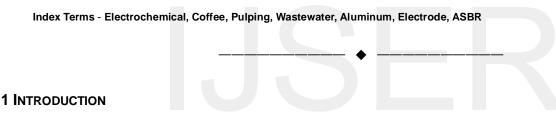
# Coffee Pulping Wastewater Treatment by Electrochemical Treatment followed Anaerobic Sequencing Batch Reactor

Asha .G and B. Manoj Kumar

Abstract- The coffee pulping wastewater used for this study is characterized by low pH, high concentration of COD, ammonia nitrogen, nitrate nitrogen and phosphorous. With aluminum plates as anode and cathode electrodes and at a current density of 93.87 A  $m^2$  the electrochemical reactor performance was found to be good. The experimental results show that the COD, ammonia nitrogen, nitrate nitrogen and phosphorus removal was found to be 95%, 90%, 89% and 94% respectively. The BOD<sub>5</sub> to COD ratio increased from 0.012 to 1.49 in 105 minutes of electrolysis duration, indicating improvement of wastewater biodegradability. The minimum energy consumption was found to be 1.102 kWh kg<sup>-1</sup> COD removal. This study showed that EC reactor performance was good in terms color, nitrogen and phosphorus removal but still remains a high concentration of COD. Since there was an improvement of BOD<sub>5</sub> to COD ratio, an attempt is made to remove the remaining COD in ASBR. The COD, ammonia nitrogen, nitrate nitrogen and phosphorus removal efficiency in ASBR was found to 69%, 99%, 99% and 99% respectively. The combined electrochemical treatment of coffee pulping wastewater followed by ASBR removes 96% of COD and ammonia nitrogen, nitrate nitrogen, phosphorus removal was nearly 100%. This study proves that the EC technique followed by biological treatment in ASBR found to provide an efficient and economic alternative for dealing with recalcitrant organics present in coffee pulping wastewater.



ndia ranked sixth in the world in coffee production and grows about 3 lakh tonne annually. Coffee grown in many regions is

processed using the wet method. Coffee pulping involves large volumes of water and wastewater. Water is a resource for coffee pulping units as it is used to separate good coffee berries from defective ones, as a method of transporting the coffee berries to the pulping machinery and in the pulping operation. The wastewater generated from wet coffee processing can be basically divided into two parts. Firstly, the pulping water with high content of quickly fermenting sugars using enzymes from the bacteria on the coffee cherries. Secondly depending on the processing method applied the water from fermentation/washing or the thick effluents from the mechanical mucilage removers. One kg of dried coffee produces 2.5 kg of wet pulp and 12.4 kg of effluent [1]. For every tonne of fresh cherry processed generates 1 to 20 m<sup>3</sup> of wastewater.

At present in India coffee pulping wastewater is let into small lagoons and these lagoons are operated only during the coffee pulping season. These lagoons often fail since

they are operated in winter at low temperatures, and sometimes they get overloaded with accumulation of Volatile Fatty Acids (VFA) and become malodorous, and pose potential hazards to ground and surface water resources [2]. The main disadvantages of these lagoons are that the requirement of more land area and releases significant amount of methane into the atmosphere.

Previous researches have made attempts to treat coffee pulping wastewater using anaerobic technology. Both thermophilic and mesophilic condition of Upflow Anaerobic Sludge Blanket (UASB) reactor have been successfully used in treating coffee pulping wastewater [3]. By cultivating the mushroom from the pre-treated wastewater of coffee pulp, when this pulp is pasteurized then the treated wastewater obtained consists of highest COD removal efficiency of 87% at high organic loading rate (OLR) of 42.87 g COD L<sup>-1</sup> day and high biogas production rate (BPR) of 2.89 L L<sup>-1</sup> day was achieved [4]. In pilot plant based on UASB reactor for treating coffee pulping wastewater. The COD removal achieved was 77.2% at an OLR of 1.89 kg COD m<sup>-3</sup> day and HRT of 22 hours. The major drawback of this was pH control also due to wide variation of the wastewater strength and flow rates prevented the stable reaction operation of the study [5]. A multi-feed biogas reactor for treating coffee pulping wastewater and biomass waste was capable of converting to biogas and compost as end product [2].

The coffee pulping wastewater is characterized by acidic pH, low BOD with high concentration of COD and nutrients. Since  $BOD_5$  to COD ratio of coffee pulping wastewater is very low, treatment by biological methods is very difficult, hence there is a need to find out a techno-economically feasible treatment method to improve  $BOD_5$  to COD ratio. Many researchers have made an attempt to use electrochemical methods for improving  $BOD_5$  to COD ratio and for the treatment of high strength wastewater. The electrochemical treatment is an emerging technology used for the destruction of recalcitrant organics present in different wastewaters. The mechanism and application of electrochemical process for the treatment of different industrial wastewater are reported by several authors [6-8].

The electrochemical treatment using chloride as the supporting electrolyte was reported for the treatment of different types of wastewaters such as textile dye [9], paper mill [10], Tannery [11] and distillery wastewater [12]. An integrated technology for the treatment of the recalcitrant contaminants of olive oil mill wastewater, involved an electrochemical pretreatment step using the electro-fenton reaction followed by an anaerobic bio-treatment [13]. During electrochemical oxidation of post-methanation distillery wastewater to improve the BOD<sub>5</sub> to COD ratio it was found that low pH condition is more favorable for the treatment of distillery wastewater [12]. From the above discussion, it is evident that, research studies on the electrochemical treatment of coffee pulping wastewater are meager.

The present study investigates the applicability of electrochemical treatment to improve BOD<sub>5</sub> to COD ratio followed by ASBR for COD and nutrients removal from coffee pulping wastewater.

## 2 MATERIALS AND METHODOLOGY

## 2.1 Characterization of Wastewater

The coffee pulping wastewater used in this study was collected from existing lagoons of coffee pulping units from the different coffee estates located in Mercara district of Karnataka state, India. The raw wastewater was analyzed for various parameters and the characteristics are shown in the Table 1. It can be seen that the COD concentration is very high with low  $BOD_5$  and pH. The initial  $BOD_5$  to COD ratio was found to be very low in the range of 0.12-1.49, which suggests that there is a presence of recalcitrant nature of organics in the wastewater.

## 2.2 Analytical methods

All the chemicals used for the analysis were of Analytical Reagent (AR) grade. The samples collected were analyzed for each parameter in duplicate. All the analytical procedures followed the standard methods for examination of water and wastewater [15]. The solution pH was measured using pH meter (Elico-LI 127, India). COD was determined by closed reflux system (Hach-389, USA) and titrimetric method. The aluminum concentration in the solution was measured using inductively coupled plasma spectrophotometer (HORIBA-JY 2000, France). Sulphate, ammonia nitrogen, nitrate nitrogen and phosphorus were analyzed as per standard methods (spectrophotometer (Hach, USA).

## 2.3 Experimental Studies

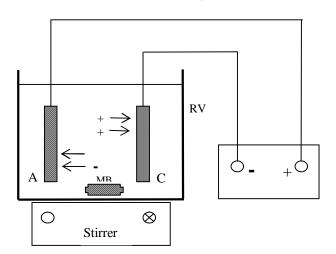
## **Electrolysis experiments**

The laboratory scale experimental setup used for electrochemical studies is schematically shown in Fig.1.The working volume of the reactor is 1 liter. The aluminum plates of size of 7 cm × 7 cm and thickness of 1.5 mm was used as anode and cathode and the electrodes were positioned in vertical and parallel to each other with an inter gap of 1 cm. A direct current power supply unit (Textronix-35D, India) with current range of 0-5 A and the voltage range of 0-16V was used for current supply. The reactor was operated under completely mixed condition facilitated by magnetic stirrer to avoid concentration gradients. The samples were collected at regular time interval from the reactor and analyzed for various parameters.

	Parameter	Range	
	рН	3.92-4.99	
	Color	Black	
	Total Solids	2500-3200	
	Total Dissolved Solids	2300-2500	
	BOD	152-160	
	COD	12000-12500	
	Ammonia nitrogen	30-32	
	Nitrate-Nitrogen	19-26	
	Phosphorous	34-42	
	Chlorides	60-64	
	Aluminum	1.2-1.3	

#### TABLE 1 Characteristic of raw coffee pulping wastewater

Note: All parameters in mg L -1 except pH and color



A-Anode, C-Cathode, MB-Magnetic Bit, RV-Reactor

Fig. 1 Schematic diagram of electrochemical reactor setup

#### **ASBR Experiments**

(1)

(7)

An Anaerobic Sequencing Batch Reactor (ASBR) of working volume 2 L with cow dung as a seed culture was used to treat the Electrochemical (EC) treated coffee pulping effluent. After EC treatment the coffee pulping wastewater was fed into the reactor and kept under anaerobic condition for 23 hours. Every day 50 % of the reactor working volume was decanted followed by addition of the same volume as feed. On alternate days samples were collected for analysis of various parameters such as COD, ammonia nitrogen, nitrate nitrogen and phosphorus.

## **3** MECHANISM OF ELECTROCHEMICAL OXIDATION

The degradation of organics and toxic materials present in the wastewater in an electrochemical process is achieved by direct or indirect oxidation. In the direct oxidation technique, pollutants are destroyed directly at the anode. The indirect oxidizing agents such as chlorine/hypochlorite are generated in situ during electrolysis for oxidation of pollutants. Chlorides present in wastewater acts as supporting electrolyte, and it generates strong oxidizing agents such as chlorine/hypochlorite during the process and the mineralization of organic pollutants. Both hypochlorite and free chlorine can react as oxidizing agents, and they lead to the following oxidation reaction [16]. The reactions involved are

Organic matter + OCI<sup>-</sup> 
$$\rightarrow$$
 CO<sub>2</sub> + H<sub>2</sub>O + CI<sup>-</sup> + product

The COD removal occurs only in the presence of chlorides in the bulk solution because of the reaction between the generated chlorine/hypochlorite and the organic molecules. The indirect electrochemical treatment involves the application of an electric current to the wastewater containing chloride to convert the chloride to chlorine/hypochlorite, and then it oxidizes the pollutants. The hypochlorous acid and hypochlorite ion can decompose organic matter because of their high oxidative potentials [17].

At anode:	$2CI^- \rightarrow CI_2 + 2e^-$	(2)
At cathode:	$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$	(3)
Bulk solution:	$CI_2 + H_2O \rightarrow HOCI + H^+ + CI^-$	(4)
	HOCI→H++OCI <sup>_</sup> .	(5)

In addition, electroflotation also occurs during electrochemical treatment of wastewater. Electroflotation (EF) is a simple process that floats pollutants to the surface of a water body by tiny bubbles of hydrogen and oxygen gases generated from water electrolysis. Therefore, the electrochemical reactions at the cathode and anode are hydrogen evolution and oxygen evolution reactions, respectively. The most widely used electrode materials in electrochemical process are aluminum and, iron and sometimes stainless steel. The electrical current causes the dissolution of metal into wastewater. The metal ions, at an appropriate pH value, can form wide ranges of coagulated species and metal hydroxides, or precipitate and adsorb dissolved contaminants [18]. In case of aluminum as electrode, the reactions are At anode:  $AI \rightarrow AI^{3+} + 3e^{-}$  (6)

At cathode:  $3H_2O + 3e \rightarrow 3/2H_2 + 3OH^-$ 

Al<sup>3+</sup> and OH<sup>-</sup> ions generated by electrode reactions (6) and (7) react to from various monomeric species such as Al(OH)<sup>2+</sup>, Al(OH)<sub>2</sub><sup>+</sup>, Al<sub>2</sub>(OH)<sub>2</sub><sup>4+</sup> and Al(OH)<sub>4</sub><sup>-</sup>, and polymeric species such as Al<sub>6</sub>(OH)<sub>15</sub><sup>3+</sup>, Al<sub>7</sub>(OH)<sub>17</sub><sup>4+</sup>, Al<sub>8</sub>(OH) 2<sup>4+</sup>, Al<sub>13</sub>O<sub>4</sub>(OH)<sub>24</sub><sup>7+</sup>, Al<sub>13</sub>O<sub>4</sub>(OH)<sub>3</sub>. During the electrocoagulation process, metal hydroxide formation occurs, and the flocs have the larger surface area, which is beneficial for a rapid adsorption of soluble organic compounds and trapping of colloidal particles. Finally these flocs are removed easily from aqueous medium by sedimentation or flotation [19, 14].

## **4** RESULTS AND DISCUSSIONS

## 4.1 Electrolysis Studies

In initial studies iron and aluminum electrodes were tried individually for electrochemical treatment of coffee pulping

wastewater. Even though good COD removal with reference to iron electrode was observed but due to turning of treated effluent into reddish color and causing interference for analysis [14] therefore only aluminum was selected for further studies. It is also reported that aluminum electrodes are more preferable under acidic conditions while iron electrode under alkaline conditions. With aluminum electrodes at anode and cathode electrochemical experiments was conducted at the existing wastewater pH of 4.5 and at a current density of 93.87 Am<sup>-2</sup>. The electrochemical reactor was operated for 105 minutes of electrolysis duration. From the Fig. 2 (a) it is seen that COD reduction was rapid during first 45 minutes and then it was gradual, the COD reduced from initial concentration of 12,240 mg L<sup>-1</sup> to 1,832 mg L<sup>-1</sup>. The maximum COD removal efficiency of 88 % was achieved at 75 minutes of electrolysis time. The COD remaining in the EC treated effluent was 560 mg L-1. The decrease in COD removal at later stage might be due to the exhaustion of hypochlorite (HCIO3-) and free chlorine generation in-situ in the reactor (indirect oxidation). Also, due to deposition of toxic metals on the anode (direct oxidation) might have further prevented COD removal. The concentration of supporting electrolyte (chloride) plays an important role in the electrooxidation process. This increases the cell conductivity and OCI<sup>-</sup> radicals, which in turn enhances the destruction of organics present in wastewater. It can be observed that the as COD decreased BOD increased from the initial value of 152 to 834 mg L-1 in 105 minutes of electrolysis duration. The increase in BOD concentration is attributed to the fact that some of the organics have broken down into smaller fragments, which are more biodegradable than parent compounds [20]. In Fig.2 (b) it can be observed that BOD<sub>5</sub> to COD ratio increased from 0.012 to 1.49 at 105 minutes, indicating improvement in biodegradability of wastewater.

From Fig.3 it is observed that there was a rapid reduction in concentrations of ammonia nitrogen, nitrate nitrogen, and phosphorus in the first 30 minutes. The ammonia nitrogen concentration reduced from 32.1 to 5.04 mg L<sup>-1</sup>, nitrate nitrogen reduced from 24 to 4.6 mg L<sup>-1</sup> while phosphorus concentration reduced from 39.6 to 12.8 mg L<sup>-1</sup>, in first 30 minutes. After 60 minutes of electrolysis time, the percent removal of ammonia nitrogen, nitrate nitrogen and phosphorus removal was 90%, 87% and was 85% respectively. When the reaction time was increased to 105 minutes the concentration of ammonia nitrogen, nitrate nitrogen and phosphorus reduced to 3.21 mg L<sup>-1</sup>, 2.5 mg L<sup>-1</sup> and 2.3 mg L<sup>-1</sup> respectively.

After several experimental runs electrochemical treated wastewater contained COD in the range of 450 mg L<sup>-1</sup> to 603 mg L<sup>-1</sup>, ammonia nitrogen values in the range 1 mg L<sup>-1</sup> to 1.8 mg L<sup>-1</sup>, the nitrate nitrogen concentration was in the range of 2.7 to 3.6 mg L<sup>-1</sup> and phosphorus concentration was in the range of 2.2 mg L<sup>-1</sup> to 2.7 mg L<sup>-1</sup>. The electrolysis experiment was conducted for total duration of 1.75 h, and by the end of 1.25 h of electrolysis duration, the pollutant removal was maximum therefore the reactor was stopped at this stage.

In this study electro flotation of the pollutants was observed with scum floating on the surface of wastewater by tiny bubbles of hydrogen and oxygen gases generated from water electrolysis. Probably, the electrochemical reactions at the cathode and anode are hydrogen evolution and oxygen evolution reactions respectively. Initially more foam was formed at high current density due more rapid gas evolution. Furthermore the foam was found to be suppressed substantially towards the end of the treatment time.

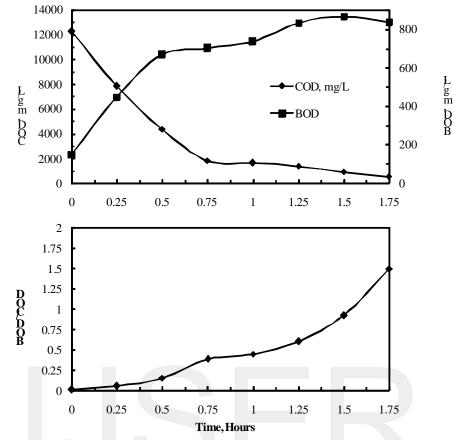


Fig. 2 Variation of COD and BOD<sub>5</sub> removal and BOD<sub>5</sub>/COD ratio as a function of electrolysis time

#### 4.2 Energy Consumption

Considering the initial and final concentrations of compounds in wastewater at different times, electric energy (EE) consumed in kilo Watt hour per kg removal of each compounds (COD, ammonia nitrogen, nitrate nitrogen and phosphorous) was calculated with the following equation

# V×1×t×1000

Electrical energy consumption = 60(co-Ct)Sv (8) Where V is Voltage in Volts; I is current in Amperes; t is electrolysis time (min), S<sub>v</sub> is sample volume in liter, C<sub>o</sub> initial concentration (gL<sup>-1</sup>), C<sub>t</sub> are the concentration value of compounds (mgL<sup>-1</sup>) at time t [21]. Results obtained were plotted in Fig. 4. These results indicate that the various organic compounds in coffee pulping wastewater have different energy consumption. For COD the rate of energy consumption is more followed by nitrate nitrogen but for ammonia nitrogen and phosphorus the rate of energy consumption was less. The minimum energy consumption was found to be 1.102 kWh kg<sup>-1</sup> COD removal.

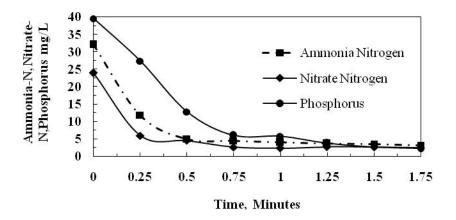


Fig. 3 Variation of Ammonia nitrate, Nitrate nitrogen and phosphorus as a function of electrolysis duration

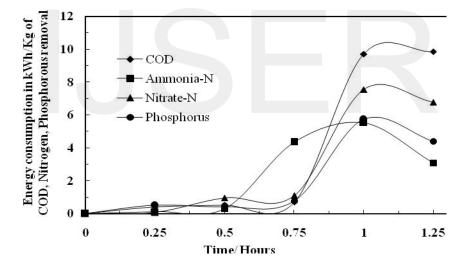


Fig. 4 Variation of energy consumption for COD, Ammonia nitrogen, Nitrate nitrogen and Phosphorus

#### 4.3 Determination of Instantaneous Current Efficiency (ICE)

Instantaneous current efficiency is the current efficiency measured at constant time interval during the electrochemical treatment of wastewater. Measurement of ICE gives information about the formation of polymeric product at the anode during treatment. Melanoidin, a polymeric material present in the coffee pulping wastewater may be adsorbed on the electrode surface and may decrease the efficiency of the electrode. ICE is defined as the ratio of the current stoichiometrically required for the oxidation of organics to that of total consumption. It was calculated using the relation [22-24].

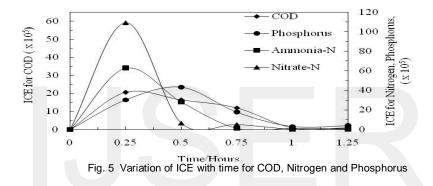
$$ICE (\%) = \left\{ \frac{(COD)_t - (COD)_{t+\Delta t}}{8 \times I \times \Delta t} \right\} F \times V \times 100$$
(9)

Where COD<sub>t</sub> and COD<sub>(t+ $\Delta t$ )</sub> are the COD values (in gram O<sub>2</sub> per liter) at time t and t+ $\Delta t$  (in seconds) respectively, I is the current in amperes, F is the Faraday's constant (96487 C mol<sup>-1</sup>), V is the volume of electrolyte (liter), t is the time (in seconds) and the value

IJSER © 2015 http://www.ijser.org 8 is a dimensional factor for oxygen equivalent mass (g eq<sup>-1</sup>).

The instantaneous current efficiency was determined for the track study results. When the electrochemical treatment was done at 16 V current. Fig. 5 shows the calculated ICE values with respect to time. ICE values increased sharply during first 0.25 h and drop rapidly during further course of electrolysis. Similar trend has been observed by many previous researchers [23,25], during electrochemical treatment of different high strength wastewater. The probable reason for increase in ICE in the initial hours may be due to initial polarization of the electrode and formation of fresh active electrode surface and later decrease of ICE may be due to the growth of an adherent passivating film on anode surface that might have poisoned the electrode or by production of stable intermediates that cannot be further oxidized by direct electrolysis. Another possible reason is that the decrease of ICE can be attributed to the adsorption of polymeric material present in the wastewater, on the electrode surface or due to the passivation of electrode surface by reaction between the metallic chloride and calcium or magnesium salts present in wastewater [26].

From the results of electrochemical experiments the concentration of parameters such as COD, ammonium nitrogen, nitrate nitrogen and phosphorus have been reduced to greater extent. EC treated effluent contained COD in the range of 450 mg L<sup>-1</sup> to 603 mg L<sup>-1</sup>, ammonia nitrogen values in the range 1 mg L<sup>-1</sup> to 1.8 mg L<sup>-1</sup>, the nitrate nitrogen concentration was in the range of 2.7 to 3.6 mg L<sup>-1</sup> and phosphorus concentration was in the range of 2.2 mg L<sup>-1</sup> to 2.7 mg L<sup>-1</sup>. Probably with increase in the electrolysis duration the COD and nitrate nitrogen concentration would have reduced further but to reduce the power requirements the effluent from electrochemical reactor was further fed to SBR and anaerobic conditions were maintained since only COD, nitrate nitrogen and phosphorus was remaining in the electrochemical treated wastewater.



#### 4.2 Anaerobic Sequencing Batch Reactor Studies

The electrochemical treated wastewater was further treated in the ASBR. The remaining COD, ammonia nitrogen, nitrate nitrogen and phosphorus was removed in the anaerobic SBR where in no power requirement will be there. From Fig. 6 it is observed that on the day 1 in end anaerobic COD concentration was 580 mg L-1 was reduced to 480 mg L-<sup>1</sup> on the day 5. On day 7 it was less than 250 mg L-1. Fig. 7 and Fig. 8 shows the ammonia nitrogen, nitrate nitrogen and phosphorus concentration reduction with days in ASBR respectively. When the electrochemical treated wastewater was subjected to biodegradation in ASBR on the day 1 nitrate-nitrogen concentration reduced 1 mg L-<sup>1</sup> to 0.14 mg L-<sup>1</sup>, phosphorus to 1.3 mg L-1 and from day 2 it was BDL. But in case of ammonia nitrogen the concentration was reduced gradually on the day 1 it was reduced to 3 mg L-<sup>1</sup> and day 4 and 5 it was 2.3 mg L-<sup>1</sup> and 1.2 mg L-<sup>1</sup> respectively, but on the day 6 onwards it was BDL. The performance of electrochemical treatment followed by ASBR is shown in Table 2

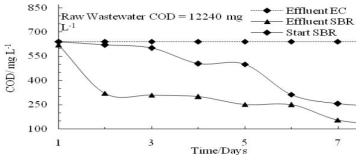


Fig. 6 Variation of COD in ASBR

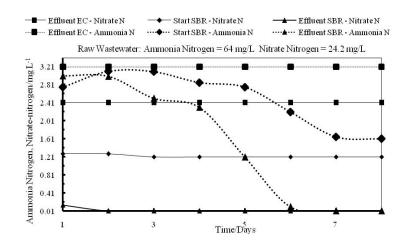


Fig. 7 Variation of Ammonia nitrogen, Nitrate nitrogen in ASBR

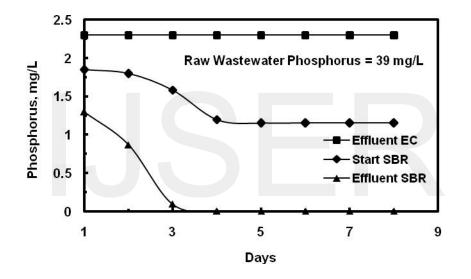


Fig. 8 Variation of Phosphorus in ASBR

## **5** CONCLUSION

On the basis of the results of this study, the EC treatment followed by ASBR method can be effectively used for the treatment of coffee pulping wastewater. The electrolytic efficiency was primarily based on the efficiency of COD removal and improvement in BOD<sub>5</sub> to COD ratio. The maximum COD removal was 96% at current density of 93.87 A m<sup>-2</sup> at pH 4.5 with 105 min of electrolysis duration. The BOD<sub>5</sub> to COD ratio has been increased from 0.012 to 1.49 indicating in wastewater biodegradability. After EC treatment followed by ASBR the COD concentration was found to be in the dischargeable limit of <100 mg L<sup>-1</sup>, while the concentration of ammonia nitrogen, nitrate nitrogen and phosphorus are BDL. So, treated coffee pulping effluent can be discharge into nearby water sources or can be used for plantation for watering purpose.

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