Research Article

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Integrated Heterogeneous Electro-fenton Process and Constructed Wetland for the Treatment of Stabilized Landfill Leachate

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ABSTRACT

Treatment of stabilized landfill leachate is a great challenge due to its poor biodegradability. The present study made an attempt to treat this wastewater by combining electro-Fenton (E- Fenton) and constructed wetland (CW). E-Fenton treatment was applied prior to CW process to enhance the biodegradability of leachate, which will be beneficial for the subsequent biological process. This study also investigates the efficiency of magnetite (Fe_3O_4) nanoparticles as a heterogeneous catalyst in E-Fenton process. The objectives of this study are to synthesize chitosan to obtain magnetite nanoparticles *via* iron ions assembly and to characterize the synthesized nanoparticles using SEM, XRD and FTIR analysis to assess its nature. The effects of initial pH, catalyst dosage, applied voltage and electrode spacing on Chemical Oxygen Demand (COD) removal efficiency were analysed to determine the optimum conditions. Heterogeneous E-Fenton process gave 84.29% COD removal at catalyst dosage of 50 mg/L, pH 2, electrode spacing 3 cm, voltage 5 V and effective electrode area 25 cm^2. The proficiency of utilizing eggshells as a media in constructed wetlands was evaluated for the treatment of wastewater previously subjected to electro-Fenton treatment. And it was found that combined E-Fenton and biological treatment (CW) resulted in overall COD removal of 95.79%, bringing down the final COD to 292 mg/L. Thus, the combination of the E-Fenton process followed by a biological process is efficient for complete mineralization with lower operational cost.

Keywords: Stabilized landfill leachate; Electro-Fenton; Constructed wetland; Magnetite nanoparticles.

1. INTRODUCTION

Urbanization is contributing to a notable increase in the generation of both solid and liquid waste and this surge in waste production poses more significant challenges for disposal processes, particularly in developing countries like India. But governments face constraints due to limited finances and inadequate services (Sohail and Afzal, 2011). Landfill is a prevalent method for waste removal and disposal, represents as end point in managing municipal solid waste. This widespread practice has emerged as a significant global environmental concern, contributing to contamination and pollution (Yaashikaa *et al.* 2022). Leachate is a contaminated liquid emerging from the landfill sites and is generated through two processes. When rainwater interacts with the waste, it permeates through, dissolving soluble substances in the process. Additionally, certain degradation reactions within the waste generate acidic liquids, which further increases the harmful nature of the resulting leachate. Leachate classification depends on the landfill's age, with categories including young ($BOD_5/COD > 0.6$), intermediate (BOD5/COD=0.3-0.6), and stabilized/ mature (BOD5/COD < 0.3). Only in case of high BOD5/COD ratio and elevated concentration of low molecular weight organics in young leachate makes it well-suited for biological treatment techniques (Baiju *et al.* 2018).

Even though there are various physical and biological methods involved in the treatment of landfill leachate, each has its own limitations. When treating aged landfill leachate with low biodegradability, biological processes may not achieve good results due to the stubborn nature of organic carbon, and the simultaneous high concentration of ammonia nitrogen and heavy metals in the leachate, since it has a strong retarding impact on microorganisms (Zijing *et al.* 2022). Physicochemical techniques emerge as a more suitable option for treating mature landfill leachate, characterized by a low BOD5/COD ratio and elevated concentrations of high molecular weight refractory organics (Deng, 2007). Among the advanced physico-chemical processes such as electrocoagulation and electro-oxidation, the necessity in constant replacement of the sacrificial anode in electrocoagulation and formation of a sludge containing significant amounts of metal ions and persistent pollutants, posing challenges in its treatment. Additionally, electro-oxidation process has a drawback of leading to the formation of toxic chlorinated byproducts, especially when treating landfill leachate with a high chloride ion content.

Advanced Oxidation Processes (AOP) are a group of efficient technologies which help in the removal of variety of persistent pollutants in matured leachate by the hydroxyl and sulphate radicals which have high oxidation potential. Among other different AOPs, Fenton and Fenton like processes have gained much attention in recent years in refractory organics (Bandala *et al.* 2021). Fenton's process is defined as the catalytic generation of hydroxyl radicals (•OH) resulting from the chain reaction between ferrous ion and hydrogen peroxide (Zhang *et al.* 2005). In the Electro-Fenton process, which is a combination of electrochemistry and Fenton's reagent, H_2O_2 is electrochemically generated to produce hydroxyl radicals, which are highly reactive and capable of oxidizing a wide range of organic compounds. By using carbon- based electrodes, the overall reaction rates can be enhanced in the Electro-Fenton process, leading to more efficient production of hydrogen peroxide. This can be attributed to the unique surface properties and conductivity of carbon materials (Klidi *et al.* 2019). The traditional Fenton method, which employs a homogeneous catalyst, faces obstacles like a slower rate of ferrous ion regeneration and increases sludge

production. In order to resolve these issues, heterogeneous Electrochemical Advanced Oxidation Processes (EAOPs) have been studied (Baiju *et al.* 2018).

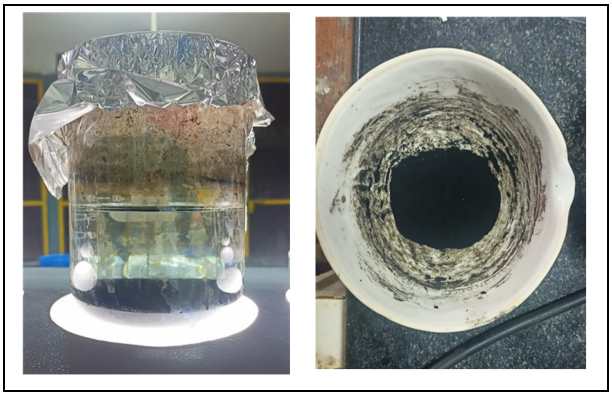


Fig. 1: Preparation of catalyst

Several biological treatment methods, such as anaerobic sludge blankets (UASB), activated sludge and sequencing batch reactors (SBR), are employed for leachate treatment. However, these processes are considered costly due to high expenses associated with construction, maintenance, and operation. In contrast, constructed wetlands offer an eco-friendly and effective alternative as a secondary treatment (Bakhshoodeh *et al.* 2020). Constructed wetlands (CW) are man-made artificial treatment systems that imitate the functions of natural wetlands in a controlled condition (Wdowczyk *et al.* 2022). Constructed wetlands, recognized as effective secondary treatment systems, purify wastewater through various processes. These include physical processes like sedimentation and filtration, chemical processes such as precipitation and adsorption, and biological actions like microbial degradation, uptake from the water, and interactions in the root zone (Bakhshoodeh *et al.* 2020). Therefore, recognizing the drawbacks of electro-Fenton and CWs, this study proposes an integration of these methods to collectively overcome their individual disadvantages. In the combined process, Electro-Fenton process as an initial step would convert the refractory organic pollutants into more biodegradable intermediates, which could be easily removed in a subsequent constructed wetland system.

2. MATERIALS AND METHOD

2.1 Study Area and Sample Collection

Leachate used in the present study was collected from Perungudi Dumping Yard which is located at 10 km from Chennai, Tamil Nadu. The location is 1.2 km from Perungudi village and dumping yard lies between 2 to 3 km west of Buckingham Canal and 3.5 to 4.5 km west of Bay of Bengal Coastline. Nearly 20 L of samples were collected in plastic cans and were stored at 4 °C to prevent biodegradation. Leachate characterization was done as per standard methods.

2.2 Chemicals

Sodium molybdate (Na_2MoO_4), orthophosphoric acid (H_3PO_4) and ferric nitrate monohydrate ($Fe(NO_3)_3.9H_2O$) were used in the catalyst preparation. Other chemicals used in the E-Fenton study include H_2SO_4 and NaOH for pH adjustment. All the chemicals used in the study were obtained from Merck and were of analytical grade.

2.3 Preparation of Catalyst

Chitosan, a polysaccharide comprising amino and acetamido groups in its monomeric units, has numerous primary amines ($–NH_2$) and hydroxyl (–OH) groups, providing active sites for effective adsorption (Bhatt *et al.* 2023). The resulting hydrogel, based on chitosan, demonstrates notable mechanical strength and chemical stability. Integrating hydrogels with chitosan elevates the overall adsorption capacity and efficiency in water treatment. The enhancement of mechanical strength in chitosan-based hydrogels can be achieved by introducing nanoparticles or crosslinking with synthetic polymers or biopolymers (Chelu *et al.* 2023). Moreover, the magnetite nanoparticles, owing to their distinctive physiochemical properties, cost-effectiveness, and facile recovery in the presence of external magnetic fields, are widely employed in water treatment applications (Aragaw *et al.* 2021).

The formulation of the catalyst has been documented in an earlier publication (Wang *et al.* 2008). The catalyst formulation involves the initial preparation of a 3% chitosan solution. This was achieved by dissolving 3 g of chitosan powder in 100 mL of a 2% (v/v) acetic acid solution. To ensure a homogeneous composition, vigorous stirring was employed, incorporating 0.3 mL of a 50% GLA (Glutaraldehyde) solution into the 100 mL chitosan solution. The solution was then left undisturbed until the chitosan hydrogel was fully formed, facilitated by the cross-linking effect of GLA. This was followed by the Synthesis of Fe_3O_4 nanoparticles induced by chitosan *via* iron ions assembly.

The chitosan hydrogel underwent a 30-minute soaking in $FeCl_3$ solution, followed by rinsing with deionized water. Subsequently, the hydrogel was immersed in $FeCl_2$ solution for another 30 minutes, followed by a rinse with deionized water. This cycle was repeated three times for thorough incorporation of iron ions into the chitosan hydrogel. The resulting chitosan hydrogel with iron ions was soaked in a 5mol/L NaOH solution for 12 hours, resulting in the formation of black Fe_3O_4 nanoparticles. To eliminate excess NaOH, the Fe_3O_4 nanoparticles in the chitosan hydrogel were washed with deionized water until the pH approaches 7, and then dried in an oven at 60 °C (Wang *et al.* 2008).

Finally, the point of zero charge (PZC) test was conducted to identify the pH at which a particular material, such as a solid surface or colloidal particles, becomes electrically neutral. At the PZC, the surface charge was effectively neutralized (Kosmulski, 2021). It was determined by solid addition method as mentioned in Wang *et al.* (2008), where initial pH (pH_0) values ranging from 2 to 10 is adjusted in 100 mL conical flasks containing 45 mL of 0.1 M KNO_3 solution. After adding 1 g of magnetite to each flask, the difference between the initial and final pH values ($\Delta pH = pH_0 - pH_f$) will be plotted against the initial pH (pH_0), and the point of intersection on the resulting curve will indicate the PZC.

2.4 Experimental set up of Electro-Fenton

In electro-Fenton process, the experiment was carried out in a batch reactor consisting of 1000 mL borosilicate glass beaker with 750 mL as working volume. Graphite electrode was used as anode and cathode respectively. Dimensions of the graphite electrode were 9 cm × 8.5 cm × 0.4 cm for anode and 9 cm × 8.5 cm × 0.5 cm as cathode. For E-Fenton process, the effective area of the electrodes will be kept as 25 cm^2.

The electrodes were connected to a DC power supply and aeration was given near cathode using a simple fish aerator *in situ*. Samples were withdrawn at 10 minutes regular time intervals and centrifuged from which the supernatant will be collected for COD estimation. All the electrodes were thoroughly cleaned after every run with water and soaked in 1 N HCl. The electrodes were stored in distilled water when not in use.

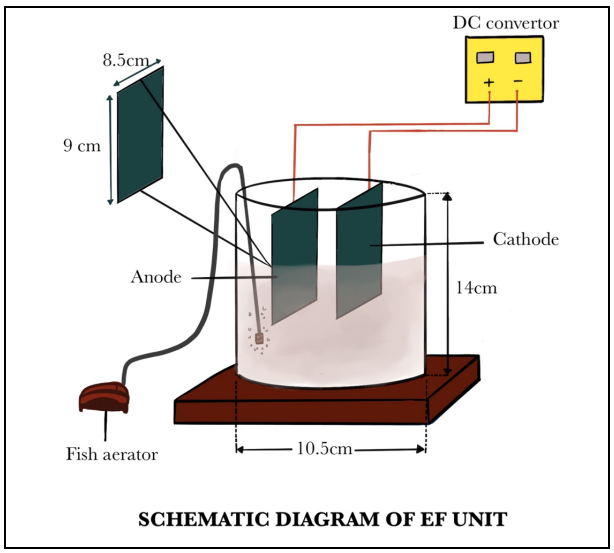


Fig. 2: Schematic representation of E-Fenton process

2.5 Experimental Setup of Constructed Wetland

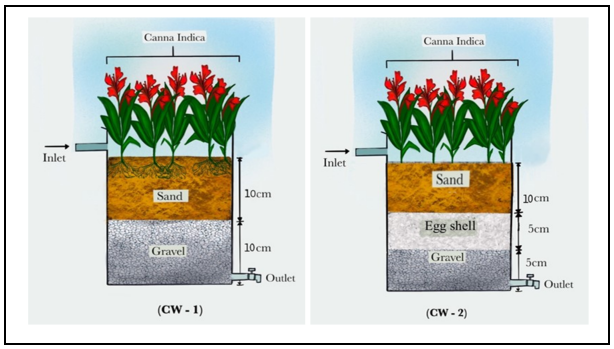


Fig. 3: Schematic representation of CW-1 and CW-2

Two Constructed Wetlands, CW-1 and CW-2 were fabricated using plastic water can of dimensions (30 cm diameter, 40 cm height and 3 mm thickness). CW-1 with 2 layers consisting of top layer of 10 cm with sand (2.36 mm passing), and bottom layer of 10 cm with gravel (5 to 10 mm) and CW-2 with 3 layers consisting of top layer of 10 cm with sand (2.36 mm passing), intermediate layer of 5 cm with eggshell (≤ 1mm) and bottom layer of 5 cm with gravel (5 to 10 mm). *Canna indica* was collected from the Anna University STP, Chennai. The bottom ends of both systems were fitted with PVC taps (sampling port) and made water tight by sealing with epoxy; while the upper ends were left free to atmosphere. The schematic diagrams of both CW-1 and CW-2 are shown in Fig. 3. The systems were kept in laboratory open to sky. The experiment analysis was started after one month when two setups with vegetation grown to a good extent.

The porosity and size of the media were analysed as per IS: 1498 – 1970. The porosity of the sand was 35%. Both sand and gravel were collected from an ongoing construction site at Anna University campus, Chennai. The collected gravel was washed, dried and sieved using a sieve size less than 2 mm, passed on 4 mm and retained on 3 mm. Gravel was used as supporting media for the above filter bed. It also acts as an attaching media for the microbes and for easy drainage of the leachate.

3. RESULTS AND DISCUSSION

3.1 Characterization of Leachate

The leachate was allowed for testing the influent characteristics of different physical and chemical parameters. The influent colour was noticed to be grey to black in colour. Leachate characterization was done as per standard methods and the obtained results are given in Table 1. The collected leachate had a pH of 8.41 and BOD/COD value of 0.169, and can thus be categorized as stabilized landfill leachate. Chemical Oxygen Demand (COD) of stabilized landfill leachate will normally be less than 4000 mg/L, but the collected leachate had COD higher than 4000 mg/L. Similar results have been reported by Baiju *et al* (2018).

Table 1. Initial parameters of leachate

Experiment	Unit	Influent		Average
pH	-	8.29	8.54	8.41
Electrical Conductivity	µS/cm	30400	29360	29880
BOD	mg/L	1110	1250	1180
COD	mg/ L	6880	7010	6945
BOD/COD	-	0.161	0.178	0.169
TDS	mg/ L	44000	44880	44440
TSS	mg/ L	650	660	655
TS	mg/ L	44650	45540	45095
Cl	mg/ L	1600	1632	1616
Ca	mg/ L	600	620	610
Mg	mg/ L	388	395	391.5

3.2 Characterization of Fe_3O_4 Nanoparticles

3.2.1 XRD Analysis

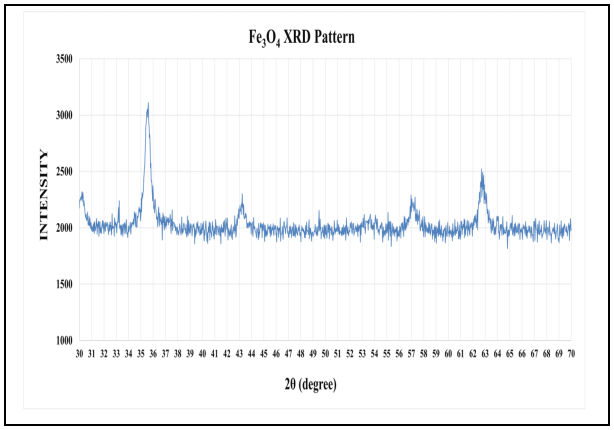


Fig. 4: X-ray diffractogram

Different pH values were used to investigate the influence of different pH on the nanoparticles, and the XRD patterns. The strong peaks at 2θ = 20.08 of the curve were attributed to the presence of chitosan. The characteristic XRD peaks at 2θ 30.05, 35.60, 43.02, 57.03, 62.70 indicated that the nanoparticles were

magnetite with an inverse spinel structure. The Fe_3O_4 existed in ion form when the pH was lower than 2.6. In contrast, the predominant form of Fe_3O_4 was colloidal at pH 3.0 and 3.5, which greatly affects the crystallinity of the nanoparticles obtained. The results of XRD indicated that the feasible pH for the synthesis should be below 2.0 (Baiju *et al.* 2018).

3.2.2 FTIR Analysis

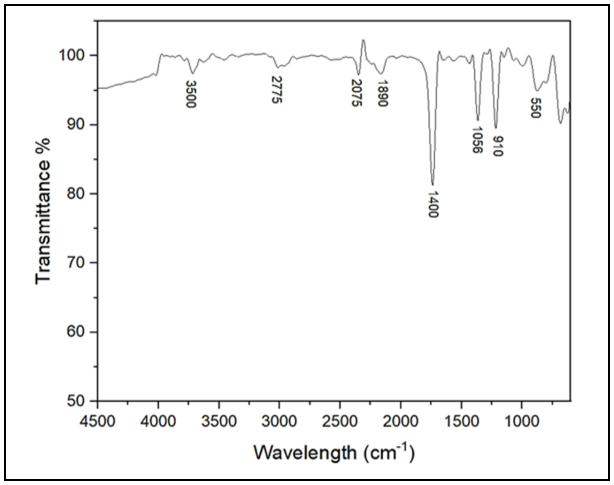


Fig. 5: FTIR Spectrum

The functional groups present in the Fe_3O_4 nanoparticles were monitored by FT-IR spectroscopy (Fig. 5). The peak near 3500 cm^{-1} correspond to the stretching vibration of $–NH_2$/–OH group, and the peaks at 2775, 2075, 1890 and 1400 cm^{-1} represented the presence of –CH and $–CH_2$ group. The characteristic band at 1056 cm^{-1} represented the presence of the –C–O– group of chitosan. The disappearance of the "free" aminogroup absorption band in sample is due to chelation of the amino groups with the magnetite nanoparticles. Thus, the chitosan coating over the magnetite nanoparticles *via* the amino group was confirmed (Baiju *et al.* 2018).

3.2.3 SEM Morphology

The pH values of the solution strongly affect the morphologies of Fe_3O_4 nanoparticles. When pH values were lesser than 2.0, the nanoparticles had diameters smaller than 0.15 µm, an oval shape, and were uniformly dispersed in the chitosan hydrogel without aggregation. The average size of Fe_3O_4 nanoparticles from SEM (Fig. 6) was larger than the result from the Scherrer formula (11 nm), which was attributed to the fact that the Scherrer formula only provides the lower limit on mean crystallite size. The diameter of the Fe_3O_4 nanoparticles indicated that the Fe_3O_4 nanoparticles are superparamagnetic. It was confirmed that the nanoparticles were Fe_3O_4 with an inverse spinel structure (Baiju *et al.* 2018).

3.3 EF Results

3.3.1 Effect of Catalyst Dosage

In the heterogeneous E-Fenton process, the effect of catalyst dosage was studied by varying dosages from 25 mg/L to 100 mg/L. The other operating parameters were maintained constant (initial pH 3, voltage 5 V, electrode spacing 3 cm). The COD removal increases when the catalyst dosage increases till it reaches saturation point after which the removal efficiency decreases. Excessive catalyst dosages lead to the surplus Fe^{2+} acting as a hydroxyl radical scavenger, subsequently oxidizing to Fe^{3+}. This process diminishes the availability of hydroxyl radicals for the oxidation of pollutants, resulting in reduced COD removal efficiency. Conversely, lower catalyst dosages inadequately produce Fe^{2+}, limiting the hydroxyl radical generation and consequently lowering COD removal efficiency (Oturan *et al.* 2001). The optimum catalyst dosage for electro-Fenton process was found to be 50 mg/L at 90 minutes (Fig. 7).

3.3.2 Effect of pH

To investigate the effect of pH on E-Fenton process, studies were performed by varying pH from 1.5 to 5. The other parameters kept constant were catalyst dosage 50 mg/L, voltage 5 V and electrode spacing of 3 cm. The optimum pH for heterogeneous E-Fenton process was found to be 2 with a removal efficiency of 84% and the plotted graph is given in Fig. 8. As the pH was increased from 2 to 5, removal efficiency decreased from 84% to 53%. This may be due to the iron complexes formed at high pH (Nidheesh *et al.* 2014). Also, the advantage of employing the Fenton process at a low pH, specifically between 2 and 3, lies in its effectiveness for eliminating inorganic carbons from wastewater. At this acidic pH range, the Fenton process exhibits a high removal efficiency, with the optimal outcome observed at pH 2. This underscores the significance of maintaining a low pH to enhance the performance of the electro-Fenton process in wastewater treatment (Zhang *et al.* 2005).

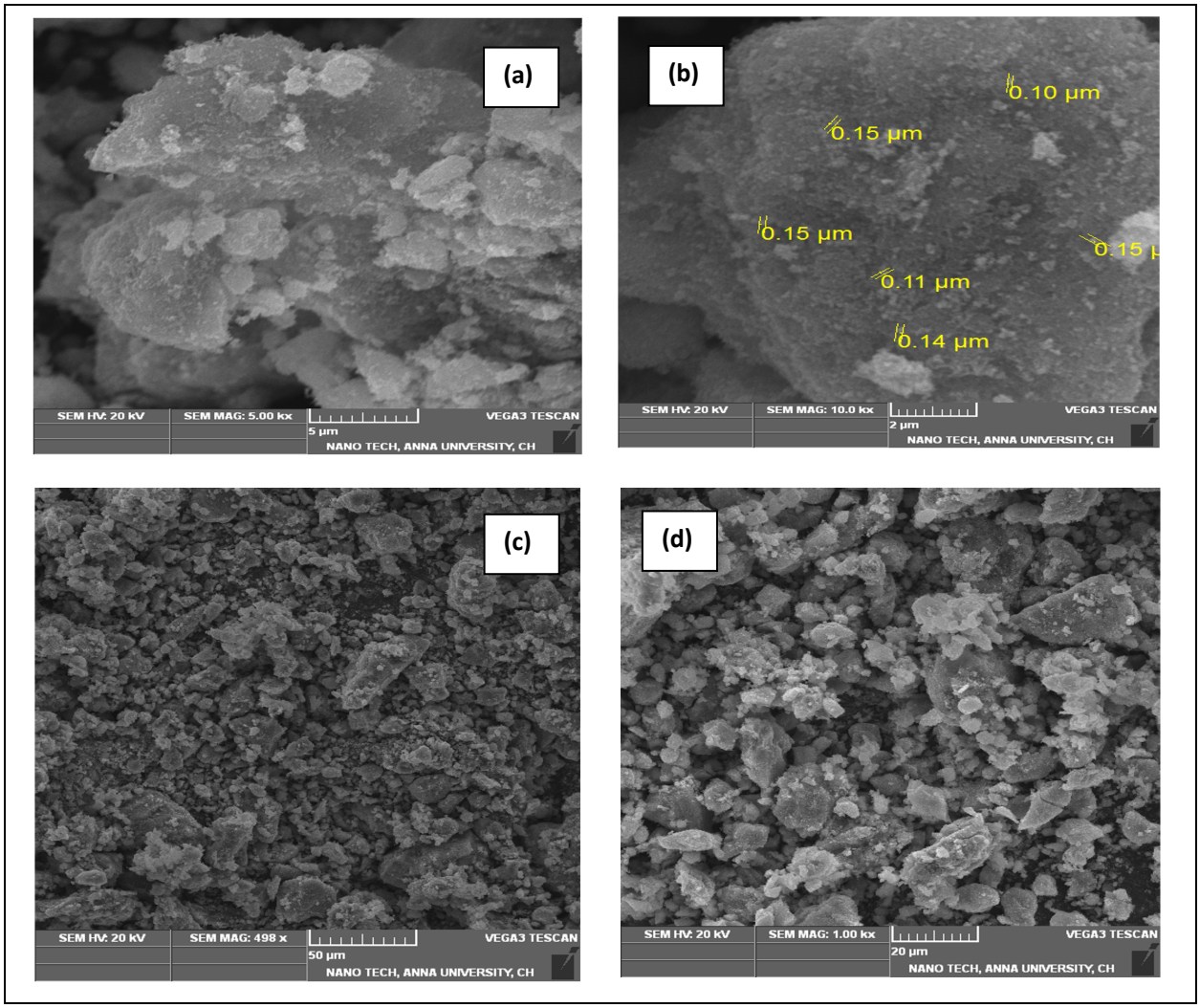


Fig. 6: SEM image of FeMo Nanoparticles

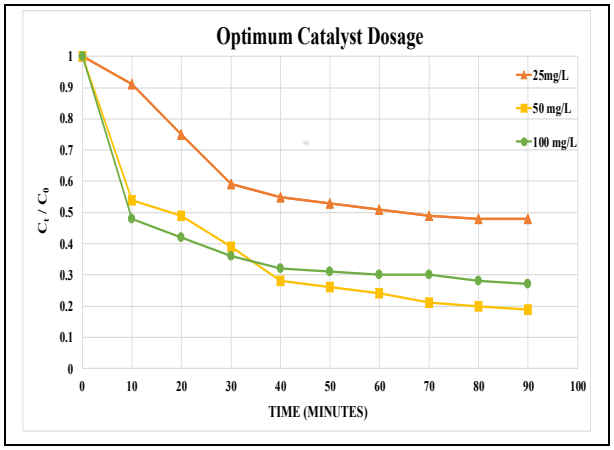


Fig. 7: Optimum catalyst dosage chart

3.3.3 Effect of Electrode Spacing

The effect of electrode spacing in heterogeneous E-Fenton process was studied by varying the electrode spacing from 2 cm to 4 cm. The other operating parameters that were kept constant are catalyst dosage 50 mg/L, pH 2 and voltage 5 V. When the electrode spacing was increased to 4 cm, it resulted in a longer path for the electric current to travel, leading to increased ohmic drop. This higher resistance hinders the electrochemical reactions, particularly affecting the mass transfer of Fe^{3+}, which is essential for in removing COD. When the electrode spacing is reduced to 2 cm, the efficiency further dropped due to the faster electro-generation of Fe^{2+} to Fe^{3+} at the anode. The optimum electrode spacing for heterogeneous E-Fenton process was found to be 3 cm.

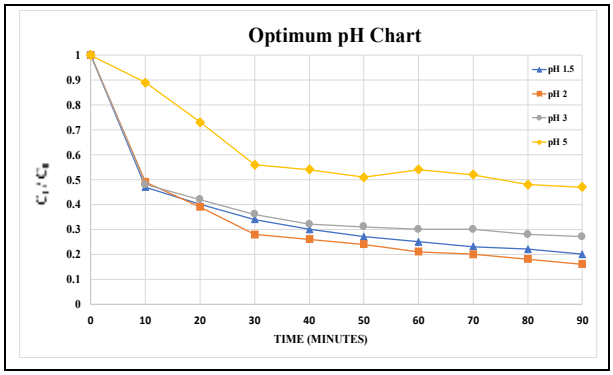


Fig. 8: Optimum pH dosage chart

Leachate characterization after E-Fenton treatment was done as per standard methods and the obtained results are given in Table 2. The characterisation was done for the effluent which was treated at optimum conditions (catalyst dosage of 50 mg/L, pH of 2 and electrode spacing of 3 cm). The effluent had an average pH of 1.985 and a BOD/COD ratio of 0.5155, indicating a significant improvement in leachate quality, as the BOD/COD ratio increased from 0.169 to 0.515. The COD of the electro-Fenton (E-Fenton) treated leachate was 1090.5 mg/L, corresponding to a removal efficiency of 84.29%. It was observed that TSS, Cl, Ca, Mg values were also reduced after the E-Fenton treatment. The EC, TDS values increased after the treatment. There was a significant improvement in colour of the treated leachate.

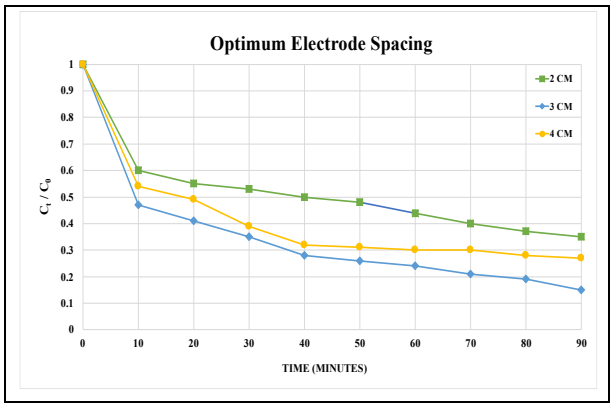


Fig. 9: Optimum electrode spacing chart

Table 2. Characteristics of E-Fenton treated landfill leachate

Experiment	Unit	Effluent		Average
pH	-	1.98	1.99	1.985
EC	µS/cm	41097	41918	41507.5
BOD5	mg/L	557	568	562.5
COD	mg/L	1069	1112	1090.5
BOD5/COD	-	0.521	0.510	0.5155
TDS	mg/L	49315	50301	49808
TSS	mg/L	435	440	437.5
TS	mg/L	49750	50741	50245.5
Cl	mg/L	1584	1617	1600.5
Ca	mg/L	589	602	595.5
Mg	mg/L	382	399	390.5

3.4 CW Results

The treated effluent obtained from the Electro Fenton process underwent mixing with activated sludge and wastewater from the Anna University Sewage Treatment Plant, Chennai in 1:1:1 ratio. This resultant mixture was employed as the influent for the experimental setup which helps in inoculating the wetland setup and increases the microbial activity. The experiments were conducted in a batch process, with two trials performed to assess the consistency of the treatment process. To maintain a balance between treatment efficiency and evaporation loss, the Hydraulic Retention Time (HRT) was carefully chosen as 3 days with working volume as 3 L. The eggshells had high adsorptive capacity to inorganic compounds, which can be attributed to their expansive surface area, featuring numerous pores ranging between 7,000 and 17,000. This inherent high porosity and natural composition helps in the utilization of eggshells in their natural state without the need for surface activation process (Zonato *et al.* 2022). Leachate characterization after CW treatment was done as per standard methods and the obtained results are given in Table 3. The effluent had an average pH of 7.315 and BOD/COD value of 0.645, and good amount of improvement in the quality of leachate was observed, as the BOD/COD value increased from 0.169 to 0.645. The COD of CW treated leachate was 292 mg/L corresponding to a removal efficiency of 95.79%. It was observed that TSS, Cl, Ca, Mg values were also reduced after the CW treatment. The EC, TDS values increased after the treatment. While comparing CW1, all parameters were improved in CW2, except Ca and Mg concentrations. This may be due to the presence of eggshell in the intermediate layer of CW2. Eggshells, rich in calcium carbonate, serve as excellent adsorbent materials for the remediation of contaminated water. Consequently, the utilization of eggshells in treatment processes leads to an elevation in the concentration of calcium in the effluent (Lee *et al.* 2022). There is a significant improvement in colour of the treated leachate in both systems. Comparing the CW-2 with control system CW-1, it was found that CW-2 gave better results than CW-1 except for few parameters. Hence, using eggshell as a wetland substrate is not only economical but also an efficient alternative in treating leachate.

Table 3. Characteristics of Constructed Wetland treated landfill leachate

Experiment	Unit	Effluent					
		Sand+ Gravel+ Gravel		Average	Sand+ Eggshell+ Gravel		Average
pH	-	7.15	6.9	7.025	7.4	7.23	7.315
EC	µS/cm	22648	23100	22874	20426	20822	20624
BOD5	mg/L	209	213	211	187	190	188.5
COD	mg/L	331	337	334	293	291	292
BOD5/COD	-	0.631	0.632	0.6315	0.638	0.652	0.645
TDS	mg/L	22191	22600	22395	19357	19744	19550
TSS	mg/L	174	182	178	153	158	155.5
TS	mg/L	22365	22782	22573.5	19510	19902	19706
Cl	mg/L	633	649	641	596	611	603.5
Ca	mg/L	177	181	179	209	215	212
Mg	mg/L	116	123	119.5	143	147	145

4. CONCLUSION

In the present study, heterogeneous E-Fenton process was used in treating for stabilized landfill leachate using Fe_3O_4 nanoparticles. The Fe_3O_4 nanoparticles synthesized *via* the assembly of iron ions with chitosan were used as a catalyst. The results indicated that COD was reduced from 6945 mg/L to 1090.5 mg/L with removal efficiency of 84.29%. COD removal was achieved at optimal conditions of pH 2, catalyst dosage 50 mg/L and electrode spacing of 3 cm. After the Electro Fenton process, an enhancement in the biodegradability was observed from the BOD5/COD ratio when it increased from 0.03 to 0.51. Subsequent biological treatment using constructed wetland using eggshell as the substrate further reduced the COD to 292 mg/L and the BOD5/COD ratio increased from 0.51 to 0.645. This also proved that using eggshells as a media is a reliable alternative compared to regular sand media but the Ca and Mg concentrations in final effluent treated by CW-2 was more compared to control system CW-1. Combined E-Fenton and biological treatment (CW) achieved an overall COD removal of 95.79% and increased the biodegradability effectively proving this

integration to be an efficient treatment for stabilized landfill leachate.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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