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TREATMENT OF AYURVEDIC PHARMACEUTICAL WASTEWATER BY ELECTROCOAGULATION

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ABSTRACT

Ayurvedic medicines are growing in popularity worldwide. Ayurvedic pharmaceutical industries have become the major contributors of water pollution nowadays. Characterization of this wastewater revealed high Chemical oxygen demand (13200 mg/L), Biochemical oxygen demand (4000 mg/L), Total solids (10000 mg/L) and acidic nature of wastewater. This highly concentrated nature of wastewater requires the provision of a proper pretreatment system before mixing into conventional biological treatments. Present study focuses on the feasibility of Electrocoagulation (EC) treatment on Ayurvedic pharmaceutical wastewater (APW). The Electrochemical treatment systems of lab scale were developed with mild steel as anode and stainless steel as cathode of size 12 X 6.5 cm at variable electrode spacing for the study. The parameters selected for the investigation include wastewater pH, inter electrode distance, Current density and electrolysis time for the system. Aiming the selection of the best operating conditions several studies were carried out by varying the parameters within the selected range and optimization of parameters were done for maximum Chemical oxygen demand removal. EC system with wastewater pH of 6, inter electrode distance 0.5 cm, current density of 119.65 A/m^2 resulted in a Chemical oxygen demand removal of 90% for a reaction time of 60 minutes.

Keywords: Chemical oxygen demand, Electrocoagulation, Feasibility, Optimization, Pharmaceutical Wastewater

I. INTRODUCTION

Directly or indirectly, water affects all facets of life. Without it, there would be no vegetation on land, no oxygen for animals to breathe and the planet would look entirely different than it does today. Water is necessary to keep people's bodies and the environment healthy and should be valued and protected as the precious resource it is.Every day an increasing amount of pollution seeps into rivers and lakes making them toxic to humans, and underground aquifers .They are being depleted at an alarming rate. Making an adequate supply of water available to everyone alive today is a monumental task, and ensuring that there is enough water for all future generations will require an unprecedented level of international cooperation and compassion.It is important to appreciate the fact that only 3 per cent of the world’s water is fresh and roughly one-third of it is inaccessible. The rest is very unevenly distributed and the available supplies are increasingly contaminated with wastes and pollution from industry, agriculture and households. Over the years, increasing population, growing industrialisation, expanding agriculture and rising standards of living have pushed up the demand for water. Industrial wastewater treatment and reuse is one of the effective solutions for the problem of water crisis. This covers the mechanisms and processes used to treat wastewater that is produced as a by-product of industrial or commercial activities. After treatment, the treated industrial wastewater (or effluent) may be reused or released

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to a sanitary sewer or to a surface water in the environment so that the remaining reserve of water remain unpolluted.

Herbal pharmaceutical industries are among the leading pharmaceutical industries worldwide. Though the product of these industries is based on herbs, synthetic chemicals are also used in the manufacturing process. Chemicals that are used include alcohol, sugar, gelatin, lactose, mineral salts, clays and different organic solvents. Herbal pharmaceutical industries generate a large volume of high strength wastewater. It cannot be discharged directly into the surface water, as it putrefies very fast. Herbal pharmaceutical waste is a complex constituent of alkaloids, plant extracts and toxic solutes, which contribute to environmental pollution. Untreated pharmaceutical wastewater discharge into the natural environment causes health hazards to existing flora and fauna and therefore, treatment of the effluents is necessary to bring down the concentration of pollutants to desired limits, before they are finally discharged into the natural systems Hence, a treatment methodology is required which is easy to operate and can be easily accepted by the local Ayurvedic manufacturing units.

Recent researches have shown that electrochemical techniques can offer a good opportunity to prevent and remedy pollution problems due to strict environmental regulations. Electro-coagulation(EC) process is a simple and efficient method that have attracted a great deal of attention for the treatments of various industrial wastewaters. The advantages of this process as compared to the conventional methods are easy to operate, less retention time, lower operating costs, absence of adding chemicals , rapid sedimentation of the electrogenerated flocs and less sludge production and requires a simple equipment. But the effectiveness of these treatments on different types of wastewater is varying. Hence it is important to study the effectiveness of electrocoagulation on Ayurvedic pharmaceutical wastewater before suggesting them as a treatment option. Treatment of ayurvedic wastewater has been mainly done by biological methods in the past years[1][2][3]. This work attempts to study the feasibility of Electrocoaagulation for COD reduction in Ayurvedic pharmaceutical wastewater(APW).

II. METHODOLOGY

The wastewater used for the study was collected from Oushadi pharmaceuticals Ltd. Thrissur. The experimental runs were conducted in the lab scale reactor and all the chemicals used were of analytical grade and were purchased from Cheminth chemicals Thrissur.

2.1 SAMPLING OF AYURVEDIC PHARMACEUTICAL WASTEWATER

The samples were taken from the equalization tank of the treatment unit after the skimming operation. The wastewater was stored at a temperature lower than 4^0C but above the freezing point of water. For the characterization of wastewater analysis were done for all samples collected as per standard methods. The range of values of characteristics of raw ayurvedic pharmaceutical wastewater (APW) is shown in the Table.1.

Table.1 Characteristics of ayurvedic pharmaceutical industrial wastewater

Parameters	Unit	Range
pH		4-5
Chemical oxygen demand(COD)	mg/L	9000-14000
Biochemical oxygen demand (BOD- 3 day)	mg/L	4000-6000
Ammonia - nitrogen	mg/L	20-60
Nitrate - nitrogen	mg/L	50-70

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Total solids	mg/L	8000-12000
Turbidity	NTU	180-270
Conductivity	mS/cm	5-9.5
Total phosphorous	mg/L	5-20

The presence of high Chemical oxygen demand and Biochemical oxygen demand indicate that it will be favorable to provide a pretreatment to the wastewater prior to biological treatment so that it will reduce the load on the latter and improve its efficiency.

2.2 Preparation of Synthetic Wastewater

Synthetic wastewater was prepared for the experimental investigation in order to keep the characteristics of wastewater unchanged throughout the period.Synthetic wastewater was prepared by mixing ayurvedic pharmaceutical products such as Chyvanaprasam(15mg/L), Sukumararasayanam(10mg/L), Haridrakhandam(1.5mg/L) and Jeerakarishtam(40 mL/L) in distilled water after several trials. The pharmaceutical products were obtained from ayurvedic medical shop and were used without further purification.The characteristics of synthetic wastewater prepared is listed in Table 2.

Table 2. Characteristics of synthetic wastewater

Parameters	Unit	Value
pH		4.3
Chemical oxygen demand(COD)	mg/L	12000
Biochemical oxygen demand (BOD- 3 day)	mg/L	3800
Ammonia - nitrogen	mg/L	33
Nitrate - nitrogen	mg/L	25
Total solids	mg/L	7600
Turbidity	NTU	273
Conductivity	mS/cm	10.2
Total phosphorous	mg/L	28

Fig.1 Synthetic wastewater prepared

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2.3 Experimental Setup

The electrocoagulation experiments were carried out in a cylindrical lab scale batch reactor, made of glass having a reactor volume of 1 liter. One pair of mild steel and stainless steel electrodes having the thickness 1 mm and dimension 12 cm X 6.5 cm with 58.5 cm^2 effective surface area was used in each experimental run.Mild steel and Stainless steel plates were used as anode and cathode respectively. The electrode gap between the two electrode plates can be varied in the trials and the electrodes were kept 5 cm from the bottom of the reactor for homogeneous agitation of the solution by using magnetic bar. The reactor is put on the magnetic stirrer. All the experiments were conducted at room temperature.0.8 L of wastewater was treated during each experimental run. Hydrochloric acid (0.1 N**)** and Sodium hydroxide (0.1N) aliquots were used for adjusting the solution pH from initial levels to desired value. A digital current power supply (0-30V, 1-5A) was used to maintain the constant current density for each run. The anode and cathode were connected to the positive and negative outlets of the DC power supply.

Fig.2 Electrode arrangement

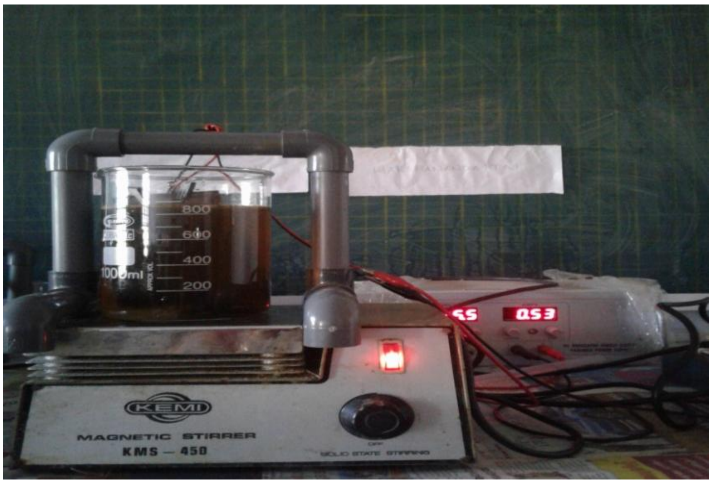

Fig.3. Experimental setup

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2.4 Experimental Procedure

Batch experiments were performed to study the efficiency of Electro-coagulation process in the treatment of Ayurvedic pharmaceutical wastewater. The process performance was determined by optimizing the parameters like pH, current density, inter electrode distance and electrolysis time. The parameters were varied in the range pH(4-10), current density(85.47-170.94 A/m^2), inter electrode distance(0.5-1.5 cm), and electrolysis time(15-90 minutes) After adjusting the parameters to desired value, the reaction mixture was continuously stirred by the magnetic stirrer. Trials were conducted with different parameter values within the selected range to obtain the optimized parameter values for both the processes. After the treatment time, samples were collected, filtered and analyzed for COD, since COD removal is a significant advantage of EC process. The electrode plates were manually cleaned with 15% HCl solution before and after treatment for their reuse. Initial trials were performed by varying parameter values in order to narrow down the range of values to obtain optimum results. ‘ One factor – at – a time’ method was used to optimize the process parameters for COD removal.

Fig.4 Wastewater before and after treatment

2.5 Analytical Methods

Wastewater characteristic were studied as per standard methods. The instruments/methods employed for the analysis were listed in Table 3.

Table 3. Analytical methods for characterization

Parameters	Method	Instrument
pH	Electormetric method	Water quality analyzer
COD	Closed reflux method	Reflux apparatus
BOD	Titrimetric method	BOD incubator
Ammonia - nitrogen	Nesselerisation method	Double beam spectrophotometer
Nitrate - nitrogen	Phenol disulphonic acid method	Double beam spectrophotometer
Total suspended solids	Filtration and drying method	Hot air oven
Total dissolved solids	Electrometric method	Water quality analyzer

Turbidity	Turbidimetric method	Turbidimeter
Conductivity	Electrometric method	Water quality analyzer
Total phosphorous	Digestion and ascorbic acid spectrophotometric method	Double beam spectrophotometer

III. RESULTS AND DISCUSSION

3.1 Optimization of Process Parameters

Identifying the influencing factors and optimizing the COD removal efficiency is of primary importance in this study. Identification of significant variables is done using One factor at a time method. The most widely used screening and optimization strategy is the Design of Experiment (DOE) method, by which certain factors are selected and deliberately varied in a controlled manner, in order to study their effects, facilitate process comprehension and even to improve the performance.

Experiments were initiated for screening the significant factors. Batch studies were conducted with working volume of 800 mL synthetic wastewater, with Mild steel (MS) as anode and stainless steel(SS) as cathode. The variables chosen for the present study were pH, electrolysis time, inter electrode distance and current density. Initial trials were conducted to study the effect of process parameters on the process.

3.1.1 Effect of pH

During the batch studies to optimize pH, the electrolysis time, inter electrode distance and current density were fixed as 30 minutes, 0.5 cm, and 170.94 A/cm^2 respectively. The wastewater pH lies in the acidic range. Experiments were conducted at acidic, neutral and basic pH levels. All the experiments were conducted at room temperature. The effect of pH in the removal efficiency of pollutants during EC is as shown in Table 4.

Table 4. Effect of pH during Electro-coagulation

pH	COD removal (%)	Turbidity removal (%)	TDS removal(%)	TSS removal(%)
4	66.67	77.65	80	85.9
10	50	81.68	81.67	90.6
7	80	86.81	89.5	96.8
6	83.33	88.64	91	95.9
8	66.67	86.44	86.67	91.8

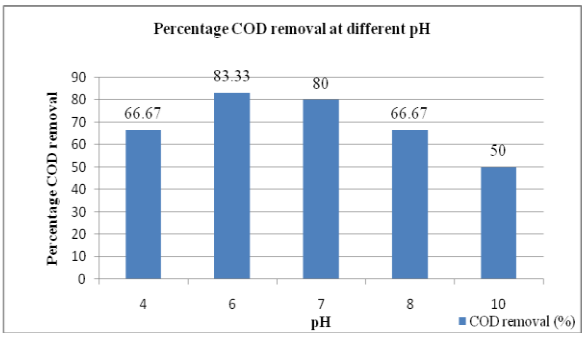

Fig.5 Percentage COD removal at different pH

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From Fig.5 it can be observed that the COD removal efficiency is very less in highly acidic and highly alkaline pH range. When pH is beyond 9, the decrease in efficiency is due to the formation of soluble metal hydroxides since only insoluble metal hydroxides of iron can remove pollutants by electrostatic interaction [4]. At a lower pH range (pH<6.5) , the concentration of proton in the solution is reduced by H_2. Thus Fe-hydroxide concentration becomes less and therefore COD removal efficiency is reduced [4]. Here from the initial trials it can be seen that Electro- coagulation has higher efficiency in COD removal near the neutral range.

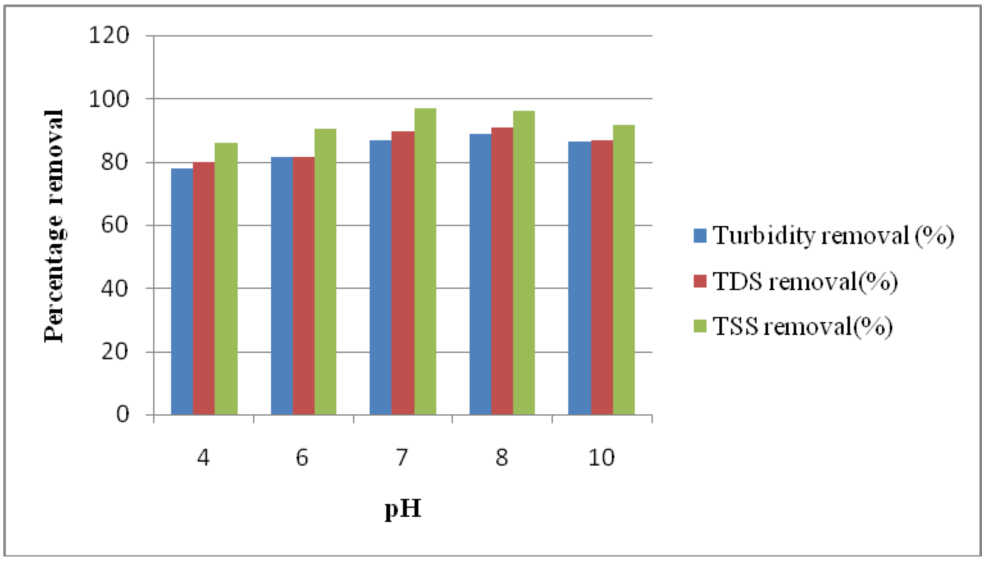

Fig.6 Percentage removal of Turbidity, TSS and TDS at different Ph

It can be observed that Electro-coagulation provides high turbidity removal. Maximum turbidity removal is observed in the range of pH 6 to 8. Hence, it can be concluded that compared to raw APW, electrocoagulation led to a significant improvement in the turbidity removal. Consequently, it can be suggested that electrolysis followed by the sedimentation process before a post biological treatment would result in high turbidity removal. It was also observed that electrolysis is effective in suspended solids removal. Electro-coagulation provided TSS removal efficiency above 85% in acidic, neutral and basic ranges. In the lower pH range the slight decrease in TSS removal is due to the lack of proper floc formation[7].

Electro-coagulation is effective in the removal of dissolved matter by charge neutralization and electrostatic interaction. The cations generated by dissolution of sacrificial anodes with applied electrical field, induce flocculation of the dispersed impurities and later they get settled down. At very acidic and very basic pH ranges the floc formations does not occur properly and TDS removal efficiency decreases. When pH exceeds 9, the formation of soluble Fe hydroxides add to the TDS of the wastewater. From the above results, it can be concluded that electro-coagulation does not function properly in very acidic and very alkaline range. Maximum efficient for EC is observed in the pH range of 6 to 8.

3.1.2 Effect of residence time on efficiency of Turbidity and Total solids removal

Water was colored green by dissolution of stainless steel electrode and then changed to yellow and turbid. The green color is due to the formation of Fe^{2+} ions and the yellow is due to Fe^{3+} which are formed during the electro-coagulation process. Fe^{2+} ions are formed at the dissolved electrode directly. They are oxidized easily under normal conditions by dissolved oxygen in water to Fe^{3+}[7]. In this experiment the effect of residence time

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in the EC reactor on the turbidity, TSS and TDS removal efficiency was investigated at constant current of 1A, electrode gap of 0.5 cm and pH of 6. Fig. 7 shows the change of turbidity and TDS removal efficiencies as a function of residence time.

Table 5 Effect of residence time on TSS, TDS and Turbidity removal

Residence time(minutes)	TSS removal efficiency (%)	TDS removal efficiency(%)	Turbidity removal efficiency(%)
3	81.25	82.75	86.44
6	93.75	90.83	91.57
9	93.43	91.83	90.47
12	91.56	91.75	89.37
15	92.5	89.92	89.17

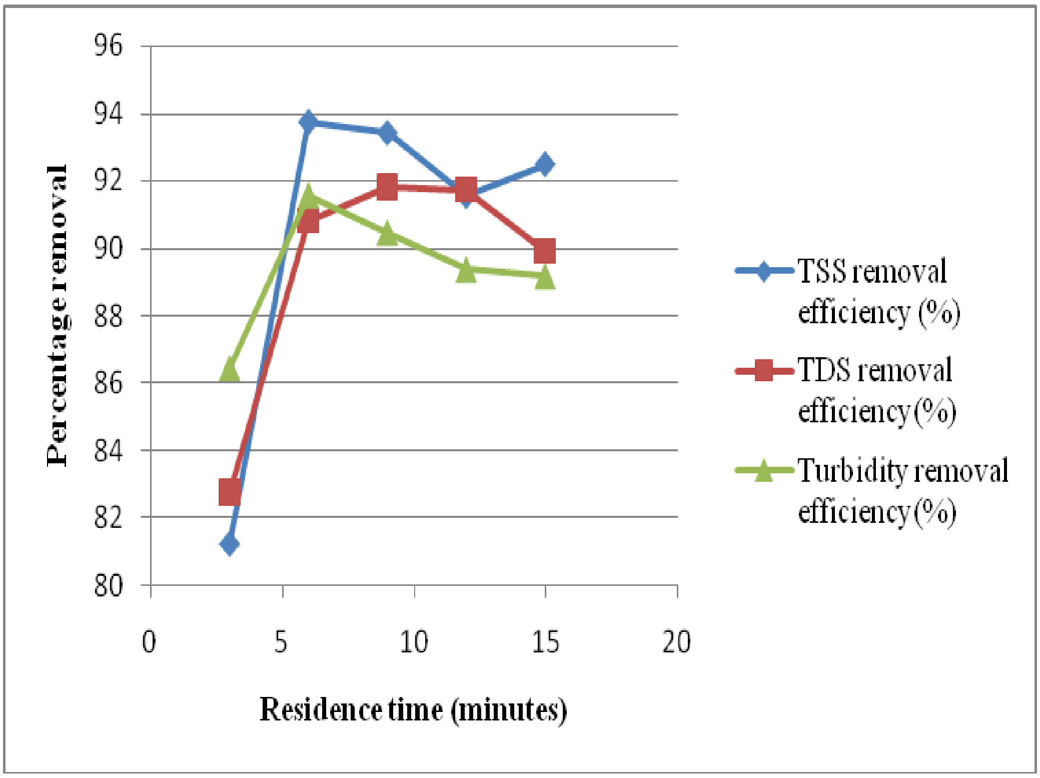

Fig.7 Effect of residence time on TSS, TDS and Turbidity removal

It is clear that Turbidity (NTU) removal efficiency is increased from 86.44% at 3 min residence time to reach a maximum of 91.57 % at 6 min residence time. There is no advantage of increasing the residence time after 6 min since thereafter the change is slight. The same is true for the effect of increasing residence time on the efficiency of TSS removal. The value has increased from 81.25% at 3 min residence time to a maximum value of 93.75% at 6 min. The value remained almost constant thereafter. The TDS removal exhibited a maximum value of 91.83% at 9 minutes. In this process electro coagulation process there are two stages: The first is destabilization and collection of particles which takes place in a short period. The second which includes precipitation of particles takes a longer time. It is clear that Fe^{2+} formed at the electrode is responsible for destabilizing the negative particles in water. The quantity of Fe^{2+} irons is small at the beginning in the EC

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reactor so Fe^{3+} is not enough to precipitate all colloids from water. So the efficiency of removing turbidity, TDS and TSS is low at small residence times.

3.1.3 Effect of inter electrode distance

In the reactor setup, three electrode gap arrangements are provided i.e. 0.5 cm, 1cm and 1.5 cm. To study the effect of inter electrode distance on the treatment efficiency trials were conducted varying the electrode gap and keeping other factors constant (i.e. pH-6, current density 170.94A/m^2 and electrolysis time-30 minutes).

Table 6 Effect of inter electrode distance on percentage COD removal

Inter electrode distance	COD removal efficiency(%)
0.5	83.33
1	73.33
1.5	66.67

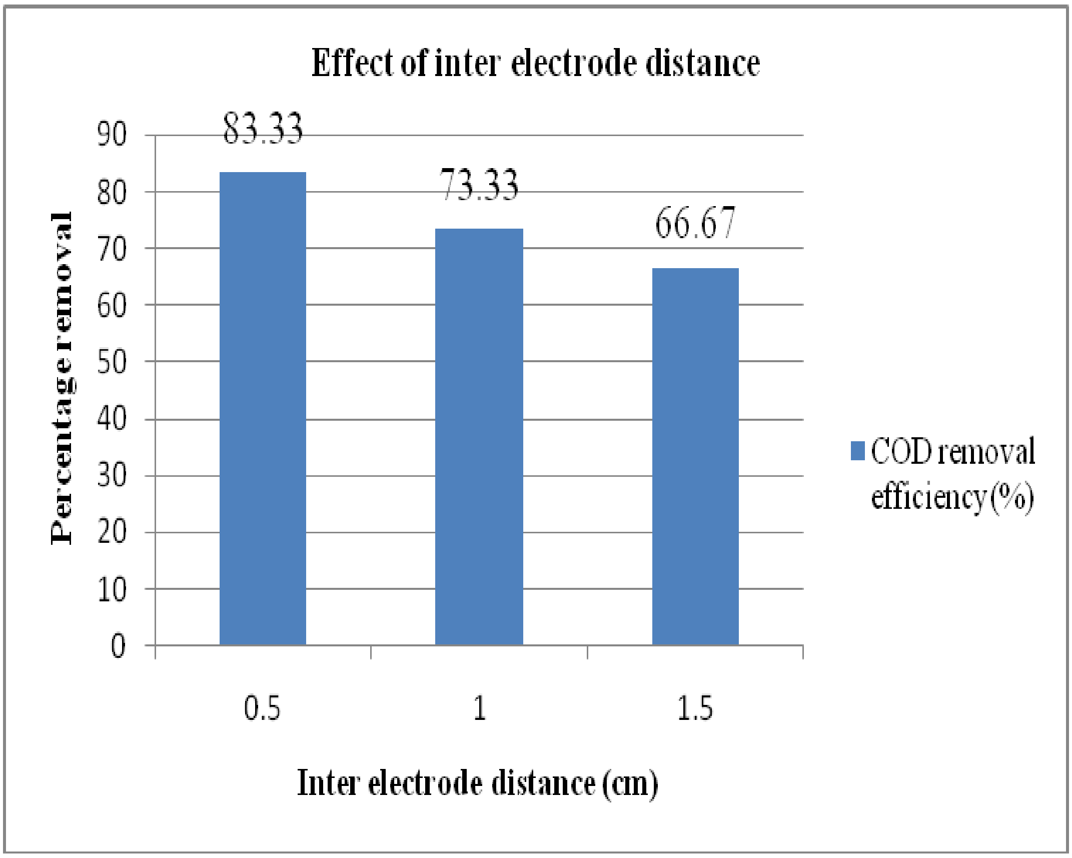

Fig.8 Effect of Inter electrode distance on Percentage COD removal

The range of electrode gaps was fixed from literature. From observations, it can be understood that as the electrode distance increases, COD removal efficiency increases i.e. better treatment occurs due to the more effective current transfer.

3.1.4 Effect of electrolysis time

In order to understand the effect of electrolysis time on the treatment efficiency, experiments were conducted varying coagulation time and keeping all other factors constant (pH -6, current density 170.94A/m^2 and electrode gap-0.5)[6].

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Table 7 Effect of electrolysis time on percentage COD removal

Time (minutes)	Percentage COD removal
15	53.33
30	83.33
60	86.67
90	80

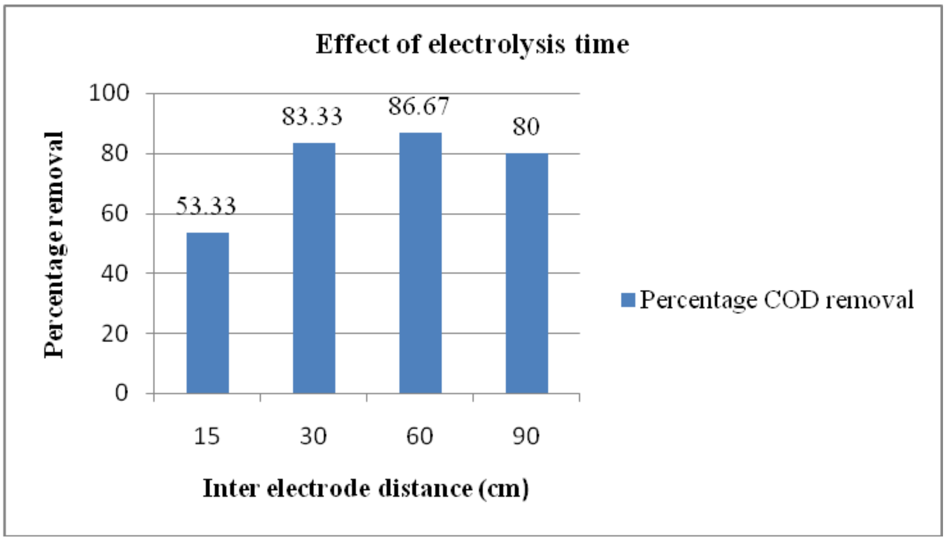

Fig.9 Effect of Electrolysis time on Percentage COD removal

It was observed that initially as the time increases, the COD and color removal efficiencies increases due to the increase in Fe^{2+} ion concentration in the suspension. But later on excess Fe^{2+} hydroxide grow like a film and limit the color and COD removal by extra resistance forced on surface film.

3.1.5 Effect of current density

Initial trials were conducted to establish the effect of current density on Electro-coagulation process by one factor at a time method.

Table 8 Effect of Current density (A/m^2)

Current density(A/m^2)	Percentage COD removal
51.28	80
85.47	83.33
119.65	90
70.94	83.33

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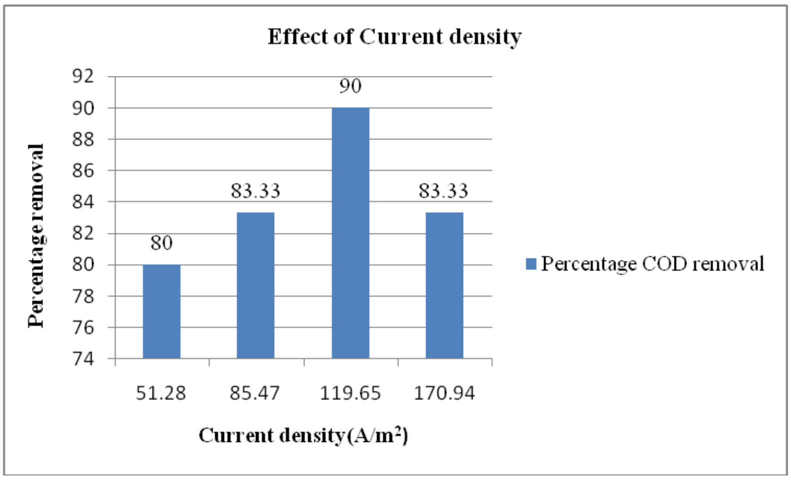

Fig.10 Effect of Current density on Percentage Cod removal

Fig.10 depicted the effect of Current density (j) on COD removal. As j increases, COD removal efficiency also increases due to the increase in Fe^{2+} ion in suspension. This increase is due to the increase in electrode dissolution at higher current densities. But as J increases beyond a limit, excess Fe^{2+} hydroxides grow like a film and limit the color and COD removal by extra resistance forced on surface film.

3.1.6 Effect of Salt concentration

To study the effect of addition of electrolyte on the performance of electrocoagulation system trials were conducted by adding sodium chloride to reactor volume at different dosage varying in the range 0-2 g/L [5]. All other parameter values were kept as the optimized values obtained from previous trials.

Table 9 Effect of salt concentration on Electrocoagulation

Dosage of electrolyte g/L	Electrical conductivity(mS/cm)	COD (mg/L)	Percentage COD removal
0	10.2	1200	90
0.5	12	680	94.3
1	12.6	600	95
1.5	13.7	520	95.6
2	13.9	480	96

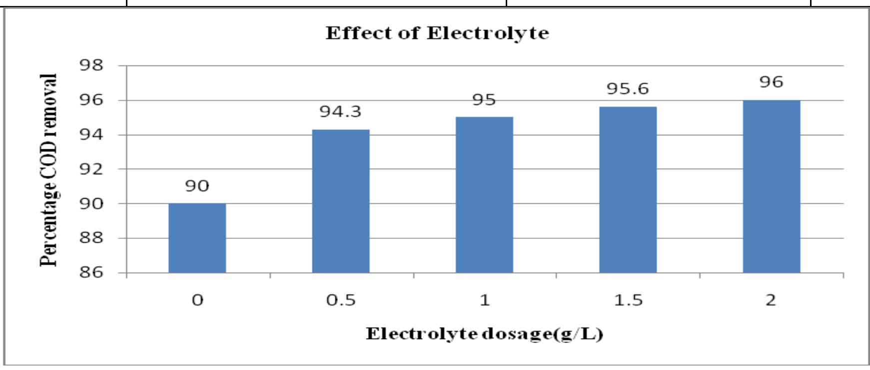

Fig.11 Effect of electrolyte on COD removal

Chlorine and hypochlorite were produced due to the addition of sodium chloride. Fig. 11 shows the effect of salt concentration on the COD removal efficiency Fig. clearly illustrated the COD removal efficiency at any value of pH increase with increase the *m* because at higher *m* , maximum amount of active chlorine species

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generated which were maximum consumed in APW degradation via direct and indirect electrolysis and consequently increases the removal efficiencies and decrease the energy consumption. But as the salt concentration increases beyond 1g/L the difference is seemed to be marginal.

3.2 Application of Optimized results in real wastewater

The tests were carried out in real wastewater collected from Oushadi effluent outlet as per optimum conditions of parameters for EC. Electrocoagulation was performed at conditions of pH -6 ; electrode gap-0.5 cm ; Electrolysis time – 60 minutes Current density - 170.94 A/m^2 in order to study the effect of process in real wastewater. Results obtained are shown in Table 10.

Table 10. Characteristics of wastewater before and after treatment

Treatment	Parameter	Unit	Value	
			Before treatment	After treatment
EC	COD	mg/L	13000	1200
	BOD	mg/L	4000	2010
	Total solids	mg/L	8000	510
	Nitrate	mg/L	50	9
	Phosphate	mg/L	20	4.5
	Turbidity	NTU	180	34

The effluent general standards as per scheduleVI inserted by Rule 2(d) of the Environment (protection) Second Amendment Rules, 1993 for the discharge of environmental pollutants is given in Table11.

Table 11.General standards for discharge of effluents

Parameters	Maximum limits for discharge	
	Into inland surface water	Into land for irrigation
pH	5.5-9	5.5-9
COD(mg/L)	250	-
BOD(mg/L)	30	100
SS(mg/L)	100	200
Nitrate(mg/L)	10	-
Phosplate(mg/L)	5	-

After EC treatments, only nitrates and phosphates have met the general discharge standards. The electrolytic treatments are mainly effective in removing the non biodegradable fraction of pollutants from wastewater. Since ayurvedic pharmaceutical wastewater is having a relatively high amount of biochemical oxygen demand (BOD), it is important to study the effect of these treatments on BOD removal efficiency as well. From the results, it can be observed that electrolytic treatments have only a marginal effect (around 50%) on BOD removal efficiency. Hence for the treatment of ayurvedic wastewater these treatments can only be used as a primary treatment and provision of a secondary biological treatment is necessary

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IV. CONCLUSIONS

Present study involved the application of electrocoagulation process for the treatment of ayurvedic pharmaceutical wastewater. The removal of various parameters by both treatments were analysed. The parameters such as pH, nitrate and phosphate were only brought down below effluent discharge standards. For EC treatment , the optimum conditions obtained were of pH – 6, Time – 60 minutes, electrode gap of 0.5 cm and Current density 119. 65 A/m^2 and the corresponding COD removal is of 90 %. Eventhough the COD removal percentage is high, the effluent COD value does not satisfy disposal standards. The BOD removal rate is relatively low. Hence it can be concluded that electrolytic treatments are suitable only as a pre-treatment for ayurvedic pharmaceutical wastewater and a biological post treatment is required. It can also be seen that the addition of electrolyte to reactor volume increases the treatment efficiency to a certain extend.

V. SCOPE FOR FUTURE STUDY

- In the present study an attempt has made by using Mild steel and Stainless steel electrodes for the treatment of color and COD from Ayurvedic pharmaceutical industry industry. Other electrode such as aluminium, Copper, graphite titanium etc can be used which has a higher electrical transmission capacity.
- The sludge generated after the EC process need to be studied as it may contain dissolved metals and may require special attention
- Operating cost analysis of EC treatments will be suitable for understanding the economic aspect of this treatment

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