



Electrochemical treatment of biodigester effluent of maize-based starch industry: COD and color removal

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ABSTRACT

Electrochemical (EC) treatment of biodigester effluent (BDE) of maize (corn)-based starch industry wastewater was investigated. Four aluminum plate electrodes were employed in a 1.5 dm³ electrolytic batch reactor. Experiments were performed in the pH range of 4–10 using current density (CD) in the range of 51–255 A/m² (1–5 A). The distance between each of the aluminum electrodes was fixed at 15 mm. At pH 9 and at a CD of 153 A/m² for an operation time of 100 min, maximum chemical oxygen demand reduction of 84.13% and color reduction of 94.89% were achieved using the EC process. The power (energy) requirement and electrode loss were calculated at varying pH. The settling and filterability characteristics of electro-coagulated sludge were studied at different pH. It was demonstrated that EC treatment is an effective treatment method to treat BDE from maize-based starch industry.

Keywords: Maize-based starch industry; Biodigester effluent; Electrochemical treatment; Aluminum electrode; Filtration; Chemical oxygen demand

1. Introduction

Starch products have manifold applications in the industries for which maize (corn) serves as one of the major raw material in the manufacturing process. Processing of maize gives many value-added products which are used for various purposes in our society. Among the various industrial applications of starch, it is mainly used as a surface sizing, binding, and coating agent in the paper and the textile industry. In integrated maize-based starch industry, each part of maize provides various valuable products like starch, glucose, maize oil, cattle, and poultry feeds. Starch production from maize conventionally employs the

wet milling process. The water consumption in the production process per ton of maize has been reported to be around 8 m³, which in turn generates 5.5 m³ of wastewater [1]. The wastewater obtained during the process has a high organic load in terms of chemical oxygen demand (COD) and biochemical oxygen demand (BOD). As the effluent contains a high BOD value, it is treated initially anaerobically to recover energy in the form of methane gas [2].

Conventionally in the maize industries, composite effluent (pH 5–6) that comes out of the integrated starch industry is not sent directly for anaerobic digestion. It is mixed with some effluent that comes out of bio-methanation reactor (pH 7.5–8), to maintain a pH 7 in the wastewater stream being sent to bio-methanation (biodigestion) reactor. The treated

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effluent which comes out from an anaerobic biodigester is called biodigester effluent (BDE), which is fed to aerobic lagoon for further treatment. In this lagoon, submerged air bubbler or surface aerator is used to supply oxygen during the treatment. This process however has a high operating cost necessitating for the need of an improvised process. The discharge of aerobic lagoon contains COD in the range of 800–1,200 mg/dm³, which is far from the discharge water quality standards for release into surface water (COD < 100 mg/dm³) and sewers (COD < 300 mg/dm³) applicable in India as per the World Health Organization norms [3]. The black color of effluent harms the aquatic life by reducing air diffusion and sunlight absorption. Dark color of effluent also hinders the natural process of photochemical reactions necessary for self purification of the surface water. Therefore, the decolorization and the removal of COD from the BDE are of immense importance from the environmental point of view.

Various methods have been reported in literature to treat organic wastewater; however for treatment of starch industry wastewater not much has been reported. The anaerobic treatment has been reported by some authors [4–6]. Duran-deBazua et al. [7] evaluated a two-stage system consisting of anaerobic treatment followed by aerobic process. Ozturk et al. reported COD removal efficiencies of 89% in anaerobic process and 85% removal in the subsequent aerobic process [8]. A membrane-assisted process (aerobic digestion ultra filtration) has been also reported to treat effluent from maize industry [9]. This process gave good results, however the equipment and operational cost are high. Since the biological process reduces only the biodegradable compound, therefore in recent years some physicochemical processes such as coagulation [2,10,11], electrochemical (EC) [12,13], thermal treatment [14,15], and wet oxidation [16,17] have been explored. Above-mentioned methods gave significant results for the treatment of wastewater from various industries containing organic waste.

Among the various processes listed above, EC process is one of the most effective one and it has been successfully applied for the treatment of a wide variety of effluents from pulp and paper mill, olive oil mill, tannery, restaurant, distillery, textile, and colored water, etc. Compared to the traditional flocculation and coagulation process, EC process has advantages of removing small colloidal particles. During the EC process when current is passed through a DC source, smaller colloidal particles are set into motion increasing the probability of their coagulation. Addition of excessive amount of coagulants can be avoided due to their *in situ* generation by electro-oxidation of a sacrificial

anode. EC equipment is simple and easy to operate. Short reaction time and low sludge production are the other advantages of this process [12].

In the present work, usefulness of EC process for the treatment of BDE coming out of an integrated maize-based starch industry has been investigated. Investigation of COD and color reduction in a batch EC reactor using aluminum electrodes with respect to the parameters, such as current density (CD), initial pH (pH_i), and electrolysis time has been reported. The process generates sludge that necessitates a need of post-treatment by separation; hence its separation by settling and filtration was also studied. Fig. 1 shows the steps involved during EC treatment studies in the present investigation.

2. Material and methods

2.1. Material

The analytical grade chemicals of Merck India Limited, Mumbai were used during the experiments. Aluminum plate was arranged from local market. BDE used in the study was obtained from Raja Ram Maize Industry Pvt. Ltd. located in Rajnandgaon, Chhattisgarh, India. The typical composition of the BDE and treated BDE is presented in Table 1. The details of reactor used for the EC process are given in Table 2.

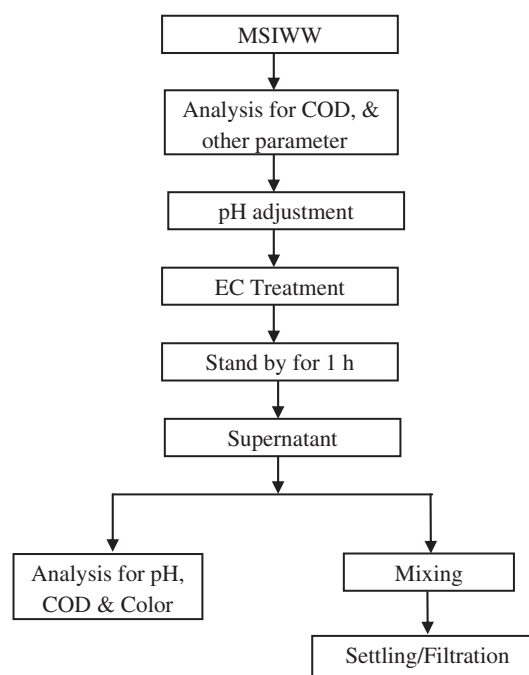


Fig. 1. Flow chart showing the steps for experiments.

2.2. Analytical procedure

The COD of the sample was determined by the close reflux method. The samples were digested at 148 °C in the digester (Merck make), then absorbance was noted at 605 nm using UV–spectrophotometer (Thermo Electron Corporation make, England) and chloride was determined by standard titrimetric methods [18]. Sulfate and phosphate were determined by following standard methods [19]. Protein was estimated by Lowry method [20]. The pH of the sample was determined using a digital pH meter (EI make, India). The color of the samples was estimated in terms of platinum cobalt unit (PCU) by measuring absorbance at $\lambda = 460$ nm using UV–spectrophotometer [19].

2.3. Experimental setup and procedure

The lab-scale batch experimental setup used for the EC degradation studies is shown schematically in Fig. 2. Experiments were carried out in a 1.5 dm³ reactor made up of Pyrex sheet. Four Aluminum plates of dimension (70 × 70 × 3 mm; length × breadth × thickness) were used in the reactor as electrodes. The total effective surface area of each electrode was 4,900 mm² and 10 mm gap was maintained between the bottom of the electrodes and the bottom of the cell for the movement of magnetic stirrer. The spacing between two electrodes was kept 15 mm. The plates when connected in series have higher resistance. In parallel arrangement, the electric current is divided between all electrodes in relation to the resistance of the individual cell. In order to reduce the resistance, the electrodes were connected in parallel. The electrode plates were cleaned manually by abrasion with sand paper, and treated with HCl solution (10%) followed by washing with distilled water prior to their use.

The anode and cathode leads were connected to the respective terminals of a direct current (DC)

Table 2
Characteristics of the EC cell

<i>Electrodes</i>	
Materials (anode and cathode)	Aluminum
Shape	Rectangular plate
Size	70 × 70 mm
Thickness (mm)	3
Plate arrangement	Parallel
Effective electrode surface area	4,900 mm ²
<i>Reactor characteristics</i>	
Make	Perspex glass
Reactor type	Batch mode
Dimensions (cm)	10.7 × 10.7 × 13.7
Volume (dm ³)	1.5
Electrode gap (mm)	15
Stirring mechanism	Magnetic bar
Power supply	Direct current (DC)
Current range (A)	0–5
Voltage range (V)	0–30

source fitted with an ammeter and voltmeter. All test runs were performed at a room temperature (30 ± 2 °C). A constant CD was maintained using digital DC supply (0–30 V, 0–5A). At the end of the experiments, the sample was filtered using Whatman filter paper 42 to remove sludge. The filtered liquid was used for color and COD analysis. In all test runs, 1.4 dm³ of BDE was taken into the EC reactor. Experiments were conducted for four plate configuration at various CDs of 51, 102, 153, 204, and 255 A/m² and voltage ranging between 0 and 30 V.

3. Results and discussion

The dissolution of metal plates (Al) takes place in the wastewater as DC current is passed through the electrodes. These metal ions form a wide range of species at different pH. The Al³⁺ also forms species Al(OH)₂⁺, Al(OH)₂²⁺, Al₂(OH)₂²⁺, Al₂(OH)₄⁴⁺, Al(OH)₄⁻, Al

Table 1
Typical composition of BDE-MSIWW before and after EC treatment. CD = 153 A/m²

Parameters	BDE	pH 4	pH 5	pH 6	pH 7	pH 8	pH 9	pH 10
COD (mg/dm ³)	1920	676	511	453	399	352	304	466.5
Color	Brown	Light yellow	Yellow	Yellow	Light yellow	Light yellow	Light yellow	Greenish yellow
TDS (mg/dm ³)	20,980	6,322	5,722	5,534	5,025	4,502	4,804	7,228
Protein (mg/dm ³)	310	21	12	20	18	19	18	22
Total hardness (mg/dm ³)	12,650	11,250	11,000	12,000	10,050	11,550	9,800	10,200
Cl ⁻ (mg/dm ³)	1826	1,650	1,610	1,580	1,510	1,470	1,420	1,560
pH	7.8	7.38	7.79	7.37	7.35	7.4	7.42	7.45
PO ₄ ³⁻ (mg/dm ³)	290	40	45	112	62	30	48	46
Absorbance ^a (at $\lambda = 460$ nM)	0.12	0.028	0.020	0.017	0.015	0.013	0.006	0.016
Color (PCU)	2,300	538.43	397.5	332.5	294.86	245.41	117.5	301.76

^aAbsorbance for color reduction was determined after 10 times dilution.

Table 3
Filterability of the slurry

Initial pH	$k_c \times 10^{-12} \text{ s/m}^6$	$\beta \times 10^{-6} \text{ s/m}^3$	$c \text{ kg/m}^3$	$\alpha \times 10^{-12} \text{ m/kg}$	$R_m \times 10^{-6} \text{ m}^{-1}$
4	0.0222	6.27	11.164	8.04	3.99
5	0.0316	5.92	17.726	7.20	3.64
6	0.3350	5.57	11.326	119.54	3.54
7.8	0.1410	4.16	6.40	89.0	2.65
9	0.1280	39.64	6.89	75.10	25.20
10	0.0149	5.2	5.28	11.40	3.31

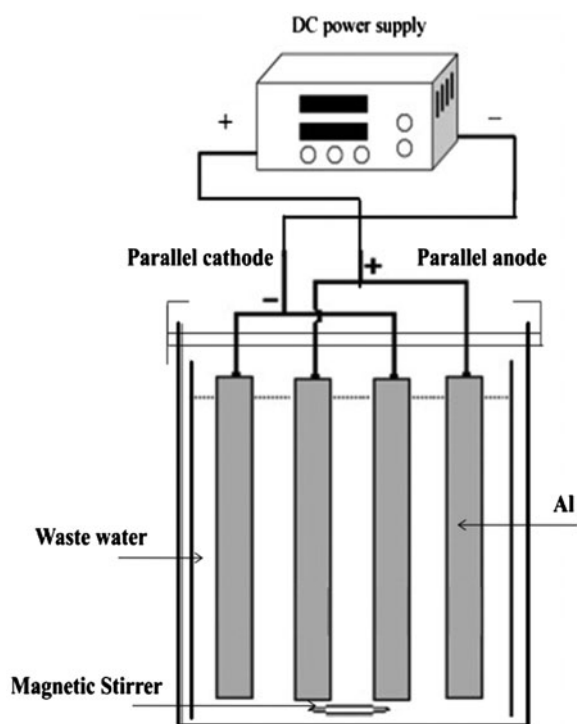


Fig. 2. Schematic diagram of the experimental setup of the EC cell.

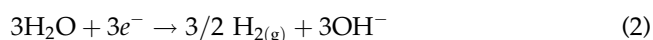
$(\text{H}_2\text{O})_5\text{OH}^{2+}$, $\text{AlO}(\text{OH})$, etc. The hydroxide components neutralize the colloids having negative charge, which make large flocs and can be removed by sedimentation [21].

The most widely used electrode material in EC process is aluminum and iron. In the case of aluminum, following reactions take place as shown in Eqs. (1) and (2) [22].

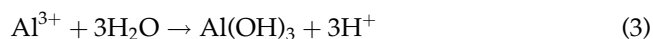
At anode:



At cathode:



Al^{3+} and OH^- ions generated by electrode reactions as per Eqs. (1) and (2), react to form various monomeric species which finally transform into $\text{Al}(\text{OH})_3$ in accordance to complex precipitation kinetics as shown in Eq. (3).



Freshly formed $\text{Al}(\text{OH})_3$ “sweep flocs” have large surface areas, which are beneficial for a rapid adsorption of soluble organic compounds. Finally, these flocs are removed easily from aqueous medium by sedimentation [22].

3.1. Effect of pH on COD and color removal

It has been established that the pH is an important parameter influencing the performance of the EC process [23]. The studies were conducted in the pH range of 4–10. To examine the effect of pH, the BDE solution was adjusted to the desired pH by adding sodium hydroxide (1 M) or sulfuric acid (1 M) solution. Fig. 3(a) shows the effect of COD reduction of BDE with respect to time at various pH values. It can be seen that at different pH, the COD decreases rapidly for the first 20 min, later on the rate of COD removal becomes slow. The maximum COD reduction was achieved at pH 9. It was observed that the COD reduction in 20 min was 57.06% increasing up to 84.13% for 100 min of operation. The COD removal efficiency depends on the formation of Al^{3+} metal ions and oxyhydroxyl aluminum species which varies with pH. It also depends on the nature of organic and inorganic components present in the effluent [22].

Color was also effectively reduced during ECT as shown in Fig. 3(b). The color reduction is expressed as percentage decrease in the absorbance of the BDE sample with respect to the untreated sample at a

wavelength (λ) = 460 nm. The maximum reduction in color was observed at pH 9. In the initial 20 min of EC treatment, color reduction was found to be 66.33% which further increased to 94.89% in 100 min of operation. It was observed that the color reduction followed the pattern of the COD reduction in the pH_i range of 4–10.

The electrical energy consumption was calculated using the Eq. (4) [22].

$$P \left(\frac{\text{Wh}}{\text{dm}^3} \right) = \frac{V_c I t}{\text{Treated volume (dm}^3)} \quad (4)$$

where V_c is voltage of the cell and I is current intensity. Energy consumption (P) as a function of pH, at a CD (j) of 153 A/m² and time (t) of 100 min is shown in Fig. 3(c). At pH 4, 5, 6, 7, 8, 9, and 10, respectively, the energy consumption was calculated as 16.11, 35.4, 13.59, 20.11, 26.91, 30.33, and 27.96 Wh/dm³ of BDE. At pH 6, the energy consumption was low. This may be due to the adherence of some flocs to the surface of aluminum electrode acting as insulator, as compared to the other pH.

The EL at different pH was also calculated and a graph between EL and pH is shown in Fig. 3(d). At pH 4, 5, 6, 7, 8, 9, and 10, respectively, electrode losses were 62.74, 53.22, 42.56, 39.185, 45.03, 49.08, and 52.395 mg/dm³ of BDE, respectively. It was observed that the electrode consumption increases as we move towards more acidic or basic environment. Maximum COD reduction (84.13%) was obtained at pH 9 with an electrode loss of 49.08 mg/dm³ of BDE and energy consumption of 30.33 Wh/dm³ of BDE. The reason for high EL at high pH (basic) may be due to the high presence of OH⁻ ions which lead to the formation of Al(OH)₃. At acidic pH, H⁺ ions are liberated and more Al³⁺ cations are released in the wastewater [22].

3.2. Effect of applied current density

The CD (j), being a surrogate parameter, was found to influence the treatment efficiency of the EC process [24,25]. Therefore, the applied CD was varied (51–255 A/m²) to investigate its effect on COD and color removal for a four plate configuration. It is well

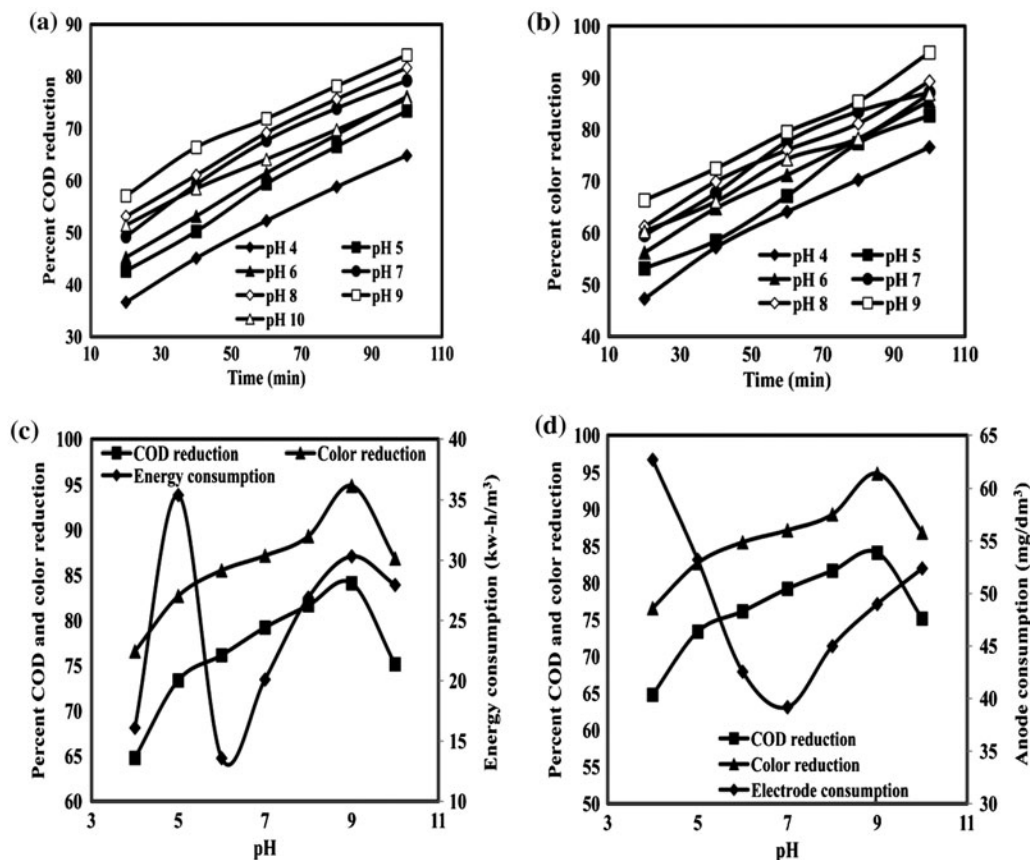


Fig. 3. Effect of pH_i on (a) COD reduction, (b) color reduction, (c) energy consumption, and (d) electrode loss (COD_i = 1940 mg/dm³, CD = 153 A/m²).

known that electrocoagulant production is a function of applied CD and hence affects the growth of flocs [23].

In the present study of COD and color reduction by EC process, the treatment at pH 9 gave good results as compared to the treatment at other pH_i. Thus at pH 9 CD was varied to find its effect on treatment of BDE-MSIWW. The results obtained are shown in Fig. 4(a). A COD reduction of 74.28, 79.15, 84.13, 85.67, and 86.31% and color reduction of 84.13, 89.48, 94.89, 95.85, and 96.5%, was achieved at a CD of 51, 102, 153, 204, and 255 A/m², respectively, in 100 min of operation. The removal of COD and color increases with an increase in the CD. Significant COD and color removal was obtained in 20 min of treatment at all current densities. Further operation of the process for 100 min resulted in a comparatively lower percentage reduction in COD and color. From Fig. 4(a), it can be observed that at pH 9 and CD = 255 A/m², COD reduction and color reduction is maximum. The increase in CD from 153 A/m² does not give an appreciable percentage reduction in COD and color, but it increases the cost of operation. The reason for removal

of organics with an increase in the value of CD may be attributed to the formation of large bubble at high CD which results in quicker flocculation of the particles, causing better COD removal efficiency. Small bubbles provide more surface area for the agglomeration of particles and the formation of dense and larger flock in the aqueous stream, resulting in a better separation. As the applied current *j* increases, the bubble production rate increases. However, these bubbles are smaller in their size, causing the higher upward flux and resulting in enhanced pollutant degradation and sludge floatation. Hence at higher CD better COD and color removal efficiencies are achieved [22].

3.3. Effect of electrolysis time on COD and color removal

Reaction time also influences the treatment efficiency of the EC process. The relationship of the COD and color removal to electrolysis time is shown in the Figs. 3 and 4. The COD and color removal efficiency depend on the concentration of metal ions produced in the wastewater resulting from the dissolution of the electrodes, up to certain concentrations of Al³⁺. Longer time of EC operation results in the higher concentration of Al³⁺ ions. After a certain limit of Al³⁺ concentration, the decrease in COD reduction is due to stabilization of neutralizing organic anions [26]. Also the rate of removal of COD decreases as EC time increases. Maximum COD removal occurs in the first 40 min. More electrolysis time will be an expensive proposition with respect to further COD and color removal.

The treatment is considered to be complete when the gap between the electrodes becomes visible. This is also the condition when floatation/settling are almost complete after oxidation and flocculation.

3.4. Change in initial pH with electrolysis time

The change in pH with processing time has been plotted and shown in Fig. 5. For different values of initial pH ranging from 4–10 at constant CD 153 A/m², it was found that the pH of the solution increases sharply at pH_i 4, 5, and 6 during the initial time period of 20 min. The pH of the wastewater in the reactor becomes nearly constant after 60 min of operation except at pH 10. While at pH 8, 9, and 10, it decreased during the initial time period of 20 min. This characteristic of pH change during the EC process is quite useful in its application to treat maize-based industrial wastewater. The pH_i was also found to affect the heat evolution during electrolysis [23].

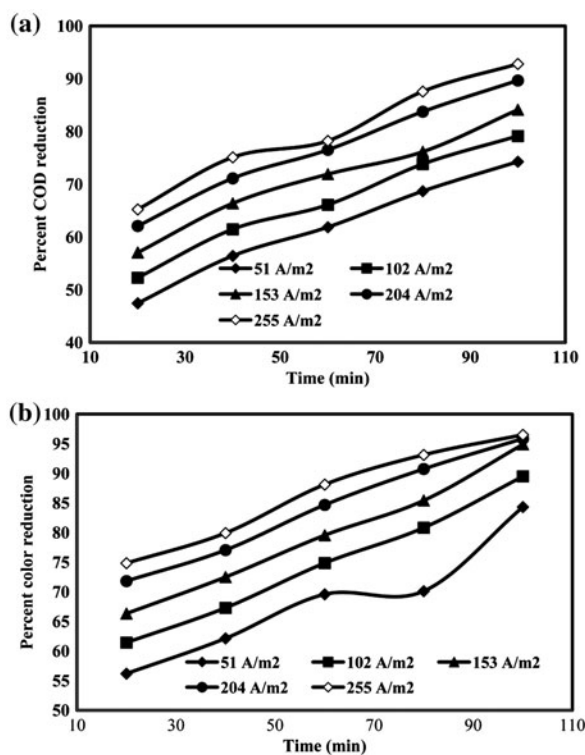


Fig. 4. Effect of CD on (a) COD reduction and (b) color reduction (COD_i = 1920 mg/dm³, 2,100 PCU).

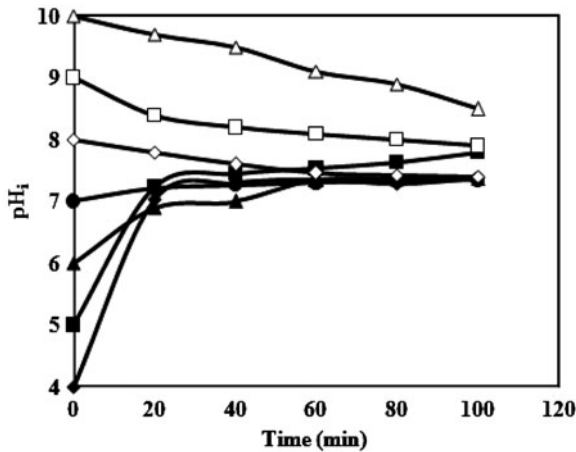


Fig. 5. Change of pH_i with time during ECT.

3.5. Settling studies

The slurry obtained from the EC treatment was subjected to a jar test to investigate sedimentation characteristics. In the sludge settling test, a clear solid/liquid interface was observed. The dimensionless height of the solid/liquid interface (H/H_0) vs. the settling time as a function of different pH_i values, ranging between pH values of 4 and 10 has been demonstrated in Fig. 6. At the beginning of the sludge settling for a very short period, a relatively slow sludge settling occurs due to the primary Brownian motion of the particles. This is followed by the steady state decrease in the height of the solid/liquid interface, exhibiting the regime of zone settling. Thereafter, the transition settling period is observed. Finally, the compression settling takes place, which has a steady state and minimal rate of decrease in height of the solid/liquid interface. The settling characteristic of treated BDE of MSIWW at pH 9 and 10 was found to

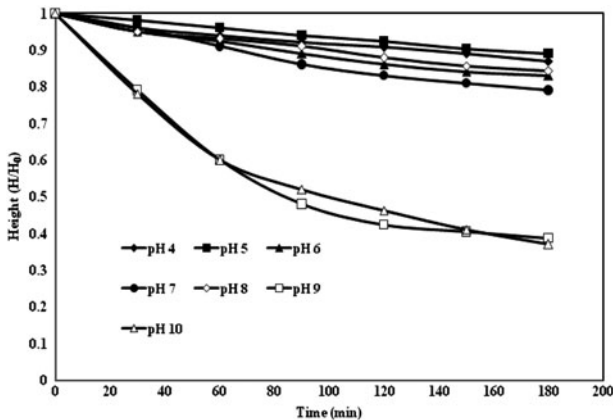


Fig. 6. Settling curve of slurry obtained from the ECD.

be better than the trend observed at other pH values. In 40 min, 60% settling occurs at pH 9 and it was 45% at pH 10. It may be due to presence of various functional group which at basic pH aid in bridging between particles, enabling the formation of heavy flocs. Hence, sedimentability is better at higher pH.

3.6. Filtration study

To separate the supernatant and solids of slurry obtained after the ECT of the BDE, the filtration experiment was also performed on Whatman 42 filter paper. The filter paper was supported on a ceramic Buckner funnel. Filtration resistances for the filter media as well as filter cake were obtained using the Eqs. (5),(6) and (7) [27].

$$\frac{dt}{dv} = k_p V + \beta \tag{5}$$

$$k_p = \frac{C\alpha\mu}{A^2(-\Delta p)} \tag{7}$$

and

$$\beta = \frac{\mu R_m}{A(-\Delta p)} \tag{7}$$

where t is the time taken for filtration (s), V volume of filtrate collected in time (m^3), k_p is the slope of the plot using Eq. (5) (s/m^6), β the intercept for the plot of the graph plotted using Eq. (5) (s/m^3), C the concentration of sludge (kg/m^3), α specific cake resistance (m/kg), μ viscosity of the filtrate (Pa s), A area of the filter media (m^2), Δp pressure drop across the filter (Pa), and R_m filter medium resistance (m^{-1}).

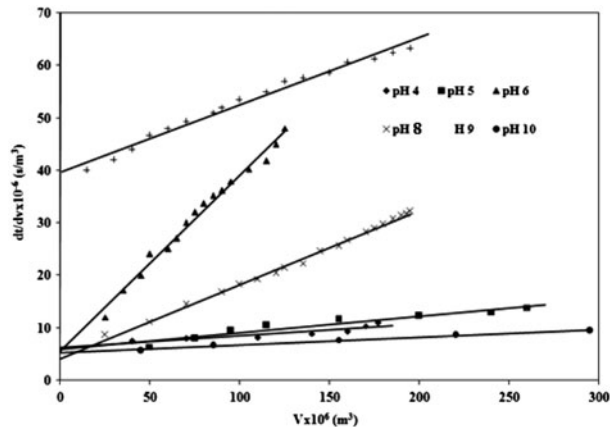


Fig. 7. Effect of pH_i on the filterability of treated BDE-MSIWW.

Table 4
Analysis of the residue obtained after the EC process. $t_R = 100$ min, $CD = 153$ A/m²

Initial pH (pH _i)	4	5	6	7	8	9	10
Final pH (pH _f)	7.38	7.79	7.37	7.35	7.4	7.42	7.45
Wt. of residue (g/dm ³)	11.164	17.726	11.326	9.89	6.40	6.89	5.28
Color	Dark brown	Brown	Light brown	Light brown	Light brown	Light gray	Gray
Nature	Lumpy mass, very hard	Lumpy mass, hard	Lumpy mass, hard	Lumpy mass, hard	Lumpy mass, hard	Lumpy mass, hard	Small particles, soft
% Convertible COD	64.81	73.38	76.16	79.23	81.68	84.13	75.7

A plot of dt/dv vs. V with pH as the control parameter is presented in Fig. 7. From the plot, the values of slope (k_c) and intercept (β) have been calculated. The α and R_m are determined from k_c and β , and values are presented in Table 4. From Table 4, it may be seen that EC treatment at highly basic (pH 10) and highly acidic (pH 4, 5) condition gives good filterability as compared to the treatment at other pH (pH 6–9). The value of α lies in between 7.2 and 119.54×10^{12} m/kg. It was lowest at pH 5 and highest at pH 6. The values of specific cake resistances for different sludges have been given by Barnes et al. [28]. These values are $4\text{--}12 \times 10^{13}$ m/kg for activated sludge and $3\text{--}30 \times 10^{13}$ m/kg for digested sludge. Thus, the specific cake resistance of EC-treated BDE showed better filterability than activated sludge and digested sludge except at pH 6. The values of the filter media resistance R_m were found to be lower (2.65×10^6 m⁻¹) at pH 7.8 and highest at pH 9 (25.2×10^6 m⁻¹). The importance of filter media is observed only at the initial stages of filtration, however at later stages cake resistance plays a significant role. Filterability will be high, when cake resistance value is low.

3.7. Analysis of filtrate and residues

The filtrates were analyzed for various parameters. The results are shown in Table 1. From Table 1, it may be seen that appreciable amount of COD, color, TDS, protein, and phosphorous have been removed in ECT. Total hardness reductions are comparatively lower. Final pH of EC-treated BDE has increased significantly except at pH 9 and 10. Color of residues was found to be different at different pH as presented in Table 4. The residue was made up of small particles with easily grindable lumpy mass and hard small particles. The calorific values were not determined, but some authors have reported moderate calorific values of residues obtained by separation of dissolved organics by various processes as thermolysis, ECT, and coagulation [2,15,29].

4. Conclusions

The present study demonstrated the applicability of EC treatment for total solids, COD, and color reduction of BDE-MSIWW. Aluminum electrode-based EC treatment of BDE-MSIWW is a promising application for reducing COD and color of BDE. The results show that at operating conditions of pH 9, CD 153 A/m², and treatment time (t_R) of 100 min, 84% COD and 94.89% color reduction was achieved. The energy consumption of 30.33 Wh/dm³ of BDE, estimated for 100 min of EC operation and electrode loss was 49 mg/dm³ of BDE.

The settling and filterability characteristic of treated BDE-MSIWW was found to be the best at pH 9. The foams and residues obtained from the EC-treated BDE sample have a possibility of being used as fuel for incineration in the furnace. However, complete removal of COD and color from the BDE-MSIWW was not possible using EC process. Thus post-treatment after ECT is needed to meet the discharge standards prescribed by pollution-control agencies. In order to obtain the industry-specified standards, EC process in combination with a membrane separation process will be advisable.

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