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Humic acid removal from aqueous solutions by peroxi-electrocoagulation process

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Abstract

Background: Natural organic matter is the cause of many problems associated with water treatment such as the presence of disinfection by-products (DBPs) and membrane fouling during water filtration. In this study, the performance of the peroxi-electrocoagulation process (PEP) was investigated for the removal of humic acids (HAs) from aqueous solutions.

Methods: PEP was carried out for the removal of HA using a plexiglas reactor with a volume of 2 L and fitted with iron electrodes and a direct current supply (DC). Samples were taken at various amounts of pH (2-4), current density (1 and 2A/cm^2), hydrogen peroxide (50-150 mg/L) and reaction time (5-20 minutes) and then filtered to remove sludge formed during reaction. Finally, the HA concentration was measured by UV absorbance at 254 nm (UV$_{254}$).

Results: Results indicated that increasing the concentration of H_2O_2 from 50 to 150 mg/L increased HA removal efficiency from 83% to 94.5%. The highest removal efficiency was observed at pH 3.0; by increasing the pH to the alkaline range, the efficiency of the process was reduced. It was found that HA removal efficiency was high in current density 1A/cm^2. Increasing current density up to 1 A cm^{-2} caused a decrease in removal efficiency. Results of this study showed that under the optimum operating range for the process ([current density] = 1A/cm^2, [hydrogen peroxide concentration] = 150 mg/L, [reaction time]= 20 minutes and [pH]= 3.0), HA removal efficiency reached 98%.

Conclusion: It can be concluded that PEP has the potential to be utilized for cost-effective removal of HA from aqueous solutions.

Keywords: Humic acid, Peroxi-electrocoagulation, Iron electrode, UV$_{254}$, Water solutions

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Introduction

Humic acid (HA) is one of the major components of humic substances which arise from the microbial degradation of biomolecules. Natural organic materials (NOM) such as HA enter into water from natural and artificial sources. They pose a variety of problems in treatment operations and distribution systems because of their specific attributes including an unpleasant smell and taste, a yellow to brown color, reaction with chlorine, and the formation disinfection by-products (DBPs), eg, trihalomethanes (THMs). These exhibit mutagenic properties during the chlorination step in drinking water production or water treatment.

The guideline for DBPs in drinking water announced by the World Health Organization state that DBPs should not exceed 100 μg/L (1-3). Consequently, the removal of HA from surface water or wastewater is important because of health and environmental concerns. HA may account for up to 90% of NOM. It is not possible to completely remove NOM in conventional water treatment plants (4,5). Different treatment technologies have been used in practice to improve NOM removal. These methods included ion exchange, sorption, membrane processes (such as reverse osmosis), and ultra-filtration, but they either provide only a partial solution or are expensive. Coagulation is a key process which may be carried out in the conventional mode (chemical) or by peroxi-electrocoagulation process (PEP) (6). Thus, PEP has been suggested as an alternative to conventional coagulation because it provides better removal capabilities, produces less sludge, and has a more readily filterable, minimal startup time (7-9).

Some limitations of the PEP are the periodical replacement of sacrificial anodes, minimum solution conductivity, the formation of toxic chlorinated organic compounds from effluent containing chlorides, the formation of an impermeable oxide film on the cathode, and resistance to the flow of electric current, which increases the cost of electricity thus increasing operational costs (10).

Stages in the PEP process include anode dissolution, formation of OH^- ions and H_2 at the cathode, electrolytic reactions at electrode surfaces, adsorption of coagulant on colloidal pollutants, and removal of colloids by sedimentation or flotation. Reactions carried out in the cathode and anode for the production of metal hydroxides include:

Mechanism 1: Anode:

$$4Fe_{(s)} \rightarrow 4Fe^{2+}_{(aq)} + 8e^- \quad (1)$$

$$4Fe^{2+}_{(aq)} + 10H_2O_{(l)} + O_{2(aq)} \rightarrow 4Fe(OH)_{3(s)} + 8H^+_{(aq)} \quad (2)$$

Cathode:

$$8H^+_{(aq)} + 8e^- \rightarrow 4H_{2(g)} \quad (3)$$

Overall:

$$4Fe_{(s)} + 10\,H_2O_{(l)} + O_{2(aq)} \rightarrow 4Fe(OH)_{3(s)} + 4H_{2(g)} \quad (4)$$

In recent years, it has been recommended to combine electrocoagulation with hydrogen peroxide to increase the power of the electrocoagulation process. This method is based on the generation of a powerful oxidant (the hydroxyl radical [$^\bullet OH$]), because the OH radical attacks the organic substrates present in the water and wastewater and then degrades them (11-15). During the electrocoagulation process, Fe^{2+} is the common ion generated at the anode; in contrast, OH^- ions are produced at the cathode (16-19). By adding hydrogen peroxide into the PEP reactor, Fenton reactions are carried out in accordance with the following:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + {}^\bullet OH \quad (5)$$

$$Fe^{3+} + H_2O_2 \leftrightarrow Fe\text{-}OOH^{2+} + H^+ \leftrightarrow Fe^{2+} + HO_2^\bullet + H^+ \quad (6)$$

Vasudevan (20) studied the removal of phenol from water by the PEP. Their results showed that phenol was removed by PEP at 92% efficiency. Evrim Yüksel et al (21) achieved a removal efficiency of 81.6% in the removal of sodium dodecyl sulfate from synthetic wastewater by the peroxi-electrocoagulation method. Vasudevan and Lakshmi (22) also studied the removal of boron from water by electrochemical processes and investigated various operating parameters such as initial boron ion concentration, initial pH, current density, and temperature. Their results showed a removal efficiency of 93.2% under optimum operating conditions. Kamaraj et al (23) studied the adsorption of 2,4-dichlorophenoxyacetic acid (2,4-D) from water by in situ generated metal hydroxides using sacrificial anodes. The results showed that the maximum removal efficiency of 91.0% was achieved with iron as the anode. The current study examined the possibility of HA removal from aqueous solutions by PEP and evaluated the influence of operation parameters such as initial pH, current density, amount of hydrogen peroxide, and reaction time on HA removal efficiency.

Methods

Material

All chemical reagents used in bench-scale tests were analytical grade. The materials used in this study included sulfuric acid (96%) and NaOH (98%) for pH adjustment and technical grade hydrogen peroxide (30% w/w and density of 1.13 kg/L), purchased from the Merck company.

Set-up and Procedure

A stock solution of HA was prepared by dissolving HA powder in 100 mL NaOH (0.1N). The prepared solution was kept for a day, and then its volume was increased to 1 L with distilled water. The solution was filtered using a Whatman filter, which was then placed in an oven at a temperature of 103°C-105°C. The difference between the primary and secondary weight of the filter was determined to be 314.5 mg/L. Working solutions (4, 8, and 10 mg/L) were prepared by diluting the stock solution in deionized water.

The batch experimental setup is schematically shown in Figure 1. Water was electrochemically oxidized in an undivided cell reactor equipped with a magnetic stirrer using a constant speed of 200 rpm in order to get a correct homogenization of the wastewater-floc mixture. The reactor consisted of an EC cell, a DC power supply, and monopolar electrodes in parallel connections. All experiments were run under isoperibolic conditions and at a temperature of 20ºC. The reaction was timed starting when the DC power supply was switched on. Two flat plates with smooth surfaces were connected as cathodes and 2 as anodes. Each electrode measured 4 cm × 3 cm, and the distance between the iron electrodes was 1 cm. To remove the influence mass transfer rate of pollutants to the electrodes (iron salts produce electrode passivation and cause a 50% increase in treatment time and power requirements), high mixing was ensured by a magnetic stirrer. To maintain

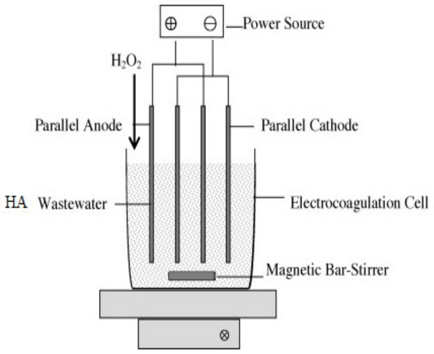

Figure 1. A schematic diagram of the electrochemical reactor.

the same experimental conditions, the Fe electrodes were polished with grinding papers and then rinsed with soapy water and deionized water after each experiment.

The effects of the main operational variables (including solution pH [2-4], current density [1 and 2 A/cm^2], reaction time [5–20 minutes], and H_2O_2 [50-150 mg/L]) on the electrochemical process in treating HA were investigated in various runs. At the beginning of a run, the HA solution was fed into the reactor; then the electrodes were connected to the DC power supply and the reaction was started while the cell content was gently stirred (200 rpm). H_2O_2 was added at desired rates to the reactor for enhanced pollutant removal.

Analytic Procedure

The HA samples were measured by HACH spectrophotometer (DR 5000) at a wavelength of 254 nm after filtration through a Millipore membrane filter with a 0.45 µm pore size. Experiments with 3 replications were conducted according to the standard methods for examination of water and wastewater (24). Data was analyzed using Excel software.

Results

To determine the optimal amount of H_2O_2, concentrations in the range of 50 to 150 mg/L were investigated, and the results are presented in Figure 2. It was observed that increasing the concentration of H_2O_2 increased the removal of HA. For example, in the initial HA concentration of 4 mg/L, increasing the H_2O_2 concentration from 50 to 150 mg/L increased HA removal efficiency from 83% to 94.5% at pH 3.0 and current density 1A after 20 minutes.

pH plays a significant role in the performance of the Fenton process. Various studies have shown that an acidic pH of 2-4 is the best range for the Fenton reaction; therefore, the performance of PEP for the removal of HA was investigated in a pH range of 2-4 (Figure 3). The highest removal efficiency was observed at pH 3.0; increasing pH to the alkaline range reduced the efficiency of the process.

The results of the influence of the current density on the HA removal are presented in Figure 4. It was found that the HA removal efficiency was high in current density 1A/cm^2. Increasing current density up to 1A/cm^2 caused a decrease in removal efficiency. The minimum energy consumption, was 10 kWh (kg HA)$^{-1}$ at current density 1 1A/cm^2 for a 20 minutes reaction time.

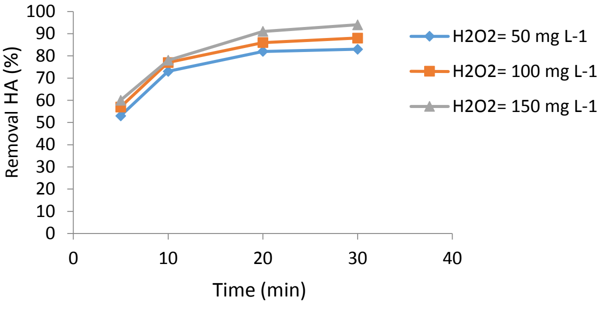

Figure 2. Effect of hydrogen peroxide on HA removal efficiency (peroxi-electrocoagulation process [PEP]; initial HA, 4 mg/L; pH, 3; current density, 1A/cm^2; distance between plates, 1 cm).

As shown in Figure 5, early on, the pollutant decomposition rate was very high, and increasing reactive time did not significantly affect the removal of HA. The effects of initial HA on removal efficiency are shown in Figure 6. Results of this study confirm that increasing the HA concentration increases the efficiency of the electrochemical process.

Discussion

Results of the current study indicated that increasing the H_2O_2 concentration increased HA removal efficiency. Determining the optimal amount of hydrogen peroxide in PEP is very important and necessary because of: (*a*) the high cost of hydrogen peroxide in high concentrations; (*b*) reduced production of hydroxyl radical exceeding H_2O_2

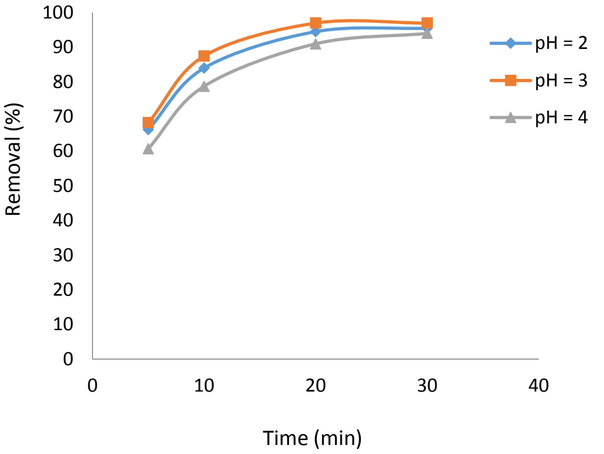

Figure 3. Effect of initial pH on HA removal efficiency (peroxi-electrocoagulation process [PEP]; initial HA, 4 mg/L; hydrogen peroxide, 150 mg/L; current density, 1A/cm^2; distance between plates, 1 cm).

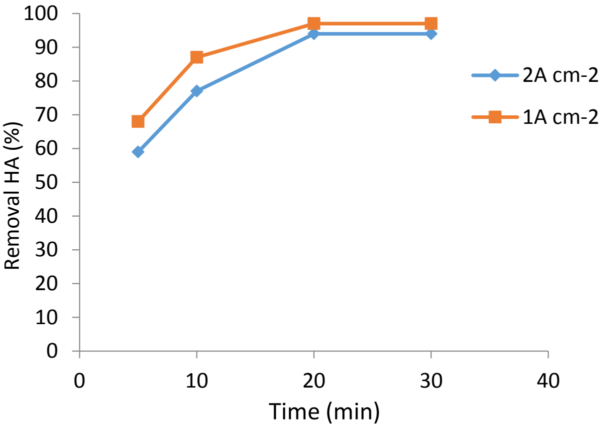

Figure 4. Effect of current densityon HA removal (initial HA, 4 mg/L; pH, 3; H_2O_2, 150 mg/L; distance between plates, 1 cm).

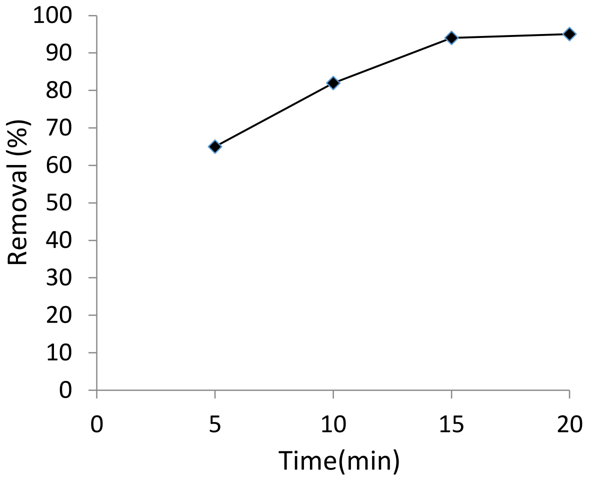

Figure 5. Effect of reaction time on HA removal efficiency (peroxi-electrocoagulation process [PEP]; initial HA, 4 mg/L; hydrogen peroxide, 150 mg/L; pH, 3; current density, 1A/cm²; distance between plates, 1 cm).

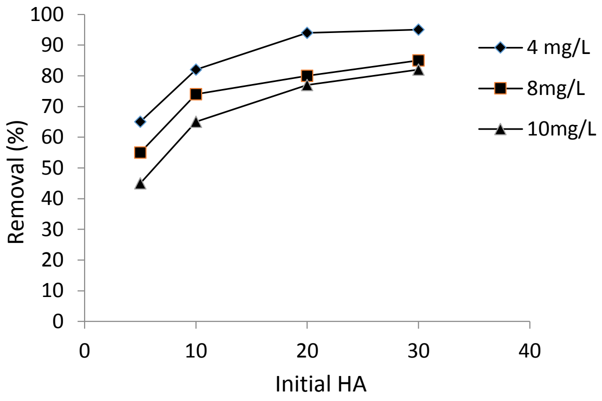

Figure 6. Effect of initial HA (peroxi-electrocoagulation process [PEP]; hydrogen peroxide,150 mg/L; Initial HA, 4-10 mg/L; pH, 3; current density, 1A/cm²; distance between plates, 1 cm).

(H_2O_2 acts as a scavenger of the OH according to reaction 7); and (*c*) increased chemical oxygen demand by residual H_2O_2 (15,16,25,26).

$$H_2O_2 + {}^{\bullet}OH \rightarrow H_2O + HO_2^{\bullet} \quad (7)$$

It can be concluded that the removal of HA increases when the concentration of hydrogen peroxide is increased. Mahesh et al (26) examined the effects of different concentrations of hydrogen peroxide on the reduction of humic substances from leachate using the Fenton oxidation process and chemical coagulation. They concluded that the removal of humic substances increases when the concentration of hydrogen peroxide is increased to a certain concentration due to the production of hydroxyl radicals. Increasing the hydrogen peroxide concentration over this amount caused a reduction in removal efficiency. They also determined that the decreased removal efficiency in excessive amounts of hydrogen peroxide can be caused by the production of OOH radicals that consume the OH. Farhadi et al (27) used PEP for COD removal from pharmaceutical wastewater. Their results showed that the maximum removal rate was nearly 42.2% for the optimum level of 300 mg/L H_2O_2 after 20 minutes of electrolysis time for the voltage of 40 V.

The decrease in removal efficiency in alkaline pH is probably due to the formation of ferric species and decreased production of ferrous ions, both of which reduce the production of hydroxyl radicals and so decrease the efficiency of removal. A decrease in the efficiency of HA removal at pH <3 could be explained by the following:

1. $Fe(OH)^{2+}$, $Fe(OH)_2^{+}$, and $Fe_2(OH)_2^{4+}$ were produced, which react more slowly with H_2O_2.
2. The scavenging effect of OH by H^+ becomes significant at very low ranges of pH (at lower pH the protons in the solution are reduced to H_2 at the cathode, and the ions of H^+ may inhibit the generation of hydroxyl and perhydroxyl radicals) (28).

$${}^{\bullet}OH + H^+ + e^- \rightarrow H_2O \quad (8)$$

Farhadi et al (27) used PEP for COD removal from pharmaceutical wastewater. The optimum pH for this process was determined to be 3.0. Su et al (29) used electro-Fenton processes for the degradation of acetaminophen (ACTP). The experimental results showed that the electro-Fenton processes had maximum ACTP degradation efficiencies of 100%. ACTP degradation efficiency increased 74% at pH 4 in the electro-Fenton process. Cheng et al (30) used the electro-Fenton process with a Ce/SnO_2–Sb-coated titanium anode for the mineralization of metronidazole. A decrease in pH from 7.0 to 2.0 increased removal efficiency from 10% to 37%.The optimum pH was found to be about 2.0.

Current density is an important factor in the electrochemical processes because of its effects on the coagulant dosage rate, bubble production rate, size and growth of the flocs. Increasing the current density increased HA removal. This is in accordance with Faraday's law; with an increase in current density and reaction time, the anodic dissolution rate increases that will give rise to Fe^{2+} being released from the anode as in equation 9 (31,32).

$$C_{Fe} = \frac{M_w I t_{EC}}{ZFV} \quad (9)$$

where C_{Fe}, Z, F, V, and M_w are the theoretical concentration of Fe^{2+} (g/m³), the chemical equivalence, Faraday's constant (96,487 C/mol), reactor volume (m³), and molecular weight of iron (g/mol), respectively. The increase of removal efficiency upon increasing current density is the result of the extent of anodic dissolution of iron, increased hydroxyl radical generation, increased bubble generation rate, and decreased bubble size. These trends are beneficial in terms of high pollutant removal efficiency by H_2 flotation (33). Increasing current density up to 1A/cm² caused a decrease in the efficiency of removal. This decrease could be due to the decomposition of hydrogen peroxide in higher current densities. Wang (34) studied COD removal from real dyeing wastewater by electro-Fenton technology using an activated carbon fiber cathode. In this study, 75% COD removal was achieved under the optimum conditions of current density= 32 mA/cm^2, pH = 3.0, Fe^{2+}= 2 mM/L, and T= 20°C (34). Vasudevan et al (35) studied the removal of copper by electrochemically-assisted coagulation. Their results showed that the optimum removal efficiency of 96.6% was achieved at a current density of 0.05 A/dm^2 and a pH of 7.0. Babuponnusami and Muthukumar (36) used the electro-Fenton process for the advanced oxidation of phenol. Their results showed 62.5% phenol degradation for the EF process at 16 mA/cm^2 in 60 minutes. Increasing current density from 0 to 16 mA/cm^2 offered faster phenol degradation and COD removal. The important economical parameter in PEP is electrical energy consumption which is the major operating cost of the electrochemical process. The minimum amount of energy consumption was 10 kWh (kg HA)$^{-1}$ at a current density of 1A/cm^2 for a 20 minutes reaction time that was calculated using the following equation:

$$E = UIt_{EC} \tag{10}$$

where E is the electrical energy in Wh, U= the cell voltage in volt (V), I= the current in ampere (A), and t_{EC} is the time of EC process per hour.

Reactive time influences the treatment efficiency of PEP. To explore the effect of operating time, the optimal conditions of these processes were used, and all processes were started simultaneously. Early on, the pollutant decomposition rate was very high, possibly due to the appropriate amounts of Fe (II) and hydrogen peroxide used to produce hydroxyl radicals. It was observed the percentage of HA removal was higher for PEP at 20 minutes, pH= 3.0, and current density 1A/cm^2. The decrease of HA as a function of elapsed time might be caused by the production of Fe^{2+} or Fe^{3+} ions from iron and the formation of OH, because by adding H_2O_2 into the system, hydrogen peroxide is catalyzed by ferrous ion to produce hydroxyl radicals, and a reaction is propagated from ferrous ion regeneration mainly by the reduction of the produced ferric species with hydrogen peroxide. Therefore, the dosage of H_2O_2 could enhance the performance of these processes. Ruppert et al (37) investigated the mineralization of several organic matters by hydroxyl radicals. They reported that organic material only affected the behavior of ferrous ions. Ferrous ions compete with organic material for the use of hydroxyl radicals. At low concentrations of organic material, ferrous ions react with hydroxyl radicals and reduce the amount of them in the environment. In the absence of organic compounds, hydrogen peroxide is decomposed slowly by ferric ions. Excessive amounts of organic matter prevent the reaction between ferrous ions and hydroxyl radicals with the increase in reactivity between ferrous ions and hydrogen peroxide.

Conclusion

The PEP was tested on HA synthetic wastewater, and the effects of operational parameters such as pH, amount of hydrogen peroxide, current density, and reaction time on HA removal efficiency were investigated. The HA removal efficiency that was achieved using PEP was 98% for the initial HA concentration of 4 mg/L. The ferric hydroxide and hydroxyl radical generated in the cell can decrease the HA present in the wastewater. The optimal conditions for the process were determined to be current density = 1A/cm^2, hydrogen peroxide concentration= 150 mg/L, reaction time= 20 minutes, mean energy consumption= 10 kWh (kg HA)$^{-1}$, and pH=3.0. Finally, given the results ultimately obtained in this research, it became clear that the process is technologically feasible.

Ethical issues

Ethical issues (including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, redundancy, etc.) have been completely observed by the authors.

Competing interests

The authors have declared that no competing interests exist.

Authors' contributions

All authors contributed equally, and all authors participated in the data acquisition, analysis, and interpretation. All authors critically reviewed, refined, and approved the manuscript.

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