

Letter to Editor

# Experimental Investigation of Chemical Oxygen Demand, Lignin and Phenol Removal from Paper Mill Effluents Using Three-Phase Three-Dimensional Electrode Reactor

M. Uğurlu\*, M. H. Karaoğlu, İ. Kula

Department of Chemistry, Faculty of Science and Arts, Muğla University, Muğla 48000, Turkey

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## Abstract

The removal of chemical oxygen demand (COD), lignin and phenol from paper mill effluents was experimentally investigated using an electrode cell consisting of graphite electrode and powder-activated carbon (as working electrode). The effects of electrolyses time, voltage, initial pH, activated carbon (AC), NaCl amount and airflow were selected as parameters. The experimental results show that removal efficiency significantly depends on the applied cell voltage, airflow, time, salt amount and pH. For electrolyses under the conditions of 25.0V cell voltage, 5.0 min, in presence of air 2.0 minL<sup>-1</sup>, NaCl 5.0gL<sup>-1</sup> at low pH and AC 20gL<sup>-1</sup>, it can be said that the removal of COD, lignin and phenol from the paper mill effluent were almost higher than 90%. The present study proves the effectiveness of electrochemical treatment for the highly concentrated organic pollutants present in paper mill effluents.

**Keywords:** wastewater, electrochemical method, phenol, lignin, COD, AC

**Nomenclature:** AC – activated carbon, COD – chemical oxygen demand (mg l<sup>-1</sup>), BOD – biochemical oxygen demand (mg l<sup>-1</sup>)

**Subscripts:** Cl<sup>-</sup> – chlorine ion, H<sup>+</sup> – hydrogen ion, H<sub>2</sub>O<sub>2</sub> – hydrogen peroxide, HOCl – hypochlorite, MO<sub>x</sub> – oxide anode, MO<sub>x+1</sub> – chemisorbed active oxygen, MO<sub>x</sub>[•OH] – adsorbed hydroxyl radical, •O – active oxygen, •OH – hydroxyl radical, OH<sup>-</sup> – hydroxyl ion, R – organic matter, RO – oxidized organic matter

## Introduction

The pulp and paper industry is the sixth largest polluter (after the oil, cement, leather, textile and steel industries), discharging a variety of gaseous, liquid and solid wastes into the environment. It is the pollution of water bodies, however, which is of major concern because large volumes of wastewater are generated per metric ton of paper

produced, depending upon the nature of the raw material, finished product and extent of water reuse.

Since the pulp produced corresponds to only approximately 40-50% of the original weight of the wood, the effluents are heavily loaded with organic matter. These effluents cause considerable damage to receiving waters if discharged untreated since they have a high biochemical oxygen demand (BOD), chemical oxygen demand (COD), phenol compounds, chlorinated compounds, suspended solids, fatty acids, tannins, lignin and its derivatives [1, 2].

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\*Corresponding author; e-mail: mnazlican@hotmail.com

Some of the pollutants listed above, notably phenol compounds, dioxin, furans, lignin and derivatives, have been classified as "priority pollutants" by the United States Environmental Protection Agency (EPA, 1998-5). It is well established that many contaminants are acute or even chronic toxins. Chlorinated organic compounds, which include dioxins and furans, have the ability to induce genetic changes in exposed organisms [3].

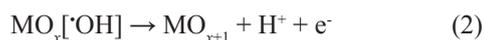
Several physical and chemical processes for colour removal in paper mill effluents have been extensively studied, including rapid filtration through soil, ultra filtration, ion-exchange chromatography, lime precipitation and modified bleaching sequences, such as peroxide addition during extraction, the replacement of chlorine by hypo chlorite, sorption on hypo-and alum-sludge, activated carbon and on allophonic compounds [4-7]. Moreover, the problem remains unsolved, since lignin undergoes a spatial rather than chemical change and thus persists albeit in a different form. Wastewaters are usually purified by conventional methods such as biological oxidation, adsorption, flotation, coagulation, UV photodecomposition and electrochemistry [2, 8]. Electrochemical technology offers ideal tools for addressing environmental problems. The main reagent used here is electron, which is a clean reagent and therefore requires no need for adding extra reagent [9, 10].

### Theories of Electrochemical Treatment

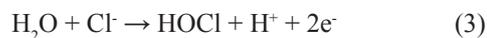
Generally, oxidation of organic matter by electrochemical treatment can be classified as direct oxidation at surface of anode and indirect oxidation distant from the anode surface; processes are influenced significantly by the anode material. Recently, oxide anodes have been of interest because of higher conductivity and oxidizability. The mechanism of oxidation of organic matter at oxide anode ( $MO_x$ ) has been suggested by Comninellis [11]. Water is electrolyzed by anodic catalysis to produce adsorbed hydroxyl radicals, given as Eq. (1):



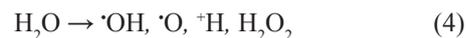
The adsorbed hydroxyl radicals may form chemisorbed active oxygen, as shown in Eq. (2):



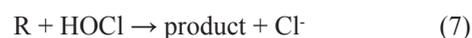
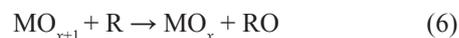
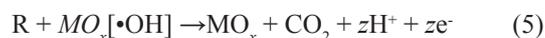
Furthermore, another strong oxidant of hypochlorite may be produced in many wastewaters containing chlorides, as given in Eq. (3) [12]:



In addition, high voltage pulse can lead to the formation of a strong electric field and free radicals such as  $\cdot OH$ ,  $\cdot O$ ,  $H^+$  and  $H_2O_2$ , etc. [13]:



Organic matter (R) included in wastewater are oxidized by hydroxyl radicals and the reactions are given in Eqs. (1)–(4) [12]:



Oxidation of organic matter depends on the anode material, concentration of NaCl, and current and voltage applied. Although the influence of NaCl and anode material has been investigated in the electrochemical treatment applied DC power supply, the effect of anode material on pulse treatment has not yet been studied.

Over the recent years there has been a growing interest in the development of efficient electrochemical methods for the destruction of toxic organic contaminants of wastewater [14]. Such methods have been successfully tested to deal with various industrial wastewaters [15-21]. However, for some dyes and organic molecules which have high water solubility and a low molecular weight, traditional electrochemical methods do not work efficiently [22]. The three-dimensional electrode is rather attractive for industrial application since it can provide a more extensive interfacial electrode surface area compared with the two-dimensional electrode [14]. The three-dimensional electrode is particularly appropriate to the treatment of low concentration solutions [23, 24]. In this paper we focused on investigating the treatment of paper mill effluents using a cell with a three-phase (solid, liquid and air) three-dimensional electrode using activated carbon as filler (working electrodes) coupled with coagulant as a pre-treatment. Phenol, lignin and chemical oxygen demand (COD) removal from paper mill effluents were selected in this investigation because phenol, lignin and COD are among the largest-group of toxic and colorants discharged in paper producing. However, it has never before been attempted to use it for dealing with paper mill effluents.

### Material and Methods

#### Materials

A commercially activated carbon obtained from CARLO ERBA was used in this study. It has an average pore size of approximately  $1.5\text{cm}^3\text{g}^{-1}$  and a specific surface of  $1500\text{m}^2\text{g}^{-1}$ , according to the BET method. The paper mill effluents utilized for this study were taken from a modern bleached Kraft mill located in western Turkey. Wastewater ( $4500\text{m}^3/\text{h}$ ) is discharged into Gökova gulf (Muğla, Turkey) by deep discharging after being collected in sedimentation pools and aerated for about a week. Wastewater

samples used in this study taken from aerated logons right before sea discharging process. The effluent characteristics are given in Table 1.

### Experimental Method

In the experimental apparatus, graphite was used as electrode. The experimental apparatus is a batch rectangular undivided three-phase three-dimensional electrode reactor as shown in Fig.1. The reactor support was made from plastic. The anode and cathode (feeder electrodes) were situated 2.0 cm apart from each other. Stirring was done by using a magnetic stirrer (7 rpm). Compressed air was spared into the bed electrodes from the bottom of the reactor.

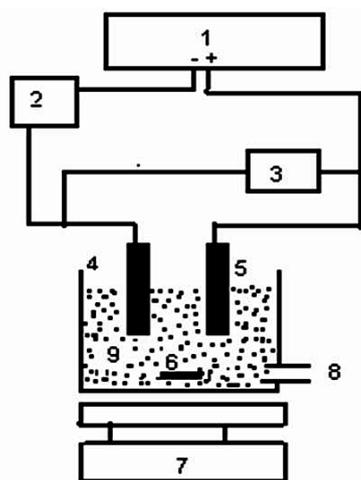


Fig. 1. Experimental apparatus: 1. DC Power Supply, 2. Digital Ampermeter, 3. Digital Voltmeter, 4. Anode, 5. Cathode, 6. Magnetic bar, 7. Magnetic Stirring Controller, 8. Compressed air, 9. Bed electrode (working electrode)

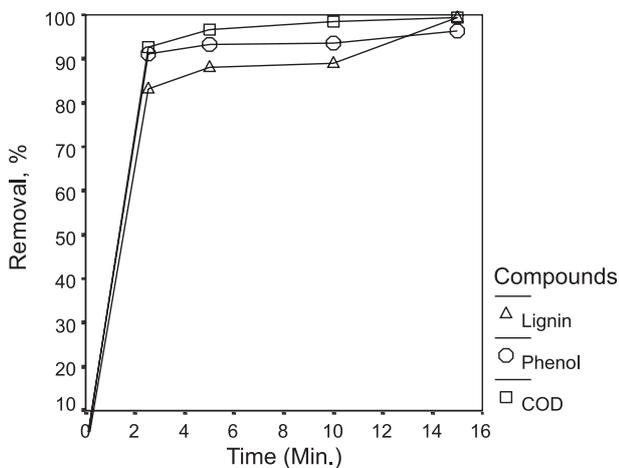


Fig. 2. Effect of electrolysis time on removal percentage of phenol, COD and lignin by using AC (operating condition: airflow: 2.0min<sup>-1</sup>, cell voltage: 25V, salt: 2gL<sup>-1</sup>, AC: 20gL<sup>-1</sup> and pH: 8.0)

Cell voltage was measured digitally. As power supply, Topward Dual-Tracking DC 6303D and as multimeter, Keithley 2010 was used. Electrolyses time, cell voltage and initial pH, airflow, NaCl and AC amount were selected as parameters. At the end of experiments the treated solution was filtered and then the concentration of phenol, COD and lignin before and after electrolysis were measured using the standard method [25].

### Results and Discussion

#### Effect of Treatment Time

In aqueous medium AC has considerable electrochemical activity in addition to its strong adsorption capacity to a lot of pollutants [26, 27]. In the present study, the AC was employed as a bed electrode (working electrode) material. The efficiency of lignin, phenol and COD removal at different electrolysis times is presented in Fig 2.

Lignin, phenol and COD removal were observed to increase rapidly during the first 2 min in Fig 2. Then the increases slow and do not improve much beyond the 15 min treatment. The apparent kinetics of COD, lignin and phenol removal can be expressed as the following pseudo-first-order rate equation:

$$\ln \frac{[COD]_t}{[COD]_0} = -kt \quad \text{or} \quad \ln \frac{[C]_t}{[C]_0} = -kt \quad (8)$$

The slope of the plot of  $\ln C_t/C_0$  versus time gives the value of rate constant  $k$ , min<sup>-1</sup>. Here,  $C_0$  is the initial COD, lignin and phenol of the effluent in milligrams per litre, and  $C_t$  is the COD, lignin and phenol of the treated solution at the electrolysis time  $t$  min. The kinetic statements written for COD can also be written for both phenol and

Table 1. Characteristics of bleached Kraft mill effluents.

Parameter	Value
Colour	Brown
pH (average)	8.0
Compounds (mg/L)	
PO <sub>4</sub> <sup>3-</sup>	0.277
NO <sub>2</sub> <sup>-</sup>	0.213
NO <sub>3</sub> <sup>-</sup>	2.65
NH <sub>4</sub> <sup>+</sup>	0.595
Phenol	3.19
Lignin	5.49
BOD (Biological Oxygen Demand)	25.5
COD (Chemical Oxygen Demand)	1633.0

lignin. Table 2 provides the rate constant ( $k$ ) and  $r^2$  values for COD, phenol and lignin removal.

### Effect of Initial pH

The initial pH of wastewater is an important operating factor influencing the performance characteristics of the electrochemical process. To demonstrate its effect on treatment efficiency, the pH of wastewater was adjusted to between 2 and 12 by using NaOH and HCl. The experimental results at various initial pH values are shown in Fig. 3. It can be seen from the figure that the highest removal percentage was observed at the pH 2.0; with increasing pH, a decrease in the removal percentage of pollutants was observed. These results can be explained through the following factors. A high amount of  $\text{Cl}_2$  was used for bleaching at the production of paper and as a result the chlorinated organic compounds occur in the paper mill effluents [1]. The chlorinated phenol and lignin compounds are non-polar molecules. It can be explained that these molecules were adsorbed at the surface of AC because of negative charge and were reduced by indirect electrochemicals in the presence of chlorine/hypochlorite.

Table 2. Kinetic summary of COD, phenol and lignin removal by the electrochemical methods.

Compounds	Rate constant $k$ ( $\text{min}^{-1}$ )	$r^2$
COD	0.2388	0.97
Lignin	0.3681	0.94
Phenol	0.0460	0.96

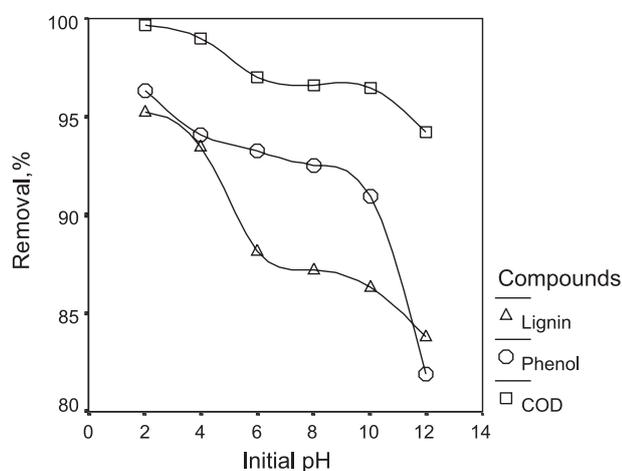


Fig. 3. Effect of initial pH on removal percentage of phenol, COD and lignin (operating conditions: airflow:  $2.0\text{minL}^{-1}$ , cell voltage: 25V, salt:  $2\text{gL}^{-1}$ , AC:  $20\text{gL}^{-1}$  and time: 5min).

### Effect of AC Amount

The effect of the removal of AC amount was investigated at the same electrochemical cell as working electrode and at different amounts. The obtained results were shown in Fig. 4. From this figure it is seen that when AC and electrochemical methods were used together in the same electrochemical cell, respectively COD, phenol and lignin removal percentages were approximately 95%, 90% and 85% (for  $1.0\text{g}/50\text{mL}$ ). These removal percentages are attributed to the increasing AC amount as working electrode, in addition to the adsorption property of AC.

### Effect of Voltage

The effect of voltage was observed by previous investigators to influence the treatment efficiency of the electrochemical process in different wastewaters [28, 29]. With sufficient power supply the phenol-like organic compounds are reduced at the cathode to smaller molecules [22,30]. The present study was carried out to investigate the effect of voltage on COD, phenol and lignin removal efficiency in electrochemical cell (Fig 5). From Fig. 5 the removal of COD, phenol and lignin was directly proportional to the applied voltage as might have been expected in any other electrochemical process. While the removal percentages were observed decreasing at low voltage, it is apparent that the high applied voltage resulted in high efficiency of COD, phenol and lignin removal percentages. An increase in the applied voltage from 15.0 V to 25.0 V didn't reveal a significant enhancement in the amount of pollutants. The removal percentages were realized to be approximately 85% for lignin, 90% for phenol and 95% for COD between these two voltages.

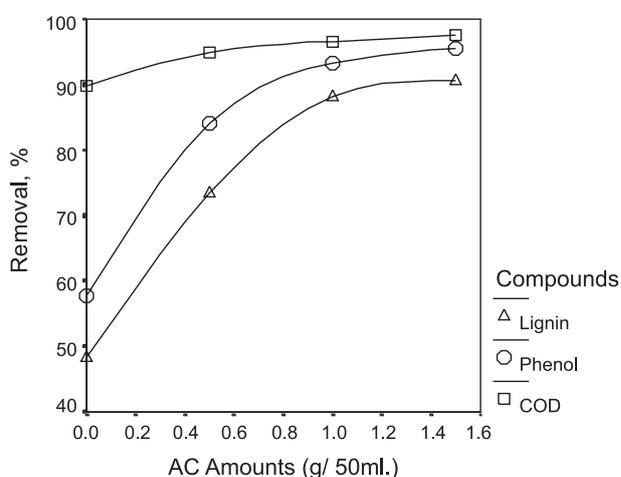


Fig. 4. Effect of using AC amount on removal percentage of phenol, COD and lignin (operating conditions: airflow:  $2.0\text{minL}^{-1}$ , voltage: 25V, salt:  $2\text{gL}^{-1}$ , AC:  $20\text{gL}^{-1}$ , pH: 8.0 and time: 5min).

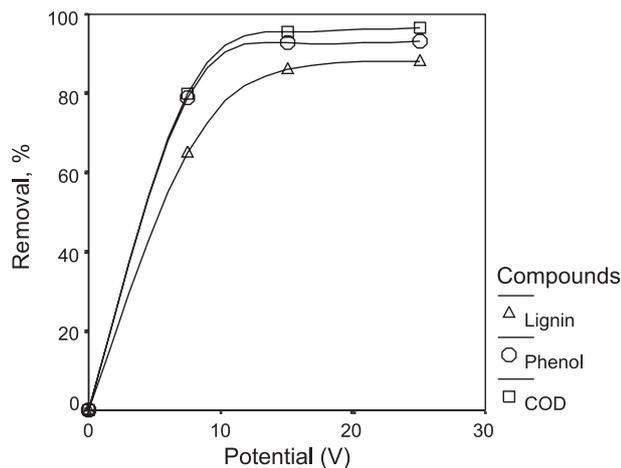


Fig. 5. Effect of cell voltage on removal percentage of phenol, COD and lignin (operating conditions: airflow:  $2.0\text{min}^{-1}$  salt:  $2\text{gL}^{-1}$ , AC:  $20\text{gL}^{-1}$ , pH: 8.0 and time: 5min)

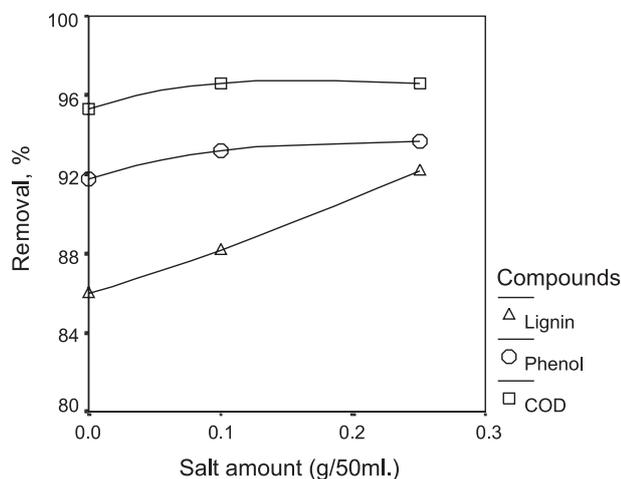


Fig. 6. Effect of electrolyte on removal percentage of phenol, COD and lignin (operating conditions: airflow:  $2.0\text{min}^{-1}$ , cell voltage:  $25\text{V}$ , AC:  $20\text{gL}^{-1}$ , pH: 8.0 and time: 5min)

### Effect of NaCl

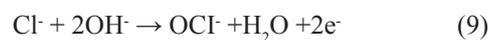
The study of the effect of salt concentration is important because it determines the electrolytic current. The enhancement of electrolytic current can improve not only the degradation of wastewater but also some side reaction such as producing  $\text{H}_2$  at cathode [31]. The results obtained in this study at varying NaCl amounts for lignin, phenol and COD removal in paper mill effluent are shown in Fig 6.

It can be seen from Fig. 6 that removal efficiencies were increased by increasing NaCl. As the amount of NaCl was increased, lignin removal efficiency increased significantly. However, it was observed that COD and phenol removal efficiency was constant  $1.0\text{gL}^{-1}$  in the same experiments. It was stated that salt was usually employed to increase the conductivity of the water or wastewater to be treated. The addition of NaCl would also lead the decrease in power consumptions because of the increase

in conductivity [32]. The main reactions occurring during the anodic oxidation of organic compounds in the presence of NaCl are [32]:

#### Anode

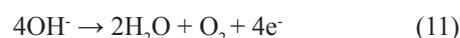
Hypochlorite formation:



Chlorate formation:



Oxygen evolution:



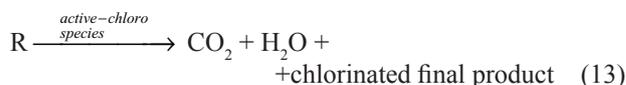
#### Cathode

Hydrogen evolution:



#### Solution and/or near the anode surface

Indirect oxidation of organic compound and its oxidation intermediate with hypochlorite:



#### Effect of Airflow

During electrolyses the compressed air was sparged into the electrochemical reactor. The sparged air serves two purposes: to agitate with magnetic stringer in order to speed mass transfer, and to supply the essential oxygen for electrochemical reaction. Some authors have reported that the oxygen can be changed into a strong oxidant agent,  $\text{H}_2\text{O}_2$ , on activated carbon electrodes by the two-electron reduction of oxygen [14, 33]. When oxygen was used in the electro oxidation procedures, electrodes can simultaneously make use of anodic oxidation and cathodic-electro-generated  $\text{H}_2\text{O}_2$  to degrade organic pollutants. As a result, it is expected that the compressed air will play an important role in the degradation of phenol, lignin and COD removal.

As shown in Fig. 7, the COD, phenol and lignin removal percentages all increase with airflow. COD, phenol and lignin removal reached, respectively, 97%, 95% and 90% in the presence of air. The enhancement effect could be contributed to a combination of the versatile removal routines of a three-phase three-dimensional electrode reactor. In addition, phenol and lignin on anode could be directly transformed into  $\text{CO}_2$  and  $\text{H}_2\text{O}$  or into polymers [34], the cathodic electrogenerated  $\text{H}_2\text{O}_2$  could also partly degrade phenol and lignin by chemical oxidation pathway [31] and

Table 3. Summary of energy consumption by electrochemical treatment (electrolyte: 2gL<sup>-1</sup>, cell voltage: 25V, AC: 20gL<sup>-1</sup>, pH: 8.0 and time: 15 min.).

Compounds	Initial Conc. (mg/L)	Final conc. (mg/L)	kWh/m <sup>3</sup> of effluent
COD	1633	49	12.5
Lignin	5.49	0.71	12.5
Phenol	3.19	0.22	12.5

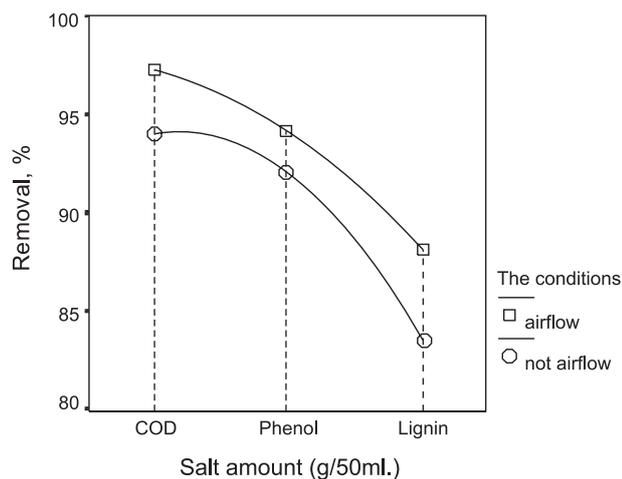


Fig. 7. Effect of airflow on removal percentage of phenol, COD and lignin (operating conditions: electrolyte: 2gL<sup>-1</sup>, cell voltage: 25V, AC: 20gL<sup>-1</sup>, pH: 8.0 and time: 5min).

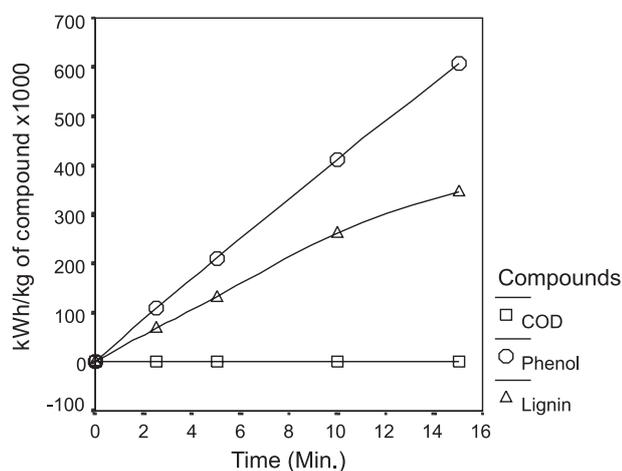


Fig. 8. Energy consumption on removal percentage of phenol, COD and lignin (operating conditions: electrolyte: 2gL<sup>-1</sup>, cell voltage: 25V, AC: 20gL<sup>-1</sup> and pH: 8.0).

the stripping-off might also partly remove the organic pollutant. However, it is difficult to calculate how much contribution each reaction makes to the enhancement effect.

### Energy Consumption

Considering the initial and final concentrations of compounds in wastewater at different times, electric energy (EE) consumed to remove 1 kg of each compounds (phenol, lignin and COD) was calculated with the following equation [35]:

$$EE \text{ (kW-h/kg of compound)} = \frac{V \times I \times t \times 1000}{60 \times (C_0 - C_t)}$$

$V$ : voltage (volt),  $I$ : current amount (ampere),  $t$ : electrolysis time (min.),  $C_0$ : initial concentrations (gL<sup>-1</sup>),  $C_t$  are the concentration value of compounds (gL<sup>-1</sup>) at time  $t$ . Results obtained were plotted in Fig. 8. These results indicate that the various organic compounds in paper mill effluents had different energy consumption. For lignin and phenol the rate of energy consumption were increased linearly but for COD the energy consumption was approximately constant. Moreover, it was reported that there are well-defined 280 organic and inorganic compounds in wastewater coming from paper mill [9]. Consumption of less energy for COD removal than removal of lignin and phenol can be explained by its concentration in wastewater.

Moreover, when 1 m<sup>3</sup> of wastewater is considered under the same conditions, 12kWh electric energy consumption was observed (Table 3).

### Conclusions

A new electrochemical reactor, a three-phase three-dimensional electrode cell consisting of graphite electrode and powder-activated carbon (as working electrode) was used to remove COD, lignin and phenol from paper mill effluent. It was confirmed that removal efficiency depended on the applied cell voltage, initial pH, AC and NaCl amount and airflow. With 25.0V cell voltage, 5.0 min, in presence of air 2.0 minL<sup>-1</sup>, NaCl 5.0gL<sup>-1</sup>, at low pH and AC 20gL<sup>-1</sup>, optimum removal of these pollutants was realized in these conditions, the removal of COD, lignin and phenol from the paper mill effluent was higher than 90%. It is expected that the electrochemical technology based on the three-phase three-dimensional electrode can be applied to treat paper mill effluent.

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