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# Treatment of Mature Landfill Leachate from Vilappilsala by Combined Chemical and Biological Process

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**Abstract**: Mature leachate contains significant quantities of refractory organic materials that are impossible to treat efficiently by a biological process. In the present study, for the treatment of mature landfill leachate from Vilappilsala, Fenton process is adopted as chemical pretreatment prior to anaerobic process. After Fenton pretreatment removal of COD, BOD, turbidity, nitrate, sulphate and TSS were 92.1%, 83.5%, 94.2%, 18.86%, 11% and 96.6% respectively (optimized treatment conditions pH 3, reaction time 30min,  $H_2O_2/COD$  ratio 3,  $[H_2O_2]/[Fe^{2+}]$  ratio 15 and 3g/L sludge recycling). Biodegradability (BOD<sub>5</sub>/COD ratio) enhanced from 0.3 to 0.65 and acute toxicity (96-hr LC<sub>50</sub>) reduced to 6.3% from 3%. Fenton treated leachate when post treated anaerobically at HRT 13 days and pH 7, COD, BOD, turbidity, nitrate, sulphate and TSS removal were 90.3%, 98.2%, 28.3%, 69.9%, 40% and 78.2% respectively and toxicity reduced to 22%.

Keywords: Acute toxicity, Biodegradability, Anaerobic process, Fenton process, Mature landfill leachate.

#### I. INTRODUCTION

Urban development, industrial and commercial growth together with increasing population is accompanied by ever increasing solid waste generation all over the world. Due to economic advantages, landfilling is still the most adopted solid waste disposal methods. The inevitable drawback of landfilling is the generation of leachate produced by physicochemical and biological decomposition of waste and percolation of rainwater through compacted wastes. Its composition is therefore site and time specific, based on the characteristics of deposited wastes, physico-chemical conditions, volume of infiltrating water and landfill age. Young leachate, usually formed within less than 2 years after waste deposition, contain more organic fraction of relatively low molecular weight and high biodegradability (BOD/COD>0.6) in comparison with old leachate, generated more than 10 years after deposition. In older leachate prevailing organics are humic and fulvic substances of relatively high molecular weight with persistent character, resulting in lower biodegradability. It has been confirmed, that biological processes are effective in treating young leachate. Among the biological treatment anaerobic treatment is more preferable because it is the most suitable treatment option for high strength waste water with comparatively less energy consumption and sludge production in addition to producing saleable end products like biogas and facilitating energy production. But with increasing age, biodegradability of leachate decreases and biological treatment could be completely ineffective. To treat the old/refractory leachate, alternative treatment methods like physical and chemical methods are needed. Treatment options like reverse osmosis, adsorption etc. are non-destructive, changing the pollutant load from one phase to another. In such a case, advanced oxidation processes represent an attractive alternative to biological processes. Advanced oxidation processes (AOPs) remove organic load to high degree together with improving the biodegradability. Of the various AOPs, Fenton process is the easiest to setup and cost effective process. Under acidic conditions, Fenton reagent (ferrous ion combined with hydrogen peroxide) produces OH<sup>-</sup> (second highest powerful oxidant). The mechanism of the Fenton process:

$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO^-$	(1)
$Fe^{2+} + HO' \rightarrow Fe^{3+} + OH'$	(2)
$HO' + RH \rightarrow H_2O + R'$	(3)

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(4)

#### $R' + Fe^{3+} \rightarrow R^+ + Fe^{2+}$

Several authors have studied the optimization of parameters influencing Fenton process and have achieved efficient removal of organics from landfill leachate [1,8,11,19]. Fenton process has been applied successfully in a wide range of other wastewater also [4,5,9]. Moreover, Fenton process have been successfully used as pretreatment methods in order to reduce the concentrations of toxic/recalcitrant organic compounds which further favored biological wastewater treatment processes. Anaerobic treatment of landfill leachate has been extensively studied [7,13,14]. Assessment of the biodegradability and toxicity during the oxidation process is necessary to determine the success of the combined process. BOD<sub>5</sub>/COD ratio is the commonly used method for assessing biodegradability. Toxicity is a consequence of numerous contaminants in leachate, their interactive or opposing effects, and different physical-chemical properties, and toxicity tests may thus give more information about leachate quality when compared to chemical analyses. Toxicity testing of Okhla municipal landfill leachate using Poecilia reticulata revealed that leachate was highly toxic with 96-h LC50 less than 3% [18]. Combined Fenton-aerobic treatment has been adopted for treatment of a wide range of wastewater [2,3,10,12,16]. Combined Fenton anaerobic treatment has been attempted and found to be successful on sludge (28.2% reduction in DS, 26.8% reduction in VS, 39.6% reduction in SS, and 46.3% reduction in VSS were obtained on anaerobic digestion of Fenton processed sludge with reference to the raw sludge) [6] and olive mill effluent (COD reduced from 112.50g/L to 8.30g/L and inhibition of Vibrio fisheri luminescence reduced from 100 to 45.2% on electro Fenton-anaerobic treatment [15], effluent COD concentrations from the ACF reactor, operated by using Fenton pre-treated OME reduced to 5700mg/L from initial 90000mg/L) [17].

#### II. **OBJECTIVE**

This research was conducted to investigate the efficacy and feasibility of Fenton pretreatment in removing the organics, improving the biodegradability and reducing toxicity of mature landfill leachate. This paper also aims to demonstrate the efficiency of a two-stage combination treatment: chemical oxidation using Fenton's reagent prior to anaerobic treatment in treating the mature landfill leachate and thus to advocate a hybrid technology of combining AOP and biological process for the treatment of wastewater containing biorecalcitrant and/or toxic pollutants so as to avoid the impact on environment.

#### III. METHODOLOGY

#### A. General

Mature leachate sample was collected from landfill site at Vilappilsala and characterization was done. Synthetic waste water similar in characteristics to the collected leachate sample was prepared and its characterization was done. Fenton treatment of synthetic leachate was done and optimization of treatment with respect to reaction time, pH,  $H_2O_2/COD$  ratio ,  $[H_2O_2]/[Fe^{2+}]$  ratio and sludge reuse done. Raw mature leachate was treated with Fenton process under the optimized conditions. Treatment efficiency including toxicity reduction and biodegradability improvement studied. An anaerobic reactor was designed and Fenton treated leachate was then anaerobically treated and optimization with respect to pH done. Efficiency of combined Fenton-anaerobic process in treating the mature leachate and reducing its toxicity studied.

#### B. Materials and Methods

1) Mature Landfill Leachate and its Characterization: The mature landfill leachate was collected from Vilappilsala municipal landfill, Trivandrum. The centralized solid waste treatment plant at Vilappilsala started functioning in the year 2000. The treatment plant occupies an area of 43 acres. This landfill is not equipped with a leachate collection and treatment system. Leachate collected was stored in closed containers at 4°C until use. The characteristics of the leachate used in the investigated period are listed in Table 1.

2) *Preparation and Characterization of Synthetic Leachate:* Proportion of components used for the preparation of synthetic wastewater was decided by trial and error so that a reasonable match with the chemical characteristics of actual leachate sample was achieved. Synthetic wastewater characteristics were determined using standard methods.



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*3)* Analytical Methods: The physical and chemical properties of the leachate were determined in accordance with standard methods (APHA,2005).Standard physical and chemical parameters, including; Turbidity, Conductivity, Temperature, Chemical oxygen demand (COD), Biochemical oxygen demand (BOD), Chlorides, Sulphate, Nitrate, Total solids, Total dissolved solids and Total suspended solids were determined.

4) Biodegradability and Toxicity Testing: Biodegradability was measured as BOD<sub>5</sub>/COD ratio. Toxicity testing done as per IS 6582-1971. Acute toxicity ( $LC_{50}$ ) of leachate determined using static 96h fish bioassay.  $LC_{50}$  is the lethal concentration that causes death for 50% of the test organisms. Test organism, *Poecilia reticulata* (guppy fish) was obtained from a commercial dealer. Test fishes acclimatized for 10 days to laboratory conditions in dilution water. Dilution water had DO content above 4mg/L. Fish were fed fairly during the acclimatization period. Fish not fed for about 48hrs before the test and during the test. Preliminary test was done in order to find the desired range of concentration to be covered in the full scale test. Full-scale bioassay was done and  $LC_{50}$  was estimated graphically for raw, Fenton treated and Fenton-anaerobically treated leachate.

5) Materials and Reagents for Fenton Process: Fenton process was carried out in a 1000 ml beaker, and constant stirring done with magnetic stirrer. Reagents used include Hydrogen peroxide as oxidant, ferrous sulphate heptahydrate (FeSO<sub>4</sub>.7H<sub>2</sub>O) as source of Fe<sup>2+</sup>, NaOH and H<sub>2</sub>SO<sub>4</sub> solutions for pH adjustment.

6) Materials and Inoculum for Anaerobic Process: A 7L laboratory scale anaerobic reactor of height 40cm and diameter 15cm was used. The reactor was a cylindrical column made of cast iron. The reactor was provided with suitable arrangements for feeding, gas collection and draining of residues. The reactor was operated at ambient temperature. Gas production was measured using the water displacement method. Conc.H<sub>2</sub>SO<sub>4</sub> and NaOH was used for pH adjustment. A mix of anaerobic sludge from biogas plant, Sreekaryam and cow dung slurry was used as inoculum.

7) Fenton Process: Fenton process was carried out in batch mode in a glass beaker of 1L capacity. Fenton process is normally composed of four successive stages: pH adjustment, oxidation reaction, neutralization and coagulation, and precipitation. For oxidation to happen, pH of 500ml sample adjusted to the required value with Conc.H<sub>2</sub>SO<sub>4</sub> and NaOH. A weighed amount of FeSO<sub>4</sub>·7H<sub>2</sub>O was added and dissolved under stirring to achieve the scheduled ferrous ions. The reaction was started by adding the required dose of 30% (w/w) hydrogen peroxide. The reaction solution stirred with a magnetic stirrer at a constant speed. After the required time, pH was neutralized to 7 to initiate coagulation. Stirrer turned off and the sludge allowed to get settled. Supernatant withdrawn from the reactor at pre-determined times and analyzed. A set of experiments was conducted and the optimum concentrations of H<sub>2</sub>O<sub>2</sub> and FeSO<sub>4</sub>·7H<sub>2</sub>O, optimal reaction time and pH found out. Effect of sludge reuse studied.

8) Anaerobic Process: Leachate treated with Fenton process was then treated anaerobically. Preparation of inoculum was done by mixing cow dung with anaerobic sludge. Start up of reactor was done by adding cow dung slurry and anaerobic sludge in the ratio 1:1. Then it was left for stabilization for about 14 days. After stabilization, the reactor was fed with leachate and kept for the desired HRT of 13 days. Leachate was allowed to stabilize under anaerobic condition. Optimization of pH was done. Biogas produced was collected by the water displacement method.

9) Experimental set up



Fig. 1 Experimental Setup for Fenton Process www.ijirset.com



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Fig. 2 Experimental Setup for Anaerobic Process



Fig. 3 Full scale bioassay test of raw leachate



Fig. 4 Full scale bioassay test of Fenton treated leachate



Fig. 5 Full scale bioassay test of Fenton-anaerobic treated leachate



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#### IV. RESULTS AND DISCUSSION

A. Characterization of Mature Landfill Leachate

TABLE I

Value Parameter pН 8 Temperature(<sup>0</sup>C) 30 Colour Black COD(mg/L) 24000 BOD (mg/L) 7500 BOD<sub>5</sub>/COD 0.3 Total Solids (mg/L) 17200 Total dissolved solids(mg/L) 8000 Total suspended solids(mg/L) 9200 VSS (mg/L) 5612 Chlorides (mg/L) 210 Sulphide (mg/L) 160 Turbidity(NTU) 280 Sulphate (mg/L) 320 Nitrates(mg/L) 200 Conductivity(micromho/cm) 17200 3.2% Acute toxicity(96-hr LC<sub>50</sub>)

#### CHARACTERISTICS of COLLECTED LEACHATE

#### B. Characterization of Synthetic Leachate

#### TABLE II

#### SYNTHETIC LEACHATE CHARACTERISTICS

Parameter	Value
pH	7.8
COD(mg/L)	24860
BOD (mg/L)	6464
BOD <sub>5</sub> /COD	0.26
Total Solids (mg/L)	16400
Total suspended solids(mg/L)	8000
Turbidity(NTU)	286
Sulphates (mg/L)	355
Nitrates(mg/L)	298

#### C. Fenton Process

Optimization of Fenton Reaction condition using synthetic leachate was done. The effect of major parameters on the Fenton process for treating mature landfill leachate was evaluated in this study by the traditional one-factor-at-a-time method using a bench-scale batch reactor. These optimized parameters were reaction time, pH,  $H_2O_2/COD$  ratio,  $H_2O_2$  to Fe (II) molar ratio and sludge reuse.

1) Optimization of Reaction Time: The objective of the first set of test was to determine the time required for the<br/>treatment. Stoichiometric weight ratio  $H_2O_2/COD=2.125$  calculated assuming complete oxidation of COD (1g COD<br/>=1g  $O_2=0.03125$  mol  $O_2=0.0625$  mol  $H_2O_2$ ). Initial test conditions chosen were pH of 7.8,  $H_2O_2/COD$  ratio 2,  $H_2O_2$ <br/>Copyright to IJIRSETwww.ijirset.com4409



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to Fe (II) molar ratio 50. Keeping these conditions, Fenton oxidation reaction time was varied from 15 to 75 min. Results showed that organic materials were rapidly degraded by Fenton reagents. Most of the removal occurred in first 30 min after which only a minor reduction in removal was observed. Lower residence times will allow energy savings for stirring.



Fig. 6 Effect of reaction time on Fenton treatment (pH =7.8, T= $30^{\circ}$ C, H<sub>2</sub>O<sub>2</sub>/COD=2, [H<sub>2</sub>O<sub>2</sub>]/ [Fe<sup>2+</sup>] = 50)

2) Optimization of pH: The tested pH range was 1 to 9 using a dosage of  $H_2O_2/COD$  ratio 2,  $H_2O_2$  to Fe (II) molar ratio 50 and the reaction carried out for the optimized time period 30 min. Results from the pH study showed that maximum

removal occurred at pH levels below 5. This is because of the fact that OH<sup>•</sup> radical are effectively produced under acidic pH. Increasing the pH above 5 decreased the removal efficiencies and in the acidic range itself pH 3 produced slightly better results.



Fig. 7 Effect of pH on Fenton treatment (Reaction time =30 minutes,  $T=30^{\circ}C$ ,  $H_2O_2/COD=2$ ,  $[H_2O_2]/[Fe^{2+}] = 50$ )

3) Optimization of  $H_2O_2$  Dosage: In Fenton's reagent, hydrogen peroxide is catalyzed by ferrous iron to produce the active OH<sup>•</sup> radical; and hence it is important to optimize the dosage of both  $H_2O_2$  and  $Fe^{2+}$ . When the peroxide dose was varied over a range of 1 to 5mg/L per mg/L of COD at a pH of 3 and an  $H_2O_2$  to Fe (II) molar ratio of 50 and oxidized for a 30min reaction time, an increase in oxidation efficiency up to mass ratio 3 was observed, after which slight reduction in removal was observed due to the fact that excessive reagent produce the hydroxyl radical scavenging effect.



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Fig. 8 Effect of  $H_2O_2$  dosage on Fenton treatment (Reaction time =30minutes, pH=3, T=30<sup>o</sup>C, [H<sub>2</sub>O<sub>2</sub>]/[Fe<sup>2+</sup>] = 50)

4) Optimization of  $Fe^{2+}$  Dosage: The molar ratio of H<sub>2</sub>O<sub>2</sub>:Fe<sup>2+</sup> is important since too little iron will result in unutilized H<sub>2</sub>O<sub>2</sub> and excessive Fe<sup>2+</sup> will destroy produced OH• radical. When H<sub>2</sub>O<sub>2</sub> to Fe (II) molar ratio was varied over a range of 1 to 100 at a pH of 3, reaction time 30 min and H<sub>2</sub>O<sub>2</sub>/COD ratio 3, an increase in the removal efficiency up to a molar ratio of 15 was observed, after which reduction in removal happened.



Fig. 9 Effect of Fe<sup>2+</sup> dosage on Fenton treatment (Reaction time=30minutes, pH=3, T=30<sup>0</sup>C, H<sub>2</sub>O<sub>2</sub>/COD=3)

5) *Effect of Sludge Reuse:* Sludge was recycled into the influent to find out the effect of sludge accumulation in Fenton treatment process. Recycling of 3g Fenton sludge per litre of influent enhanced the COD removal efficiency up to a significant level.



Fig. 10 Effect of sludge recycling on Fenton treatment (Reaction time=30minutes, pH=3, T=30<sup>o</sup>C, H<sub>2</sub>O<sub>2</sub>/COD=3,  $[H_2O_2/COD=3, CD_2]/[Ee^{2+1}] = 15)$ 

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$$[H_2O_2]/[Fe^{2^+}] = 15)$$
  
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6) Treatment of Raw Leachate Using Optimized Reaction Conditions: Optimized Fenton reaction conditions of reaction time 30 min, pH 3, H<sub>2</sub>O<sub>2</sub>/COD ratio 3, H<sub>2</sub>O<sub>2</sub> to Fe (II) molar ratio 15 and 3g/L sludge reuse applied to raw leachate. The Fenton treatment removed 92.1%, 83.5%, 94.2%, 18.86%, 11% and 96.6% removal of COD, BOD, turbidity, nitrate, sulphate and TSS respectively. Biodegradability of leachate improved to 0.65 and toxicity reduced to 6.3%.



Fig. 11 Removal efficiency of raw and synthetic leachate upon Fenton treatment



Fig. 12 Biodegradability of raw and synthetic leachate after Fenton process

#### D. Anaerobic Treatment of Fenton Treated Leachate

Fenton treated leachate at pH 7 with improved biodegradability and reduced toxicity was fed to anaerobic reactor and allowed to stabilize under an HRT of 13 days. pH variation of 6 and 8 was also attempted in order to study the variation in treatment efficiency and it was observed that slightly higher removal occurred at pH 7 itself.



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Fig. 13 Effect of pH on anaerobic treatment efficiency of Fenton pretreated leachate

CHARACTERISTICS OF RAW, FENTON TREATED AND FENTON-

ANAEROBIC TREATED LEACHATE			
Parameter	Raw leachate	Fenton treated	Fenton- anaerobic
COD(mg/L)	24000	1896	treated 189.6
BOD(mg/L)	7500	1237.5	24.75
Turbidity(NTU)	280	16.24	11.69
Nitrates(mg/L)	200	162.28	48.8
Sulphate (mg/L)	320	284.8	170.88
TSS (mg/L)	9200	312.79	68.18



Fig. 11 Raw, Fenton treated and Fenton-anaerobic treated leachate

#### V. CONCLUSION

Landfill leachate obtained from the Vilappilsala landfill site was treated using combined chemical-biological process. Based on the test results of each treatment, optimal operating conditions identified. Fenton treatment at optimized conditions (pH 3, reaction time 30min,  $H_2O_2/COD$  ratio 3,  $[H_2O_2]/[Fe^{2+}]$  ratio 15 and 3g/L sludge recycling) removed 92.1%,83.5%,94.2%,18.86%,11% and 96.6% of COD, BOD, turbidity, nitrate, sulphate and TSS respectively.



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Treatment with Fenton process improved biodegradability from 0.3 to 0.65 and reduced toxicity by 50% which together favored biological post treatment. Fenton process was an efficient chemical pretreatment prior to anaerobic process. Anaerobic post treatment of Fenton treated leachate at HRT 13days and pH 7 further removed 90.3%, 98.2%, 28.3%, 69.9%, 40% and 78.2% COD, BOD, turbidity, nitrate, sulphate and TSS respectively. Combined chemical-biological process reduced COD, BOD, turbidity, nitrate, sulphate and TSS to levels 189.6mg/L, 24.75mg/L, 11.69NTU, 48.8mg/L, 170.88mg/L and 68.18mg/L respectively. Toxicity was reduced to 22% after the combined treatment. Thus the overall treatment chain of Fenton's process followed by anaerobic treatment seems to be an effective process for the treatment of mature leachate.

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