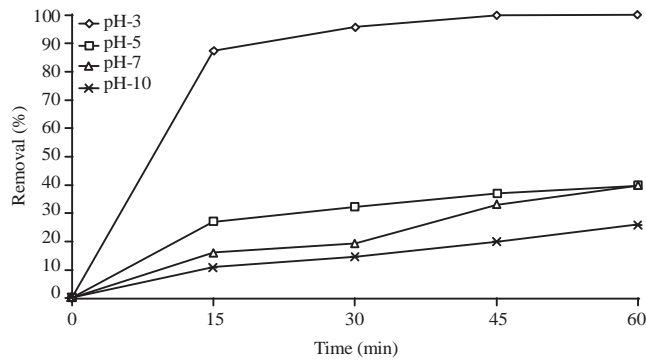


Fig. 4: Effect of pH on phenol removal by electro-Fenton process (operation condition: Initial phenol  $50 \text{ mg L}^{-1}$ ,  $\text{H}_2\text{O}_2$   $100 \text{ mg L}^{-1}$ ,  $\text{Na}_2\text{SO}_4$   $100 \text{ mg L}^{-1}$  and current density  $5 \text{ mA cm}^{-2}$ )

radical and the concentration of ferrous ions conducted with solution pH (Nidheesh and Gandhimathi, 2012; Wang *et al.*, 2008; Zhou *et al.*, 2007). At higher pH, the efficiency of EF process decreases quickly, especially  $\text{pH} > 5$ . This is due to the  $\text{H}_2\text{O}_2$  is unstable and volatility in basic solution,  $\text{H}_2\text{O}_2$  rapidly disintegrate to  $\text{O}_2$  and water (Nidheesh and Gandhimathi, 2012; Shemer and Linden, 2006). Also with an increase in pH decrease in oxidation potential of  $\text{OH}^\bullet$  (Babuponnusami and Muthukumar, 2012) because iron species begin to precipitate as ferric hydroxides complexes (Nidheesh and Gandhimathi, 2012). These complexes would extra when the pH was greater than 9 (Babuponnusami and Muthukumar, 2012). Generally, all researches reported, optimum pH in EF process between 2-4 (Nidheesh and Gandhimathi, 2012; Umar *et al.*, 2010).

**Effect of current density:** The influence of current density ( $3.5\text{-}7\text{ mA cm}^{-2}$ ) on the phenol degradation was studied, experiments was performed using initial phenol concentration of  $50\text{ mg L}^{-1}$  of pH 3,  $\text{H}_2\text{O}_2$  concentration of  $100\text{ mg L}^{-1}$  and the results obtained are shown in Fig. 5. The results showed 100% phenol removal at  $5\text{ mA cm}^{-2}$  after the 45 min of reaction while at  $7\text{ mA cm}^{-2}$  100% degradation was observed in 30 min after of electrolyzing. The applied current in electrolyte, make the reduction of oxygen to the production of hydrogen peroxide at the cathode surface. Higher applied current, increases the quantity of hydrogen peroxide produced, thus increasing the concentration of hydroxyl radicals in the solution (Sankara Narayanan *et al.*, 2003). Increase of applied current density in solution intimate, higher the applied voltage on the electrochemical cell (Wang *et al.*, 2010). Also higher electro-regeneration of ferrous ion from ferric ion with increasing current (Eq. 6) (Nidheesh and Gandhimathi, 2012):



But the efficiency of EF process will be less at highest current density efficiency, this is due to interfere reactions in the electrochemical system, the separation of oxygen at anode (Eq. 7) and the formation of hydrogen at cathode (Eq. 8) happen at a higher current. These reactions interfere with principal reactions such as reactions Eq. 9 and 6 (Nidheesh and Gandhimathi, 2012):



Some studies reported that the current density in the EF process should be ( $6.4\text{-}10\text{ A m}^{-2}$ ) (Nidheesh and Gandhimathi, 2012).

**Effect of  $\text{H}_2\text{O}_2$  concentration:** In this study the  $\text{H}_2\text{O}_2$  is manually added and the effect of  $\text{H}_2\text{O}_2$  concentration on phenol removal efficiency was studied at a constant phenol concentration of  $50\text{ mg L}^{-1}$ , pH 3 and applied current  $5\text{ mA cm}^{-2}$ . The results obtained are shown in Fig. 6. With increase  $\text{H}_2\text{O}_2$  concentration increased the phenol degradation efficiency, the maximum phenol degradation of 100% were obtained with  $100\text{ mg L}^{-1}$  of  $\text{H}_2\text{O}_2$  in 30 min after the reaction. The initial

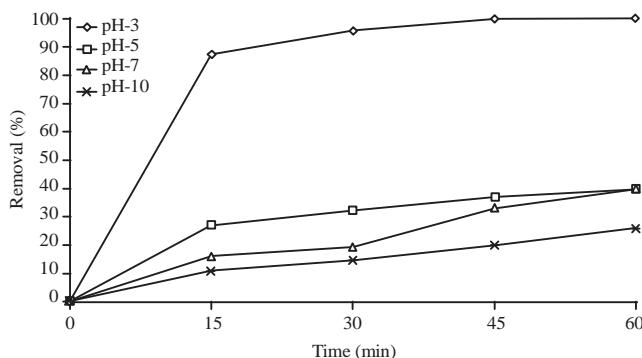


Fig. 5: Effect of current density on phenol removal by electro-Fenton process (operation condition: Initial phenol  $50\text{ mg L}^{-1}$ ,  $\text{H}_2\text{O}_2$   $100\text{ mg L}^{-1}$ ,  $\text{Na}_2\text{SO}_4$   $100\text{ mg L}^{-1}$ , pH 3)

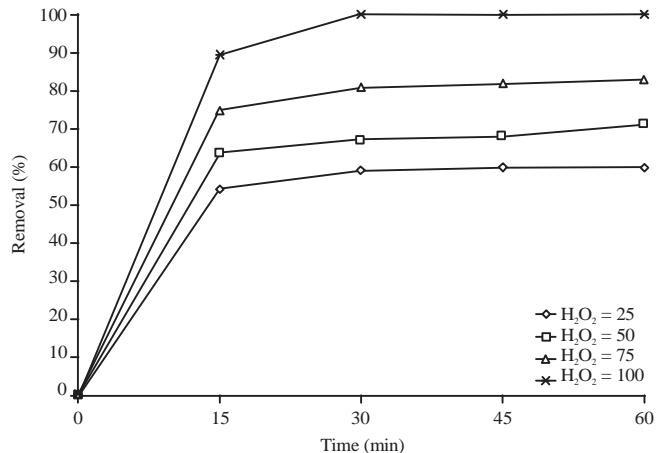


Fig. 6: Effect of H<sub>2</sub>O<sub>2</sub> concentration on phenol removal by electro-Fenton process (operation condition: Initial phenol 50 mg L<sup>-1</sup>, applied current 5 mA cm<sup>-2</sup>, Na<sub>2</sub>SO<sub>4</sub> 100 mg L<sup>-1</sup>, pH 3)

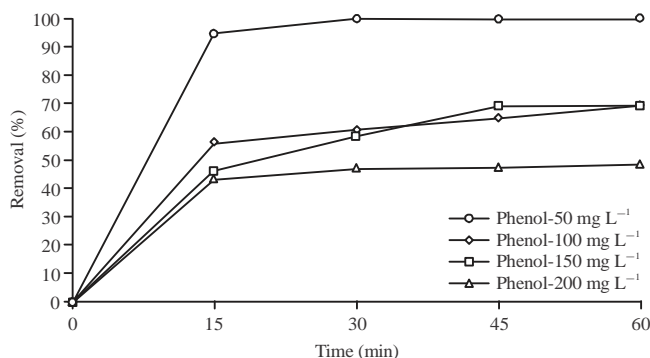


Fig. 7: Effect of initial phenol concentration on phenol removal by electro-Fenton process (operation condition: H<sub>2</sub>O<sub>2</sub> 100 mg L<sup>-1</sup>, applied current 5 mA cm<sup>-2</sup>, Na<sub>2</sub>SO<sub>4</sub> 100 mg L<sup>-1</sup>, pH 3)

concentration of H<sub>2</sub>O<sub>2</sub> plays a critical role in the EF process, removal of pollutants increases with increase in H<sub>2</sub>O<sub>2</sub> concentration. The increase in the removal efficiency was due to the increase in hydroxyl radical concentration as a result of the addition of H<sub>2</sub>O<sub>2</sub> (Eq. 1) (Ting *et al.*, 2009). But at a high concentration of H<sub>2</sub>O<sub>2</sub>, the decrease in removal efficiency was due to the hydroxyl radical scavenging effect of H<sub>2</sub>O<sub>2</sub> (Eq. 10 and 11) (Nidheesh and Gandhimathi, 2012; Ting *et al.*, 2009):



**Effect of initial phenol concentration:** The effect of initial phenol concentration on the removal efficiency of phenol is shown in Fig. 7. These results show that with increase the concentration of phenol from 50-200 mg L<sup>-1</sup>, the removal efficiency decreased from 100-46.85% after 30 min of electrochemical reaction. This is due to the fact that at a constant current density, the same number of iron ions and hydroxyl radical passes to the solution at different phenol concentrations (Babuponnusami and Muthukumar, 2012; Zazouli and Taghavi, 2012).



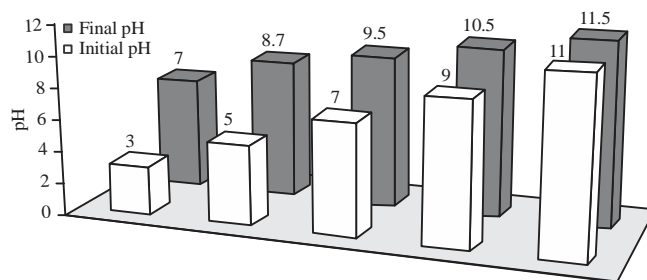


Fig. 8: Effect of EF process on solution pH (operation condition: Initial phenol  $50 \text{ mg L}^{-1}$ ,  $\text{H}_2\text{O}_2$   $100 \text{ mg L}^{-1}$ , applied current  $5 \text{ mA cm}^{-2}$ )

**Effect of Electro-Fenton (EF) process on final pH:** In this step of experiments the effect of EF process on final pH was performed using initial phenol concentration of  $50 \text{ mg L}^{-1}$ ,  $\text{H}_2\text{O}_2$  concentration of  $100 \text{ mg L}^{-1}$  and applied current  $5 \text{ mA cm}^{-2}$ . The results show that the solution pH was increased during the EF process (Fig. 8). The increase in pH during electro-Fenton process leads to electrocoagulation whereby pollutants are removed by electrostatic attraction and/or complexation of reactions due to the conversion of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  to  $\text{Fe}(\text{OH})_n$  (Mollah *et al.*, 2001; Nidheesh and Gandhimathi, 2012).

## CONCLUSION

Experimental data indicated that pH,  $\text{H}_2\text{O}_2$  and phenol concentration have principal role in the process. The results show that removal efficiency of phenol decreased with increasing pH, so that the maximum efficiency 26% was obtained at pH 10, also decreasing of  $\text{H}_2\text{O}_2$  concentration decreased the efficiency. Optimal condition for removing 100% of phenol was found after 30 min of reaction at pH = 3.0,  $\text{H}_2\text{O}_2$  concentration  $100 \text{ mg L}^{-1}$  and  $5 \text{ mA cm}^{-2}$ . Electro-Fenton process using iron electrodes for phenol degradation and remediation of wastewater is a promising process in terms of environmental and low-cost features.

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