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Research

STUDIES ON COMPARISON OF SLUDGE PRODUCED FROM CONVENTIONAL TREATMENT PROCESS AND ELECTROCHEMICAL PROCESSES OF SOYA OIL REFINERY PROCESSING WASTEWATER

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ABSTRACT

The soybean [*Glycine max* (L.) Merr.] is grown worldwide for its high protein and oil contents. Soya edible oil refinery processing wastewaters are the pollution and harmful to environmental point-of-view. A study was carried out for biological treatment and electrochemical wastewater treatment. The development of innovative combined electro oxidation and coagulation is the crucial factor towards the objective of developing a new sustainable development. A port of the present study, treatment of soya oil refinery wastewater by electro oxidation using graphite and electro coagulation using aluminum and iron electrodes was investigated. The effects of initial pH 3 to 10, sodium chloride 0.1 M (NaCl) as a supporting electrolyte and electrolysis time (180 min) with using consent at current density of 2.27 mA cm^{-2}. Satisfactory results were attained working with 500 ml wastewater and adjusting pH to 3 to 10. In this way, alkalinity, total hardness, TDS, nitrate, phosphate, chloride, potassium sodium and sulphate and compared to biological treatment electrochemical treatment sludge production was 28% decreased. A conventional and electrochemical generated sludge was investigated through FTIR studies functional groups of sludge and microscopic characterized by scanning electron microscope.

INTRODUCTION

Soybean is the dominant oilseed produced in the world due to its favorable agronomic characteristics, high quality protein and valuable edible oil. During the recent decades, demand for vegetable oils has been on the rise due to two main factors; firstly, the increasing demand caused by higher consumption of edible oils due to population growth, improvement in the standards of living together with changing diets and secondly, the development of the biofuels industry which is felt mostly in the European Union, USA, Brazil, Argentina, China and India (Rosillo-Calle, *et al.,* 2009).

Oil produced by mechanical pressing or, more often, by solvent extraction of soybean is termed crude soybean oil. Crude edible oil refining is an essential step for the production of vegetable oils and fats since the process removes undesirable components in order to make the oil fit for consumption. The final stage of edible soybean oil manufacture is the complex refining process, of which the most delicate phase is purification. In a vegetable oil industry, the effluent mainly comes from the degumming, de-acidification, neutralization, bleaching, and deodorization steps, etc. (Kale, *et al.,* 1999; Rajkumar, *et al.,* 2010). Several pre-treatment technologies have been developed and applied to treat oily effluent which divided to physical methods such as mixing, sedimentation, coagulation and flocculation; and also chemical methods like chemical sedimentation, absorption and disinfection. In order to eliminate the pollutants,

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conventional biological treatments of aerobic and anaerobic treatments or facultative digestion are the most commonly used (Rajkumar, *et al.,* 2010). However, these biological treatment methods need proper maintenance and monitoring because these methods depend solely on microorganisms to degrade the pollutants. The microorganisms are very sensitive to the changes in their environment and thus great care is needed to ensure that a suitable environment is maintained for the micro-organisms to grow in the process.

In recent years, electrochemical treatment methods such as electro-oxidation (EO) and electrocoagulation (EC) have attracted great attention as an eco-friendly and cost-effective process (Chen, 2004; Holt, *et al.,* 2005). EC involves the *in situ* generation of coagulants by electrolytic oxidation of an appropriate sacrificial anode (e.g., iron and aluminum) upon application of a direct current. The metal ions generated hydrolyze in the electrocoagulator to produce metal hydroxide ions and neutral $M(OH)_3$. The low solubility of the neutral $M(OH)_3$, mainly at pH values in the range of 6.0 to 7.0, promotes the generation of sweep flocs inside the treated waste and the removal of the pollutants by their enmeshment into these flocs. Many researchers have investigated the electrochemical process of various types of effluent such as landfill leachate (Li, *et al.,* 2001), food-processing industrial (Barrera-Diaz, *et al.,* 2006), p-chlorophenol and p-nitrophenol (Borras, *et al.,* 2003), tannery (Murugananthan, *et al.*, 2004), pesticides (Vlyssides, *et al.*, 2004), olive oil mill (Gotsi, *et al.*, 2005; Un, *et al.,* 2006), textile (Muthukumar, *et al.,* 2007; Rajkumar and Muthukumar, 2012), paint (Korbahti, *et al.,* 2007), paper mill (El-Ashtoukhy, *et al.,* 2009), and sugar factory (Guven, *et al.,* 2009).

The objective of this study is focused on the systematic evaluation of sludge produced during conventional treatment process and compared with electrochemical processes from soya oil refinery processing wastewater treatment.

MATERIALS AND METHODS

Chemical reagents

All analytical chemicals were obtained from (Loba chemie, Mumbai, India), analytical grade were used in this study. The graphite materials used were obtained from M/S Carbone Lorraine, Chennai, India. The electrical resistivity of graphite sheets was 0.001 Ωcm. The pH of the aqueous sample was adjusted by adding 0.1 N HCl and 0.1 N NaOH and determined before and after treatment by using a pH meter (Susima pH meter AP-1 Plus, Chennai, India). All the solutions were prepared using deionized water.

Industrial effluent

Soya edible oil refinery effluent used in this study was obtained from M/S Sakthi Sugars Limited-soya division, Pollachi, Tamilnadu, India. The physico-chemical characteristics of soya oil refinery effluent are shown in the Table 1.

Table 1. Physico-chemical characteristics of soya oil refinery effluent

Colour	Yellowish
Temperature (°C)	45 ± 2
pH	11.44 ± 0.43
Electrical conductivity	1.623 ± 0.023
Total dissolved solids (mg/l)	1015 ± 52
Total suspended solids (mg/l)	1655 ± 12
Volatile suspended solids (mg/l)	116 ± 5
Alkalinity (mg/l)	36 ± 2
Chemical oxygen demand (mg/l)	36000 ± 2500
Oil and grease (mg/l)	6750 ± 275
Nitrate (mg/l)	39.87 ± 2
Phosphate (mg/l)	38 ± 3
Chloride (mg/l)	109 ± 10
Potassium (mg/l)	35.2 ± 2
Calcium (mg/l)	68 ± 3

Experimental set-up

The reactive graphite electrodes in the EO process and aluminium, iron electrodes in the EC process were connected to an external DC power source (the schematic diagram can be seen in Fig. 1). In the electro oxidation and coagulation study, graphite, aluminium and iron reactive electrodes with dimensions of 1 mm × 25 mm × 115 mm and 3 mm × 20 mm × 50 mm was used respectively (active electrode surface dipped in wastewater). The total effective electrode area was 57.5 and 20 cm^2 for EO and EC process respectively, and the spacing between electrodes was 15 mm for both process electrodes.

Experimental procedure

An electrochemical study were performed under batch conditions at room temperature in a plastic container of 400 ml filled with 250 ml of soya oil refinery effluent in the both process with 0.1 M concentration of NaCl. The pH of sample was adjusted using diluted HCl (1 N) and NaOH (1 N) and measured using pH meter (Susima, Chennai, India). The anode and cathode were connected to a DC power supply (SATO, 500 mA output) to control the current density applied (current densities from 2.27 mA cm^{-2} were applied) during the EC operation period (from 0 to 180 min). During EC operation the

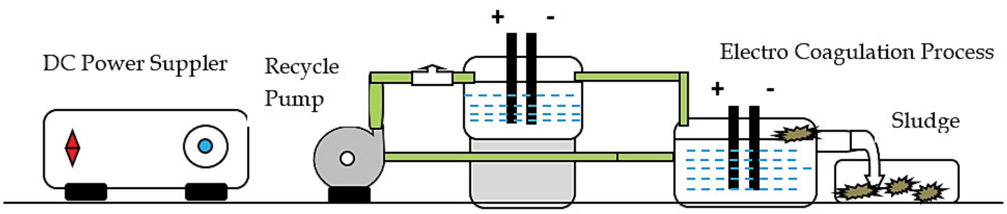


Fig. 1 Schematic diagram of the electro chemical procedure in soya edible oil wastewater treatment.

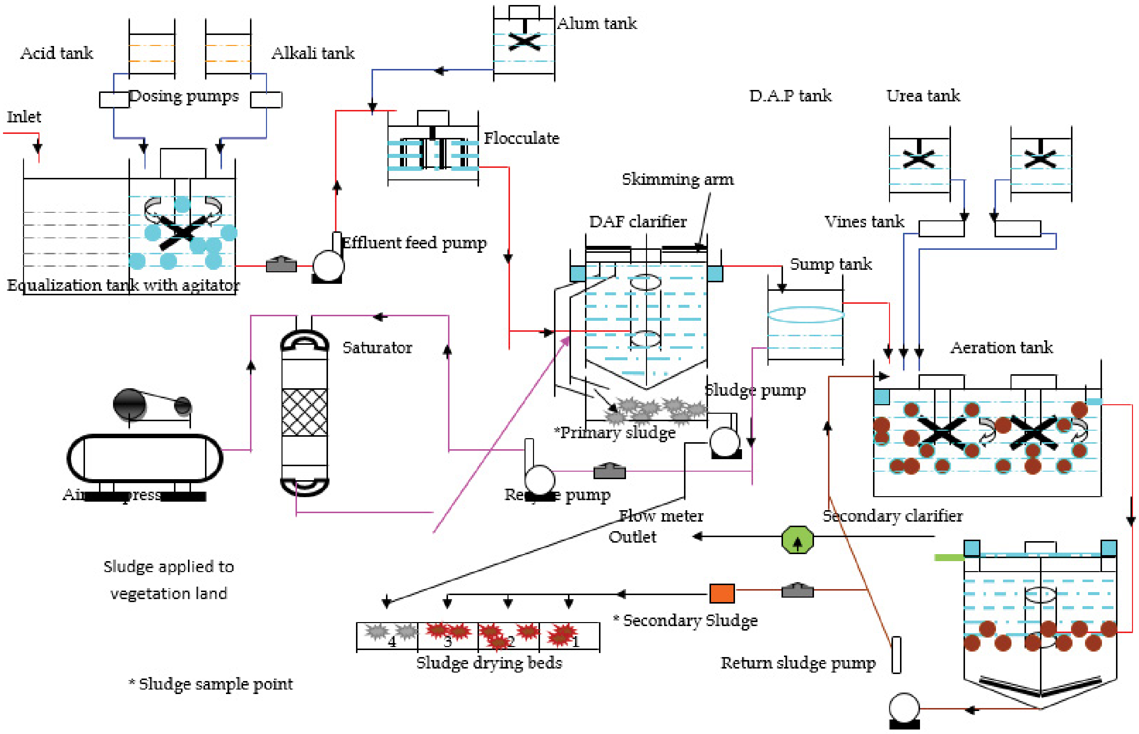


Fig. 2 Simplified schematic diagram of the conventional treatment system for the soya edible oil refinery wastewater.

effluent an evenly was stirred using a recycle pump at 1 ml /Sec through entire process (Fig. 1). After treatment time DC power supply and mixing purpose used recycle pump was stopped immediately and to allow sedimentation/flotation. The sedimentation/ flotation sludge collated for analysis purpose. The biological wastewater treatment showed in Fig. 2 and sludge collected from primary and biological sludge for this study.

Analytical techniques

The aqueous samples were taken at before treatment and physicochemical characterization using APHA standard method (Clesceri, *et al.*, 1998) and further, electrochemical generated sludge are collected and moisture is removed before the analysis. After natural drying and drying at constant temperatures of 103°C for 12 hr in oven, water in sludge is completely evaporated then sludge is stored in dryer. Finally, it is milled and passed through a 200 mesh sieve and analyzed for total organic carbon content using a Shimadzu TOC analyzer (TOC-VCPH model, Japan). The functional groups were observed by FTIR (Nicolet 10, USA). The physical characterization of sludge was obtained from SEM (LaB6 JEM-2010 (HT)-FEF (HRTEM) England).

RESULT AND DISCUSSION

A comparison of sludge produced during conventional (biological) treatment and electrochemical process with respect to soya oil refinery processing wastewater were evaluated and the results are present in Table 2 and Fig. 3-14.

Effect of initial pH and EC on treatment time

The initial pH of the solution is of vital importance in the performance of the electro chemical oxidation process. The generation of metal ions takes place at the anode and the hydrogen gas gets released at the cathode. The hydrogen gas helps in the flotation of the flocculated particles out of the water (Kumar, *et al.*, 2009). To study the effect of initial pH and EC on electro chemical oxidation, experiments were carried out by varying the initial pH from 3, 7 and

Table 2. Comparisons with conventional treatment sludge and electrochemical sludge

Treatment Process	Conventional Treatment		Electrochemical Process Sludge		
	Primary sludge	Biological sludge	pH 3	pH 7	pH 10
pH	5.18	5.64	6.72	7.12	6.52
EC (mS)	3.14	2.87	6.67	3.64	4.13
TDS (mg/l)	2790	2460	4100	2810	3430
Alkalinity (mg/l)	1500	1750	1800	1920	1860
Chloride (mg/l)	779.2	567.2	1276.2	283.6	1205.3
Total hardness (mg/l)	2380	2220	1620	1000	1280
Phosphate (mg/l)	187.5	25	55	40	15
Potassium (mg/l)	93.1	90.4	43.8	46.6	48
Nitrate (mg/l)	40	25	31.5	15	12.5
Sulphate (mg/l)	10	10.5	9.2	6.5	11
Sodium (mg/l)	121.6	69	275.5	82.9	186

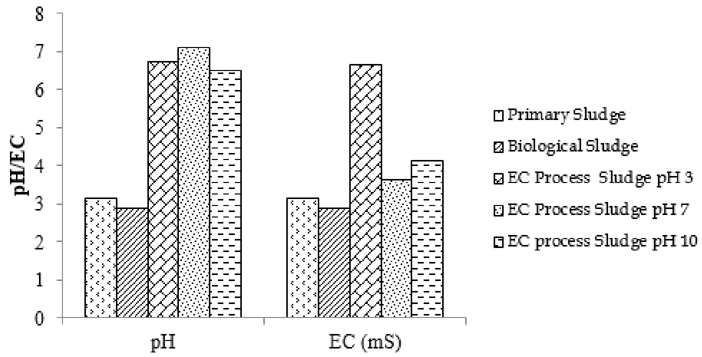


Fig. 3 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to pH and electrical conductivity.

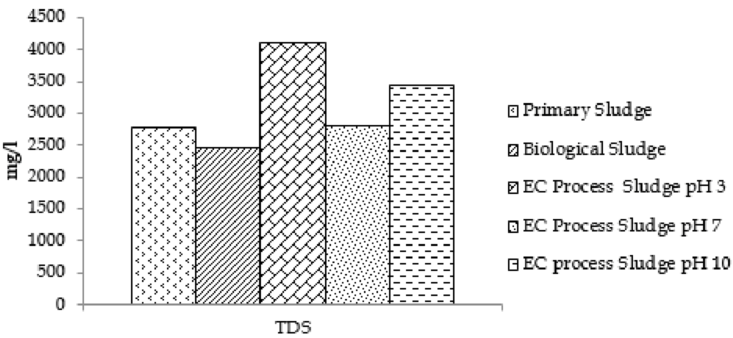


Fig. 4 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to total dissolved solids.

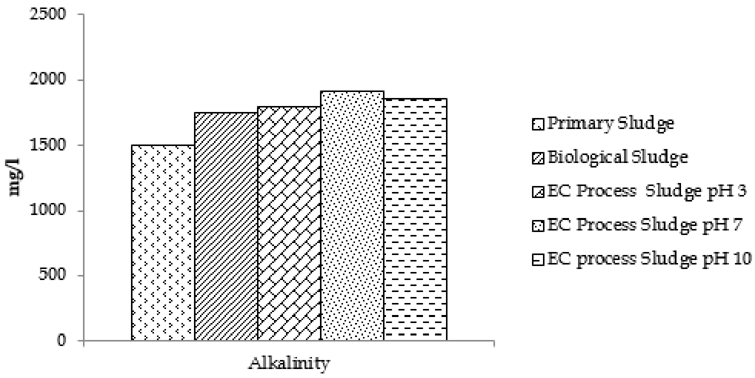


Fig. 5 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to alkalinity.

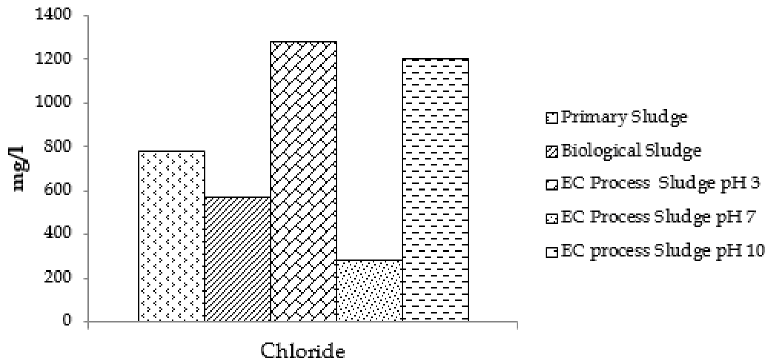


Fig. 6 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to Chloride.

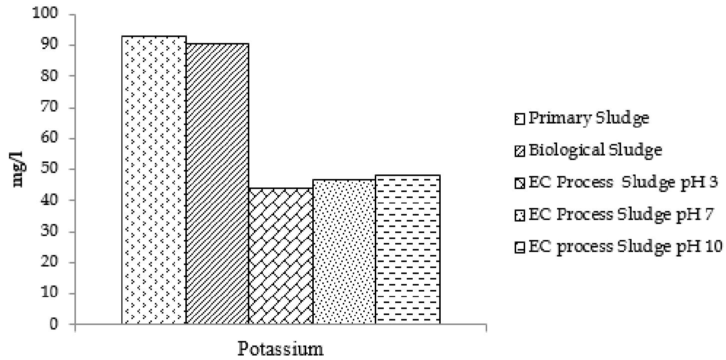


Fig. 7 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to potassium.

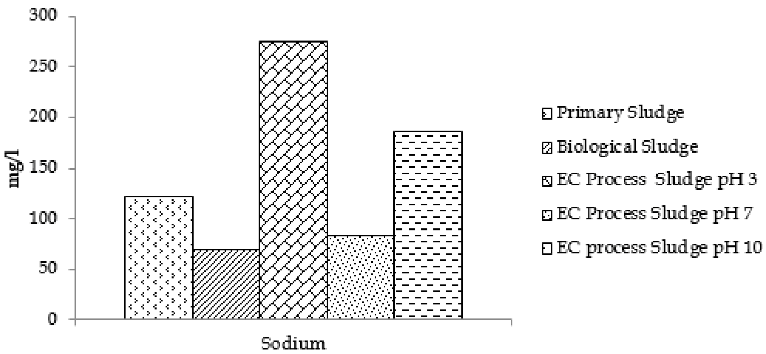


Fig. 8 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to sodium.

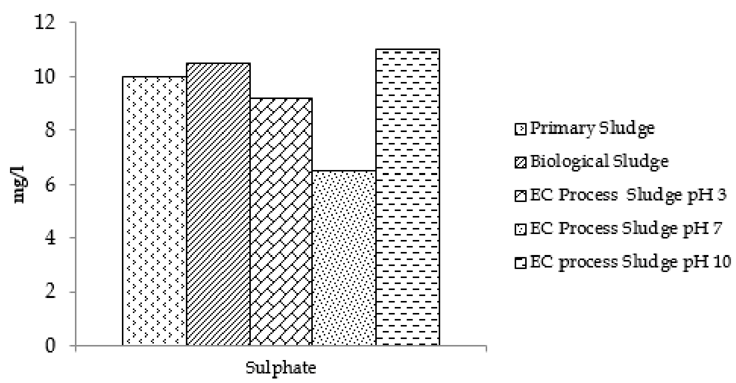


Fig. 9 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to sulphate.

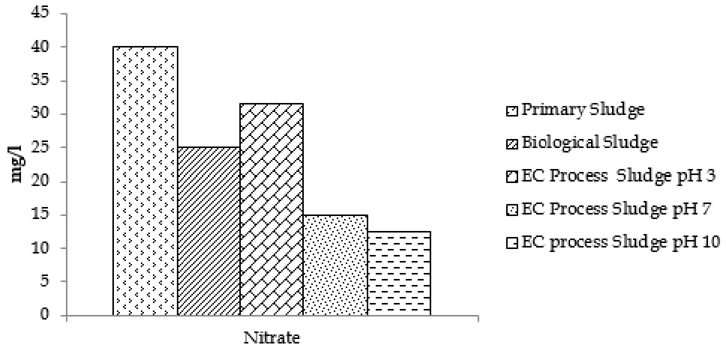


Fig. 10 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to nitrate.

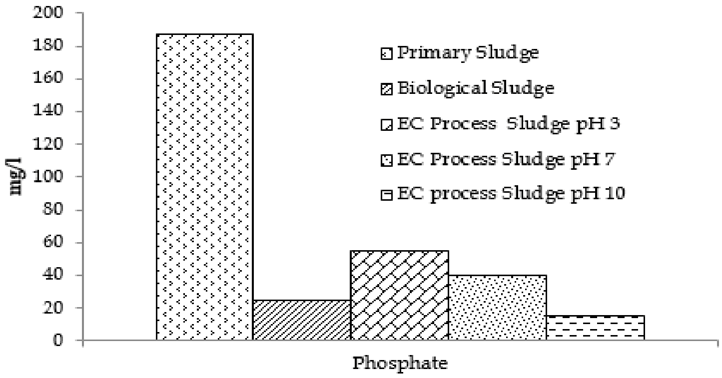


Fig. 11 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to phosphate.

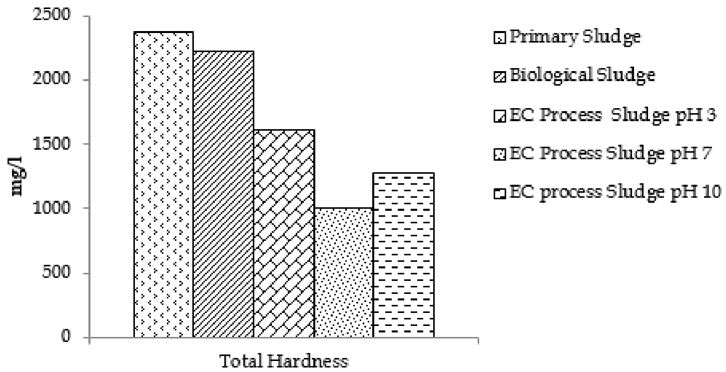
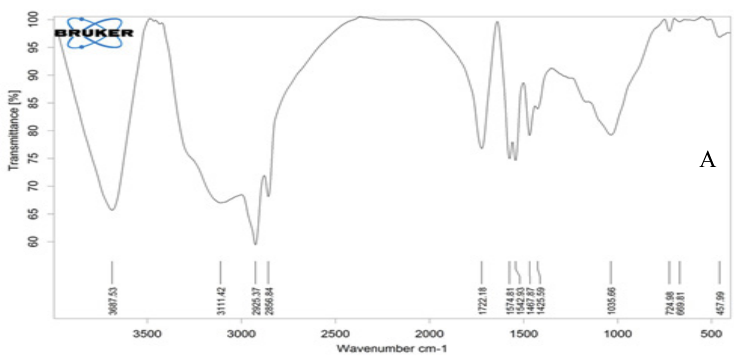


Fig. 12 Comparison of sludge produced during the conventional treatment of electrocoagulation process with respect to total hardness.



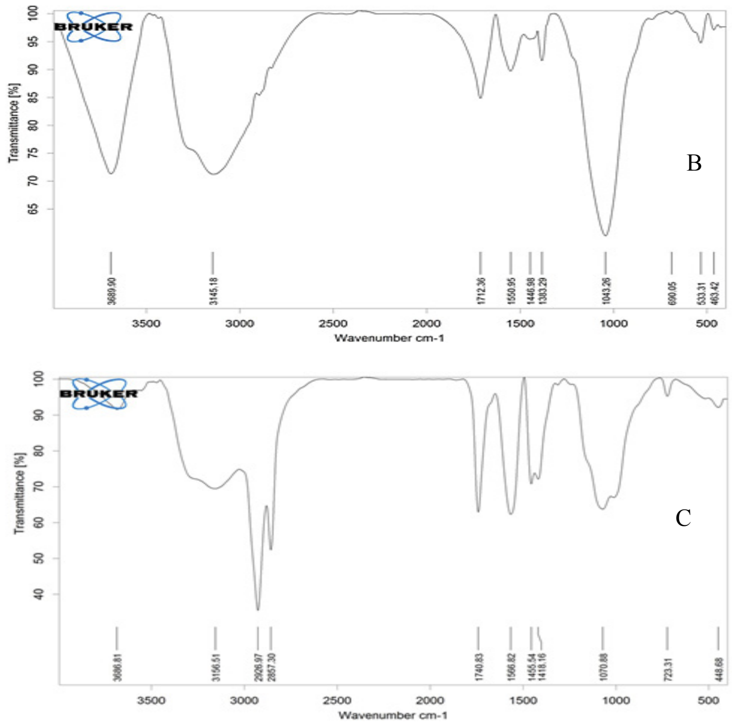


Fig. 13 Soya oil refinery traditional treatment sludge (a) Primary sludge (b) Secondary sludge and (c) electrochemical process sludge.

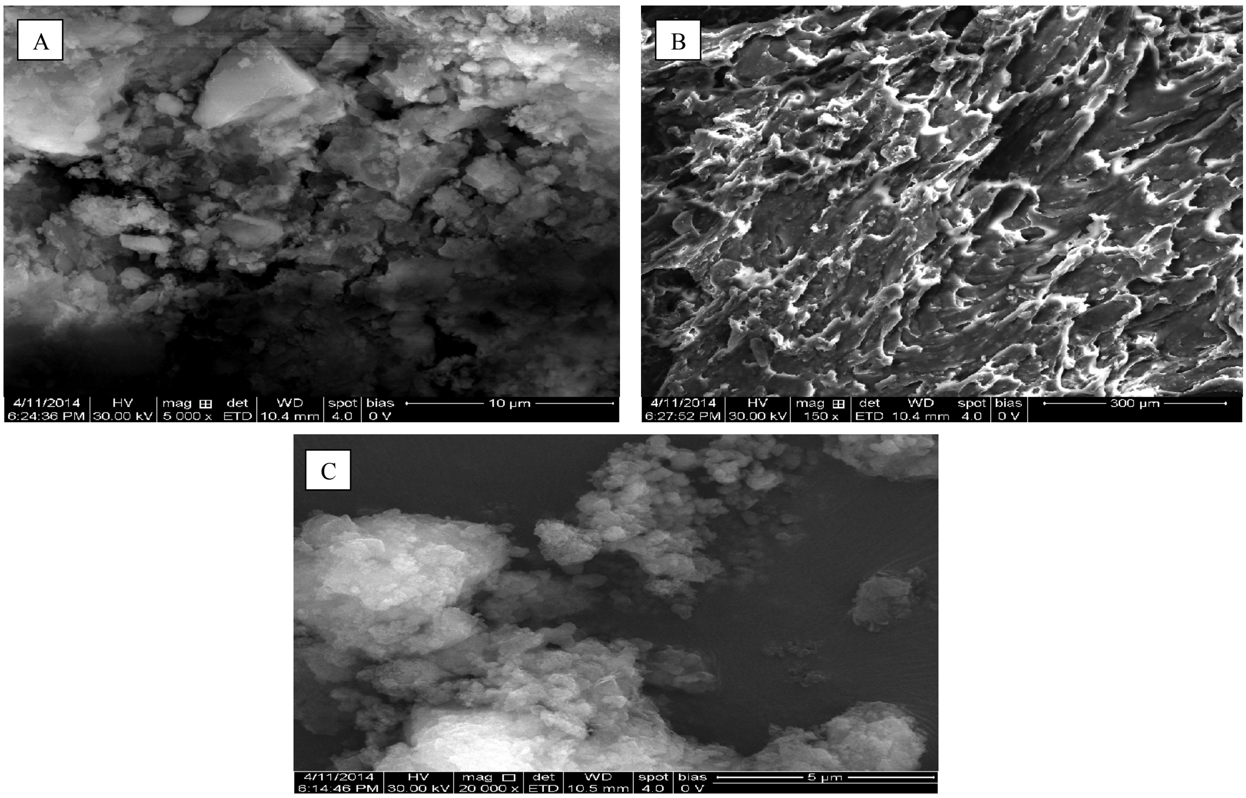


Fig. 14 SEM image of soya oil refinery conversional treatment sludge (a) Primary sludge 5000 x magnification (b) Secondary sludge 150 x magnification and (c) electrochemical treated sludge 20000 x magnification.

11 and at 3 hr of electrolysis time. The supporting electrolyte concentration of 0.1 M and applied current density 2.27 mA cm^{-2} of the sample was maintained for all the experiments.

The results are illustrated in Fig. 3. The results reveal that after electro chemical process the samples does not have their initial pH and EC range. Initially samples have to adjust the pH such as 3, 7 and 10 after 3 hr electro chemical process, the pH and EC value of the sample increased simultaneously in the range 6.8 to 7. In each electrochemical cell, there is a pH profile between the anode and the cathode. On the anode, the water oxidation process generates a high concentration of protons, resulting in a high pH.

Effect of total dissolved solids

From the result reviled that primary and secondary sludge (Biological sludge) have TDS value of 2800 mg/l and 2500 mg/l, respectively. After electrochemical process the total dissolved solid range will be increased (Fig. 4). The sample contain pH 3 the TDS value contain 4100 mg/l. Then the pH value of 7, 10 have TDS range 3000 mg/l and 3500 mg/l. Because of during the electrochemical process an electrodes getting decomposing and released the TDS in the process.

Effect of Alkalinity

The alkalinity of water is normally due to the presence of carbonates, bicarbonates and hydroxides of Ca, Mg, Na, and K. Borates, phosphates and silicates also contribute to alkalinity. Primary and biological sludge normally have acidic pH and alkalinity range between 1500 to 1700 mg/l. Ends of the electro chemical oxidation process pH 3 sludge have alkalinity of 1750 mg/l, pH 7 sludge have alkalinity range of 2000 mg/l, pH 10 sludge have alkalinity range of 1800 mg/l (Fig. 5).

Effect of chloride

The major impact that chlorides impart on the receiving waters is the permanent hardness. They are also known to increase the rate of sedimentation and thereby decreasing the water column depth. When such effluents are disposed on land, chlorides tend to initially percolate some distance, but over a period of time, they cause surface salt formation, thereby causing increased alkalinity of the soil, thereby resulting in loss of soil fertility (Aptesagar, *et al.,* 2011). Fig. 6 compare with other removal process electrochemical process has high chloride removal efficiency below result conform that the process. The chloride removal in order to pH 10>3>7 (1900, 1300 and 250 mg/l, respectively).

Effect of potassium

Potassium ions appear to play an important role in determining the nature of activated sludge flocs. Relative to sodium, the concentration of potassium ions in most industrial activated sludge is typically low. The concentration of potassium affected the concentration of readily extractable (slime) proteins in the floc and the proteins in the surrounding solution. Fig. 7 showed that the results confirmed initial samples (Primary sludge and biological sludge) have a potassium value of 92 mg/l and 90 mg/l after electro chemical process pH 3, 7 and 10 contain 48, 50 and 52 mg/l, respectively. So that the result proves electro chemical process effective to remove potassium especially pH 3 has excellent removal of potassium.

Effect of sodium

The content of Mg, C and Na in furnace ash used for the experiment was much higher than their soil concentrations. Fig. 8 results shows neutral pH has efficient removal of sodium ions 51 mg/l compare to acidic pH 250 mg/l and alkaline pH 200 mg/l.

Effect of sulphate

Wastewaters from edible oil production contain high concentration of organic pollutants, as well as high concentrations of phosphates and sulphates. Normally lime milk added to removes sulphates, which according to the Ruffer's (Ruffer, *et al.*, 1998) theory, permit sulphates precipitation by means of lime to a level above 2000 mg SO_4/dm^3. The Fig. 9 shows that primary and biological sludge have sulphate concentration of 8 mg/l and 10 mg/l, respectively. After electro chemical treatment process acidic and neutral pH (pH 3) sludge have low sulphate concentration of 8.5 mg/l and 6.0 mg/l in case of pH 10 sludge have higher concentration of sulphate 11 mg/l.

Effect of nitrate

Nitrate removal from wastewaters sludge is commonly achieved by employing the bacterial process of denitrification, in which nitrate is reduced to innocuous nitrogen gas (N_2). The process requires an electron donor to supply electrons (energy) to the bacteria. The electron donor is typically supplied in the form of a soluble organic substance, such as methanol. This is a costly procedure, which, however, results in high denitrification rates and low reactor volumes (Sivan Klas, *et al.,* 2006). In case of electrochemical oxidation process nitrate removal efficiency was showed in Fig. 10. Removal rate of nitrate in acidic pH is 31 mg/l, neutral pH 15 mg/l and alkali pH 13 mg/l, compared with other biological treatment process electro chemical process has high removal efficiency was high in sort time duration and within low cost.

Effect of phosphate

Removal of phosphates from waste water is important in order to protect lakes and other natural waters from cultural eutrophication. Conventional biological treatment processes remove only 50% or less of the sewage phosphate and substantial improvement is needed to achieve 90% or more removal to reach effluent concentration of 0.25 to 1.0 mg/l (Fig. 11). This can be accomplished by chemical means either in physical-chemical treatment processes or as part of the activated sludge process of wastewater treatment. In these processes, salts of iron, calcium, or aluminum are added to form sparingly soluble phosphates, which are then removed by settling. After electro chemical process pH 3, 7 and 10 have 45, 40 and 15 mg/l, respectively.

Effect of total hardness

Hard wastewater can cause scaling problems in water heaters and soap does not lather well in hard water. Therefore, some water utilities soften water to improve its quality for domestic use. Hardness in wastewater is primarily the result of concentrations of calcium and magnesium. Thus, some water utilities remove calcium and magnesium to soften the water and improve its quality for domestic use. Primary sludge has 2400 mg/l and biological treatment process sludge has 2300 mg/l of total hardness. So both processes don't have sufficient removal capacity. Fig. 12 showed that the after electro chemical process removal of total hardness pH 3, 7 and 10 have 1500, 1000 and 1300 mg/l, respectively.

FT-IR analysis

The FT-IR spectroscopic study of soya oil refinery traditional treatment sludge (a) primary sludge (b) biological sludge and (c) electrochemical process sludge is shown in Fig. 13. The sample showed four major absorption bands at 2900 cm^{-1} to 3500 cm^{-1}, 1300 cm^{-1} to 1750 cm^{-1}, 1000 cm^{-1} to 1250 cm^{-1} and 450 cm^{-1} to 750 cm^{-1}. A wide band with two maximum peaks can be noticed at 2930 cm^{-1} and 3450 cm^{-1}. The band at 3450 cm^{-1} is due to the absorption of water molecules as result of an O-H stretching mode of hydroxyl groups and adsorbed water, while the band at 2930 is attributed to C-H interaction with the surface of the carbon. However, it must be indicated that the bands in the range of 3200 cm^{-1} to 3650 cm^{-1} have also been attributed to the hydrogen-bonded OH group of alcohols and phenols (Yang, *et al.,* 2003). In the region 1300 cm^{-1} to 1750 cm^{-1}, amides can be distinguished on surface of the activated carbon which has two peaks at 1640 cm^{-1} and 1450 cm^{-1}. These functional groups were obtained during the activation process as a result of the presence of ammonia and primary amines that usually exist in the sludge. Moreover, the band at 1500 cm^{-1} may be attributed to the aromatic carbon–carbon stretching vibration. The two peaks at 1125 cm^{-1} to 1150 cm^{-1} yield the fingerprint of this carbon. The sharp absorption band at 1125 cm^{-1} is ascribed to either Si-O or C-O stretching in alcohol, ether or hydroxyl groups. The band at 1150 cm^{-1} can also be associated with ether C-O symmetric and asymmetric stretching vibration (-C-O-C- ring. This band could also be attributed to the anti-symmetrical Si-O-Si stretching mode as a result of existing alumina and silica containing minerals within the sludge samples. The region 450 cm^{-1} to 750 cm^{-1} show two bands in the 480 cm^{-1} and 485 cm^{-1} which are associated with the in plane and out-of-plane aromatic ring deformation vibrations. Peaks at 598 cm^{-1} and 680 cm^{-1} are assigned to the out-of-plane C-H bending mode. These spectra were also suggested to be due to alkaline groups of cyclic ketones and their derivatives added during activation.

SEM analysis

The structural morphology of the primary, biological sludge from conventional treatment and electrochemical process was analyzed using scanning electron microscope at an accelerating voltage of 10 kV under magnifications of 5000, 150 and 20000, respectively. Fig. 14 shows that structure morphology of sludge. The SEM images of the biological treated sludge shown in Fig. 14 (a and b), were irregular image and porous surface could be observed. Fig. 14 (c), the porous surface on the sludge gets filled by the oxidation ions. This observation indicates that oxide ions are adsorbed to the functional groups present inside the wall of the sludge surface. So, the morphological study of electro chemical treated sludge confirms that the oxidation takes place the surface of the electrodes.

CONCLUSIONS

The novelty of theme is particularly the case study and the conclusions that were drawn from this study. Soya oil refinery industry adopted biological treatment process produced large quantity of sludge in the form of primary and biological sludge. The sludge reduction observed electrochemical treatment system and comparison of both processed sludge micro and macro nutrients were observed and it was found that the sludge may have used as a fertilizer.

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