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Removal of Fatty Acids from Palm Oil Effluent by Combined Electro-Fenton and Biological Oxidation Process

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Abstract The main objective of this study was to find out a cost-effective treatment methodology for the treatment of palm oil effluent (POE) obtained from a food processing industry. An electro-Fenton pretreatment and biological oxidation has been suggested for the removal of recalcitrant contaminants present in POE. An initial COD of about 6,700 mg/L of POE was subjected to electrolytic degradation for 2 h and subsequently by biological oxidation. The biological oxidation was carried out using *Aspergillus niger* and *Pseudomonas putida* in anaerobic condition. Electro-Fenton process removed 48.35% of the COD. Biological oxidation subsequently decreased the COD to 86.12% and BOD to 85.23%. In the combined process, a high reduction in TOC and TN were achieved. Experimental conditions have been optimized and performances of these techniques have been discussed. The treated water can be reused for general and agricultural purposes.

Keywords Palm oil effluent · Electro-Fenton · Biological oxidation · COD · BOD · pH

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1 Introduction

Palm oil is used across various food industry segments, especially in the confectionary and sweets industry and also for cooking. At present, the effective duty on crude palm oil is 46.35%. Export countries from where India receives palm oil are mainly Malaysia and Indonesia. Solvent Extractors' Association of India (SEA) has reported that the import of palm oil during November 2007 to January 2008 was 990,453 tons, compared to 737,528 tons for the same period of last year, an increase of 13%. In total, India imports every year roughly 3–3.5 million tons of palm oil and its variants (Food Industry India).

Over the recent years there has been an increasing concern for environmental risk of industrial activities associated with oil extraction, hydrocarbon processing, food processing, oil refining, and transportation of oil through pipelines. These industries have increased the threat of oil pollution to the environment and subsequently concomitant discharged into the natural environment creates major ecological problem throughout the world (Agustin et al. 2008).

Residue oil is one of the key ingredients of palm oil effluent (POE) which influences the high chemical oxygen demand (COD) and biochemical oxygen demand (BOD) values. POE is rich in organic carbon with a BOD higher than 20 g/L and nitrogen content around 0.2 g/L as ammonia nitrogen and 0.5 g/L total nitrogen (Vijayaraghavan et al. 2007; Vijayaraghavan and Ahmad 2006; Vijayaraghavan et al. 2006). POE contains significant amounts of oil (triacylglycerols) and degradative products such as di- and monoacylglycerols and fatty acids which all contribute to the high COD and BOD of the effluent (Valdez-Vazquez et al. 2005). Crude palm oil contains triglycerides as the major constituent. The minor constituent, approximately 1%, includes carotenoids, tocopherols, sterols, triterpene alcohols, phospholipids, glycolipids, terpenic and aliphatic hydrocarbons, and other trace impurities (Umerie et al. 2004; Goh et al. 1985). POE is a colloidal dispersion of biological origin and with an unpleasant odor. It has total solids content of 5–7% which a little over half is dissolved solids, and the other half being a mixture of various forms of organic and inorganic suspended solids. These characteristics make it not only highly polluting but also extremely difficult to treat by conventional methods (Zinatizadeh et al. 2006; Bhatia et al. 2007).

Electro-Fenton process was used already to treat olive oil effluent which is characterized by high concentration of several organic compounds including sugar, tannin, pectin, lipids, and phenolic substances, which are responsible for their high COD and BOD. Their concentration generally varies between 80 and 200 g/L for COD and 12–63 g/L for BOD (Kallel et al. 2009). Alcohol distillery wastewater has also been treated effectively by electro-Fenton (Yavuz 2007); the reactions involved in electro-Fenton are explained as follows:

In EF process; Fe^{2+} and H_2O_2 can be generated on-site electrochemically, either separately or concurrently. H_2O_2 can be electrogenerated by the reduction of efficiency depends on the catalytic activity of the anodes used, dissolved oxygen, and Fe^{2+} by the reduction of Fe^{3+} or the oxidation of a sacrificial Fe anode as given in reactions (1)–(3) (De Francesco and Costamagna 2004):

$$O_2 + 2e^- + 2H^+ \rightarrow H_2O_2 \quad (1)$$

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

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + OH^\bullet + H_2O \quad (3)$$

The reaction between H_2O_2 and Fe^{2+} produces hydroxyl radical, a non-selective strong oxidant, according to the reaction (4):

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^\bullet + OH^- \quad (4)$$

The removal efficiencies of EF process depended heavily on factors such as the quantities of H_2O_2 and Fe^{2+} cation, pH of the solution, current density, and supporting electrolyte concentration if the electrical conductivity of the working solution is low. Detailed explanation about the Fenton reactions has been given by Szpyrkowicz et al. 2001.

Fenton oxidation pathway may consist of an attack by the hydroxyl radicals on the alkyl chain of fatty acids. The OH radical has strong ability to rupture the aromatic ring attached with hydroxyl groups present in the fatty acids. This leads to the formation of water-soluble compounds via abstraction of hydrogen and insertion of oxygen atoms and with the participation of ferrous and/or ferric ions. The process yields lower aliphatic compounds, derived from the breaking of longer hydrocarbon chains (C–C) of fatty acids and finally leads to the mineralization of the initial organics (Fig. 1).

The palm oil mills generate many by-products and wastes beside the liquid wastes. Since the ultimate goal of wastewater management is towards zero discharge, the best wastewater treatment scheme is

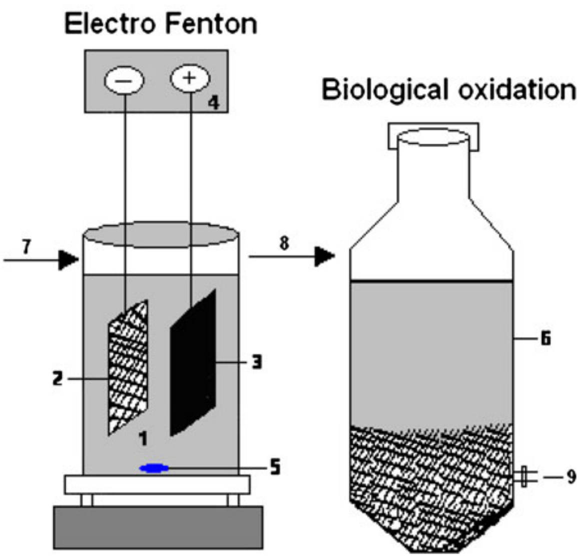

Fig. 1 Schematic representation of palm oil effluent treatment process. (*1*) POE, (*2*) anode, (*3*) cathode, (*4*) DC power supply, (*5*) magnetic stirrer, (*6*) bio setup, (*7*) crude POE, (*8*) pretreated POE, (*9*) outlet of treated water

inevitably a treatment that allows 100% reuse and recycling of the water (Najafpour et al. 2005).

Ahmad et al. (2005) reported the removal of residual oil from palm oil mill effluent using synthetic rubber powder as the adsorbent. POE has generally been treated by anaerobic digestion resulting in methane as a value added product. Aerobic digestion of POE decreases carbon content and inorganic nitrogen and changes pH from the acidic range to an alkaline one. Such treatment also increases the ratio of organic nitrogen leading to the production of a better fertilizer (Oswal et al. 2002).

Hence, POE treatment requires a sound and efficient system in facing the current challenges such as low cost treatment, high BOD and COD reduction efficiency, zero discharge etc. The current treatment technology of POE typically consists of biological aerobic and anaerobic digestion or facultative digestion. The fatty acids present in the POE are inhibitory to many microorganisms making it difficult to treat biologically prior to discharge in waterways at initial stage (Ahmad et al. 2003).

In such cases combinations with chemical oxidation processes may improve the overall efficiency and efficacy. Most suitable oxidation processes for combination with biological treatment are wet air oxidation, ozonation, hydrogen peroxide treatment, and other advanced oxidation processes. Most effective are OH-radicals produced in all these oxidation processes. Chemical oxidation produces intermediates with usually improved biodegradability. Process combinations may be serial or with recycling between chemical oxidation and biological treatment (Jeworski and Heinzle 2000).

In the bid to achieve a zero discharge of the palm oil effluent, an electro-Fenton pretreatment step followed by biological oxidation has been investigated and suggested as one of the economical treatment methods for the recalcitrant contaminants present in POE obtained from a food processing industry. The treated water has been recommended to reuse for general and agricultural purposes.

2 Materials and Methods

2.1 Palm Oil Wastewater

POE was collected from a food processing industry. The effluent pH, total nitrogen (TN), total suspended solids, total dissolved solids (TDS), BOD, and temperature were measured. COD was measured with Spectroquant TR 420, Merck by close reflex method. The characteristics are presented in Table 1.

2.2 Electro-Fenton Treatment

Fenton reagent is one of the strong oxidants, which can oxidize both biodegradable and non-biodegradable organics. Experiments were carried out in a 600-ml glass reactor for the electro-Fenton treatment. The solution of reactants was stirred continuously with the aid of magnetic stirrer to avoid concentration gradients. The electro-Fenton reactor was formed by one pair of anode (Ti/RuO_2) and cathode (stainless steel) which were separated approximately 1 cm distance apart from each other and immersed into the effluent. The immersed area of anode was 23.7 cm^2. Experiments were carried out at 0.05A/cm^2. The current input was supplied by a dc power supply (APlab L-3210). H_2O_2 was added to the solution to enhance the reactivity and also 3 mM of $Fe_2SO_4.7H_2O$ was added initially. The overall duration of the process was 2 h. At the end of the process, COD was measured. After electro-Fenton, the pretreated POE was further degraded by biological oxidation under different conditions.

2.3 Biological Oxidation

The pure culture of microorganism was grown in 250-ml conical flask, containing 100 ml nutrient broth [composition (g/L): peptone—10, NaCl—5, yeast extract—2, and beef extract—1] at 30±2°C for 24 h under static condition. Sabouraud dextrose agar [composition (g/L): peptone—10, D(+)glucose—20, and agar-agar—17]

Table 1 Characteristic of the palm oil effluent

Parameter	Before treatment	After treatment (Electro-Fenton)	After treatment (biological oxidation) at pH 6	
			A. niger	*P. putida*
Color	Brown	Yellow	Colorless	Colorless
pH	5.3	6.7	7.62	7.53
COD (mg/L)	6,712	3,464	1,182	1,140
BOD_5 (mg/L)	4,239	2,742	858	830
TDS (mg/L)	231	206	144	142
TN (mg/L)	64	41	18	16
TOC (mg/L)	712	423	134	125

was also used. Based on the biochemical tests, *Aspergillus niger* and *Pseudomonas putida* were identified as effective organisms for degradation of palm oil effluent and used in this study. All these cultures utilize the carbon as a sole source of energy. They were inoculated into the pretreated POE to study the degradation behavior by measuring final COD and BOD. Tests were conducted at different pH (4, 6, and 8) and it was adjusted by H_2SO_4 and NaOH.

2.4 Analytical Methods

Total organic carbon (TOC) and TN was measured using a flow injection analyzer (SKALAR-TOC/TN analyzer). A typical analysis for TOC measures both the total carbon present as well as the inorganic carbon (IC). Subtracting the inorganic carbon from the total carbon yields TOC. TOC is measured directly in the sample by acidifying the sample to a pH value of 2 to release the IC gas. The remaining non-purgeable CO_2 gas contained in the liquid aliquot is then oxidized releasing the gases. These gases are then sent to the detector for measurement. In our study, 5 mL POE before and after treatment was taken in test tubes supplied by SKALAR. From which 20 μL was injected into the system for analysis.

The biodegradability of the wastewater was measured in terms of BOD_5. COD was determined by closed reflux method (APHA 1992). Total dissolved solids were determined by drying the sample at above 100°C. The pH of the effluent was measured by using a pH meter (LT Lutron pH-201, India). From the results, % reduction of COD and BOD was calculated. Spectral analysis of the samples was carried out using Fourier transform infrared (FTIR; Nexus 670 model) and hydrogen-1 nuclear magnetic resonance (^{1}H-NMR; Brucker 400 MHz NMR spectrometer).

3 Results and Discussion

3.1 Pretreatment by Electro-Fenton Process

Initially POE was treated with electro-Fenton to study its effect on the changes in effluent characteristics. At the end of electro-Fenton (2 h), removal of COD was 46%. During electro-Fenton treatment, pH increased from 5.3 to 7.4, the pH value of electro-Fenton-treated POE can be considered favorable for biological oxidation to achieve highest reduction of COD, BOD, TOC, and TN. Many authors have reported that the biodegradability index (BOD_5/COD) should be in between 0.4 and 0.8 (Aboulhassan et al. 2008) for any wastewater to be treated by biological process. The BOD_5/COD for POE was 0.63 initially and hence it was planned to treat by electro-Fenton as pretreatment for POE. At the end (2 h) of electro-Fenton, the BOD_5/COD was enhanced to 0.8. This result shows that the electro-Fenton process eliminates the oxidizing organics present in POE such as long chain fatty acids. Mae et al. (2000) reported that the Fenton treatment of palm oil shell wastes converts high molecular weight fatty acids into smaller weight fatty acids such as acetic acid, formic acid.

3.2 Biological Oxidation

The biodegradation rate of strains of *A. niger* and *P. putida* in degrading pretreated palm oil wastewater was tested for 5 days. However, *P. putida* successfully reduced the COD and BOD about 86.12% and 85.23%, respectively, while *A. niger* correspondingly removed COD and BOD about 73% and 67%, respectively, under the same operating conditions. From the results, the two organisms were found to degrade the fatty acid content present in POE. It was noted that microorganisms utilize the organic substances present in POE as nutrients and finally breakdown the organic matter into simple end-product gases such as methane, carbon dioxide, and hydrogen sulfide, and water (Sumathi 2004; Ballesteros Martín et al. 2008; Kamath and Vaidyanathan 1990). Figure 2 shows the

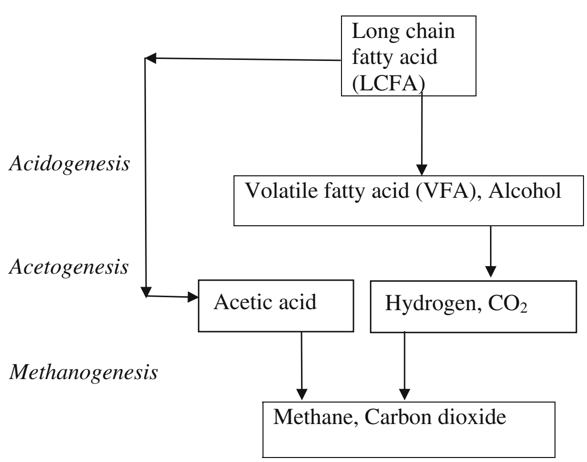

Fig. 2 Simple representation of the anaerobic digestion process of fatty acid

simple representation of anaerobic digestion of long-chain fatty acids.

In order to find the effect of pH on the degradation of palm oil wastewater, anaerobic digestion was carried out varying initial pH of the pretreated effluent as 4, 6, and 8. At pH 8, the COD removal percent was found to be lowest and highest at pH 6. Figures 3 and 4 illustrate the % reduction of COD and BOD at different pH.

Table 1 shows the characteristics of POE before and after electro-Fenton and biodegradation processes.

3.3 FT-IR Spectral Studies

Figure 5 shows IR spectra of raw POE (A), treated effluent by electro-Fenton (B), and treated effluent by biological oxidation (C). The major peaks were presented in Table 2 (George 2001). It can be seen that some structural changes might have occurred during the combined electrochemical–biological process. The peaks at 3,390 cm^{-1} and 1,745 cm^{-1} indicates the presence of functional groups –OH and C=O, respectively. In the spectrum B and C, intensity of the characteristic peaks was reduced and some new peaks were appeared. This may due to the destruction of organics in the POE and converted in to some secondary products. At the end of the process, the appearance of a new peak at 2,366 cm^{-1} corresponds to the characteristic peak of CO_2. While adding microorganism a new peak mostly occurred at 1,640 cm^{-1} which can be identified as C=C of the simple aliphatic chain of the low molecular weight fatty acid. This indicates that the organics present in the POW may have converted into acids like formic, oxalic, and acetic acids. Further these acids may get converted to CO_2.

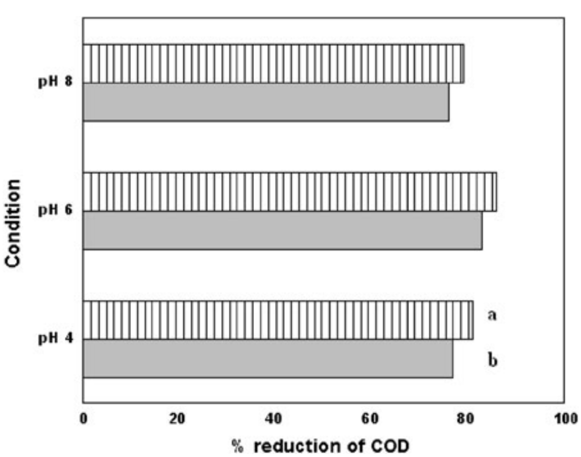

Fig. 3 Comparison of the % reduction of COD at different pH. (*a*) *P. putida*, (b) *A. niger*

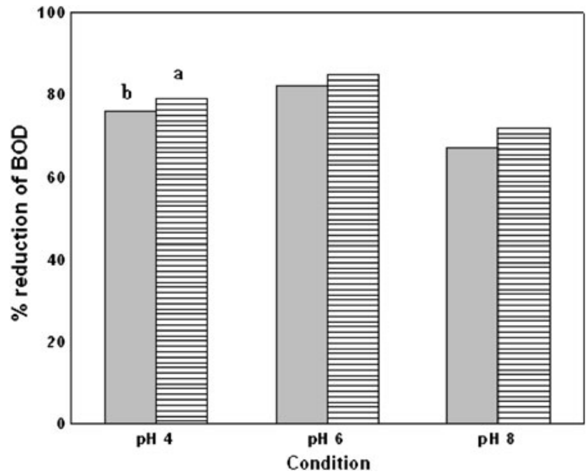

Fig. 4 Comparison of the % reduction of BOD at different pH. (*a*) *P. putida*, (*b*) *A. niger*

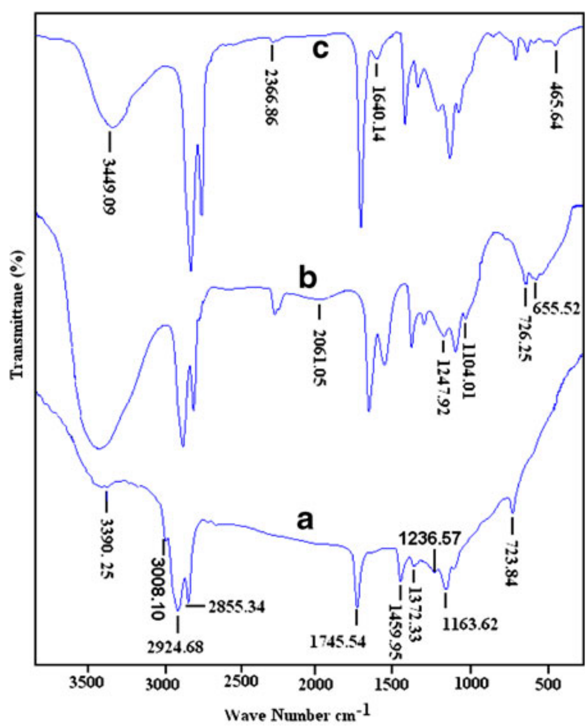

Fig. 5 The figure shows IR spectra of raw POE (*A*), treated effluent by electro-Fenton (*B*), treated effluent by biological oxidation (*C*)

Table 2 FT-IR peak assignments

S. no.	Peak (cm^{-1})	Assignment
1.	3,390	–OH
2.	3,008	C=C
3.	2,924	Unsaturated C–H
4	2,855	Saturated C–H
5	2,366	Characteristic of CO_2
6	1,745	C=O
7	1,640	C=C of simple fatty acid
8	1,459	$–CH_2$ bending
9	1,372	Methyl group
10	1,236	C–O stretching
11	723	C–H out of plane

3.4 ^{1}H-NMR Studies

Application of ^{1}H-NMR had been reported for the fatty acid present in the POE. From Fig. 6, ^{1}H-NMR of raw POE shows major peaks at one to three chemical shifts. It indicates the presence of aliphatic protons. Peak at 0.91 corresponds to the terminal $–CH_3$ group of fatty acid present in palm oil. The remaining peak in the region of 1.6 may due to backbone of $–(CH_2)_n–$ group present in the fatty acid. Similar trend were observed in spectrums B and C. It can be explained that the longer aliphatic chains may break down into smaller one. The peak at 5.2–5.4 may be due to the presence of olefinic protons. After the treatment, the intensity of these protons was reduced. It reveals that the some olefinic protons were ruptured. Similar reduction was identified with the peak at 2.34 corresponds to α-methylene group to acid ($–CH_2–COOH$). This may be due to the conversion long-chain acidic groups into smaller length acids like formic, acetic, and oxalic acid. From the NMR it can be assumed that the biological oxidation occurs via the addition of oxygen by the organism which means that large amount of carbon and hydrogen consumed by the organisms for its utilization. Figure 7 summarizes the overall simplified degradation scheme for palm oil wastewater.

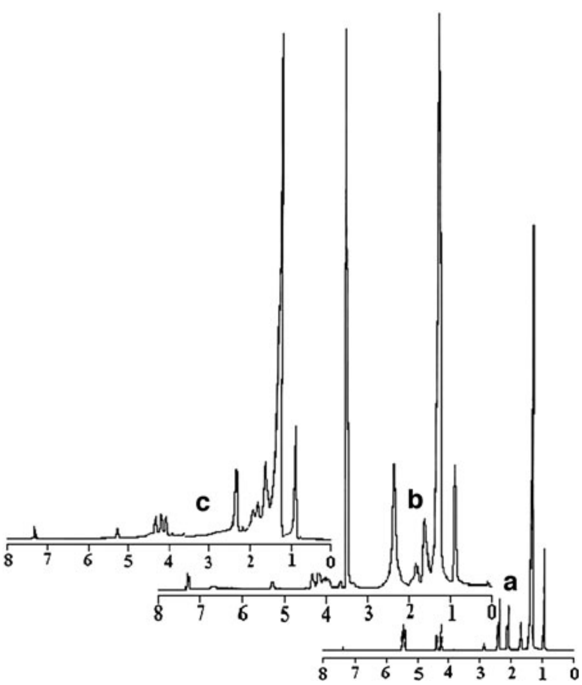

Fig. 6 The figure shows ^{1}H-NMR spectra of raw POE (*A*), treated effluent by electro-Fenton (*B*), treated effluent by biological oxidation (*C*)

3.5 Energy Consumption

The energy consumption for electro-Fenton pretreatment process for degradation of POE was calculated using the following equation (Vlyssides et al. 2002):

$$\text{Energy}\left(\text{KWh}/\text{m}^3\right)(V \times I \times t/\text{vol}) \times \left(\text{COD}_{\text{ini}}\Big/\Delta(\text{COD})_{\text{exp}}\right) \quad (5)$$

Where V is the cell voltage (in V), I the applied current (in A), vol the solution volume (in l), COD is the chemical oxygen demand (in mg/L), and t the electrolysis time (in h). It was estimated that the energy consumed for treating 1 l of POE was 87.44 Wh. The operating cost for the energy used (87.44 Wh) is approximately Indian Rupees 0.70 or 70 paise (1\$US=Indian Rupees 46).

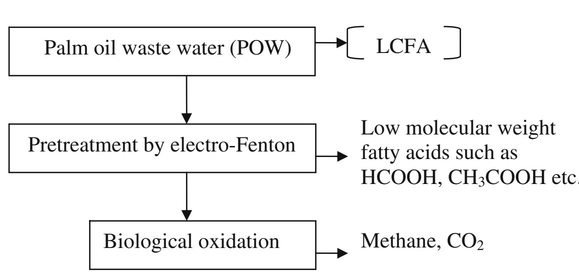

Fig. 7 Simplified representation of the overall POE treatment process

In biological oxidation process, the operating cost is mainly dependent on procuring nutrients for microorganisms. Nutrient broth and Sabouraud dextrose agar were used in this study which is less expensive. The cost for treating 1 l of POE (pretreated by electro-Fenton) including the nutrient medium was determined and it is Indian rupees 0.35.

The total cost for treatment of 1 l of POE is Indian Rupees 1.05. The amount is slightly higher than what we expected. But it can be reduced in due course of study. Hence, it has been suggested that the treatment of POE by combined electrochemical–biological process is an economical method.

4 Conclusions

The combined degradation method of palm oil wastewater is effective and economical. Low molecular weight fatty acids were obtained at the end of electro-Fenton pretreatment and it was further degraded to CO_2 by biological oxidation. Both the combined electro-biological methods remove COD (86%) and BOD (85%) to a larger extent. As pH play a vital role towards the biological oxidation, optimization of pH was determined. At pH 6, maximum removals of COD and BOD were obtained. FT-IR and ^{1}H-NMR studies confirmed the structural changes occurred during the treatments.

Electro-Fenton enables the successful increment of biodegradability index of the wastewater. From the study, it is obvious that the combined process is an eco-friendly method and plays an important role in wastewater management, thereby reducing the environmental risks. The treated water can be reused for general purpose in industrial application.

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