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Research Article

ELECTROCOAGULATION TREATMENT STUDY OF DYEING INDUSTRIAL EFFLUENT IN PRESENCE OF ADSORBENT

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ARTICLE INFO	ABSTRACT
Article History:	The residual dyes from different industries (e.g., textile, paper and pulp, dye, pharmaceutical
Received 06 th March, 2015 Received in revised form 14 th April, 2016 Accepted 23 rd May, 2016 Published online 28 th June, 2016	industries, tannery <i>etc.</i>) are considered a wide variety of organic pollutants introduced into the natural water resources or wastewater treatment systems. Several conventional treatment methods have been carried out for this purpose such as adsorption, coagulation, flocculation <i>etc.</i> However, each method has some disadvantages. Electrocoagulation is an attractive method and effective
Kon Words.	method for the treatment of various kinds of wastewater. In the present study, characterization, correlation analysis and treatment by Electrocoagulation (EC) were carried out for the dyeing
Rey Worus.	effluent samples collected from the industry located between Arunnukkottai and Kariyanatti near
Electrocoagulation, effluent, adsorbent, correlation, graphene, carbonnanotubes,	Madurai, Tamilnadu, India. Most of the water quality parameters (WQPs) of dyeing effluent were found to be higher than the limit prescribed by Bureau of Indian Standards (BIS) for the discharge of industrial effluent. EC technique is employed for the treatment of dyeing effluent with and without adsorbent using iron and aluminium electrodes. The decrease in values of Total Dissolved Solids (TDS) was higher in the case of EC with and without Graphene (GR) and Multi Walled Carbon Nanotubes (MWCNTs) than Commercial Activated Carbon (CAC). The decrease in

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INTRODUCTION

One of the main sources of water pollution worldwide is the textile industry and its dye-containing wastewaters. The discharge of dye-containing effluents into the water environment is undesirable, not only because of their colour, but also because many of dyes released and their breakdown products are toxic, carcinogenic or mutagenic to life forms mainly because of carcinogens, such as benzidine, naphthalene and other aromatic compounds¹⁻⁶. Without adequate treatment these dyes can remain in the environment for a long period of time. Several conventional treatment methods have been carried out for this purpose such as adsorption, coagulation, flocculation, biological and chemical oxidation. Adsorbents are usually difficult to regenerate, chemical coagulation causes additional pollution due to the undesired reactions in treated water and produces large amounts of sludge and biological methods are not suitable for most dyeing wastewaters due to the harmful effects of some commercial dyes on the organisms used in the process. Conventional methods are also usually

expensive and treatment efficiency is inadequate because of the large variability of the composition of textile wastewaters. Considering all the above facts, a detailed study of dyeing effluent is required in order to curtail water pollution⁷⁻¹². This paper deals with the discussion of the results obtained from the studies on the characterization, water quality index, correlation, linear regression, assessment of irrigation quality and electrocoagulation treatment of industrial effluent discharged from the dyeing industry located between Aruppukkottai and Kariyapatti near Madurai, Tamilnadu, India.

Experimental Methods

concentration of both anions and cations are relatively higher in EC with and without GR and MWCNTs than CAC. The result of present investigation on dyeing industrial effluent revealed that generally the EC process with adsorbent is an effective tool for the treatment of industrial effluent.

Chemicals and Regents

The adsorbent materials CAC and MWCNTs were purchased from Sigma-Aldrich Chemicals used without any further purification. GR was synthesized by the modified Hummer's method¹³⁻¹⁶. All the other chemicals and reagents used in the studies are analytical grade and used as received⁴¹. The samples were collected bimonthly over a period of one year in a 2L polythene can. The values of physico-chemical characteristics of dyeing industrial effluent are shown in Tables 1.and 2

potential. The WQPs of effluent have been analyzed after 20min.

Table 1 Water quality parameters of dyeing industrial effluent

S.No.	Т	pН	K	TDS	TSS	THA	HAT	HAP	Cľ	SO4 ²⁻	Na^+	\mathbf{K}^{+}	Ca ²⁺	Mg ²⁺	BOD	COD
1	30.4	7.8	3412	3125	1640	1486	518	968	897	215	294	17	416	108	532	976
2	32.6	8.2	2610	1242	3810	448	201	247	282	235	244	6.5	76	66	46	446
3	29.4	11.6	12860	18961	17648	438	204	234	1512	4760	776	110	101	49	258	3241
4	30.8	9.8	10065	8246	6892	464	198	266	916	452	546	82	292	18	1068	1745
5	31.3	8.9	4140	4058	1210	924	522	402	1068	272	332	26	118	164	678	1165
6	32.1	11	9150	13210	12555	310	146	164	1068	3431	545	78	72	34	2942	3726
7	29.1	6.5	3664	6358	1682	768	82	686	2462	932	476	42	154	97	1882	8682
8	30.6	8.2	1654	814	112	442	262	180	278	132	412	48	86	57	115	318
9	31.4	8.3	7026	6141	5440	560	357	203	792	618	496	44	100	90	4882	6052
10	30	12	3417	3672	1242	340	90	250	1292	232	314	26	108	17	1120	1264
11	30.5	7.4	5978	8868	754	440	195	245	1368	914	332	23	162	8	1116	1462
12	30.9	6.4	7128	7858	5851	625	115	510	1682	1081	556	74	168	42	3244	4782
13	29.2	8.2	4292	4228	2013	685	165	520	1082	312	413	37	98	95	642	1062
14	29.9	7.5	5630	4848	3342	770	245	525	1245	794	457	32	82	122	968	1872
15	28.6	10.1	3963	3892	1453	810	120	690	1385	265	584	73	182	78	1961	3041
16	29.4	8.9	2152	1672	451	865	110	755	1622	135	465	75	276	26	421	1006
17	29.7	8.8	4410	4876	1362	930	180	750	1065	268	542	65	302	45	1652	1841
18	30.4	9.7	3872	3852	1672	975	75	900	992	232	496	51	215	112	1126	2261
19	32	8.4	3002	3465	1016	855	175	680	1072	210	495	62	282	40	1116	1442
20	32.9	9.2	3210	2772	1672	620	175	445	1248	192	542	70	185	34	976	1856
21	30.1	8.1	1060	12246	5665	1290	515	775	1992	1842	596	75	364	84	3162	4694
22	30.6	8.7	4314	4244	2368	420	165	255	1115	292	314	40	94	42	825	1256
23	31.5	8.1	7996	12846	10112	895	225	670	2612	2210	912	102	76	171	3115	7062
24	33.2	7.8	4010	4413	1447	375	195	170	1340	265	506	73	104	21	1162	2026

Units: T in °C, K in µmho/cm and remaining parameters except pH are in mg/L

Table 2 Statistical data of dyeing industrial effluent

S.No	WQPs	Ν	Range	Minimum	Maximum	Sum	
1	Т	24	4.60	28.60	33.20	736.60	30.69
2	pН	24	5.60	6.40	12.00	209.60	8.73
3	K	24	11800.00	1060.00	12860.00	119015.00	4958.96
4	TDS	24	18147.00	814.00	18961.00	145907.00	6079.46
5	TSS	24	17536.00	112.00	17648.00	91409.00	3808.71
6	THA	24	1176.00	310.00	1486.00	16735.00	697.29
7	HAT	24	447.00	75.00	522.00	5235.00	218.13
8	HAP	24	804.00	164.00	968.00	11490.00	478.75
9	Cl	24	2334.00	278.00	2612.00	30387.00	1266.13
10	SO_4^{2-}	24	4628.00	132.00	4760.00	20291.00	845.46
11	Na^+	24	668.00	244.00	912.00	11645.00	485.21
12	K^+	24	103.5	6.50	110.00	1331.50	55.48
13	Ca ²⁺	24	344.00	72.00	416.00	4113.00	171.38
14	Mg^{2+}	24	163.00	8.00	171.00	1620.00	67.50
15	BÕD	24	4836.00	46.00	4882.00	35009.00	1458.71
16	COD	24	8364.00	318.00	8682.00	63278.00	2636.58

The electrochemical cell consisted of two mono polar electrodes, one cathode (mechanically polished) and another anode viz., iron (mild steel-MS) and aluminium, respectively. Both the electrodes are purchased from the local market (purity: Al = 99.5%, Fe = 99%). The dimension of iron electrode and aluminium (anode) electrode is 10.4cm×2.5cm×0.6cm each. The spacing between the electrodes was maintained at 2.8cm. The electrodes are connected to a DC power supply (120V, 20A). About 100mL of well-mixed, screened, homogeneous industrial effluent was taken in the borosilicate electrochemical cell. The temperature of the effluent before EC was noted to be 30 °C. The temperature was maintained throughout EC (deviation ± 1 C). For efficient electrochemical coagulation, 30V DC was passed through the electrodes throughout the EC process by getting a constant current dens125Am⁻². The whole set-up was placed on a magnetic stirrer and the sample under study was subjected to constant stirring in order to avoid concentration over Similar EC experiments were carried out in the presence of CAC, MWCNTs or GR with constant, slow stirring to facilitate effective electro coagulation. After each EC process the effluent was filtered through What man 42 filter paper and analyzed for various water quality parameters¹⁷⁻²³.

RESULTS AND DISCUSSION

Comparison of WQPs of dyeing industrial effluent with BIS Limit

The average **temperature** of the dyeing industrial effluent propose that it is not thermally polluted. The pH values, by and large, exceeded the permissible limit prescribed by BIS. **Total Dissolved Solids (TDS)**, which is also a measure of salinity that affects the taste of potable water. The salts are used as additives in dyeing process. The average value of specific conductance of the dyeing industrial effluent is 4959µmhocm⁻¹ which is against the tolerance level of 3000µmhocm⁻¹ prescribed by BIS. From the mean values of specific conductance as well as TDS of dyeing industrial effluent, it is concluded that the effluent is polluted beyond the tolerance limit prescribed by BIS. The high value of **Total Suspended Solids (TSS)** of the dyeing industrial effluent suggests that the effluent is polluted severely. The average value of **total hardness** as mg of CaCO₃ per litre (THA, in mg/L) is 697mg/L, which indicate that the effluent sample is hard and highly polluted. The average value of temporary hardness, as mg of CaCO₃ per litre (HAT, in mg/L) of the dyeing industrial effluent is 218 and it is beyond the permissible level of BIS. The average value of **permanent hardness** (HAP, in mg/L of CaCO₃) of the dyeing effluent is 479. Permanent hardness could be removed by using water softening techniques like ion exchange, lime soda process *etc.*, or by electrocoagulation technique which successfully removes the dissolved ions.

Correlations among Water Quality Parameters of Dyeing Effluent

The Correlation analysis of the WQPs of dyeing industrial effluent was carried out using the software known as "Statistical Package for Social Science" (SPSS) with the following objectives: (i)The task of rapid monitoring of water pollution and to predict linear relationship among WQPs. (ii) Only for WQPs pair with significant 'r' value linear regression study was considered the study^{22,23}. The correlation co-efficients (r values) between each pair of the 24 WQPs are calculated and are presented in the form of correlation co-efficient matrix in Table 3 and linear regression studies are given in Tables 4.

Table 3	Correlation	coefficient	matrix	of dyeing	industrial	effluent
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WQPs	Т	pН	K TI	DS TSS	ТНА	HAT	HAP	Cl	SO4 ²⁻	Na ⁺	\mathbf{K}^{+}	Ca ²⁺	Mg ²⁺	BOD	COD
Т	1	•											0		
$_{\rm P}{\rm H}$	-0.094	1													
K	0.000	0.332	1												
TDS	-0.091	0.253	0.756 1												
TSS	0.055	0.387	0.83 0.8	88 1											
THA	-0.287	-0.303	-0.381 -0.0	95 -0.224	1										
HAT	0.125	-0.198	-0.142 0.0	78 0.018	0.535	1									
HAP	-0.404	-0.253	-0.374 -0.1	51 -0.271	0.900	0.114	1								
Cl	-0.270	-0.197	0.145 0.4	95 0.243	0.258	-0.127	0.369	1							
SO_4^{2-}	-0.061	0.346	0.714 0.9	32 0.930	-0.167	0.019	-0.205	0.369	1						
Na^+	-0.050	0.151	0.529 0.6	85 0.651	0.081	-0.128	0.161	0.595	0.622	1					
K^+	-0.004	0.281	0.501 0.6	20 0.614	-0.052	-0.217	0.049	0.472	0.579	0.905	1				
Ca ²⁺	-0.207	-0.108	-0.282 -0.0	96 -0.240	0.722	0.319	0.685	0.063	-0.229	-0.023	0.077	1			
Mg^{2+}	-0.130	-0.253	-0.055 0.0	36 0.047	0.564	0.461	0.427	0.236	0.030	0.185	-0.137	-0.106	1		
BOD	0.093	-0.160	0.238 0.3	93 0.302	0.079	0.104	0.040	0.388	0.252	0.442	0.308	-0.026	0.170	1	
COD	-0.106	-0.252	0.300 0.5	18 0.401	0.114	-0.053	0.162	0.727	0.425	0.589	0.386	-0.112	0.336	0.755	1

The average value and the range of **chloride** of effluent are 1266 and 278-2612. And it is above the prescribed limit by BIS. The considerable presence of chloride in dyeing industrial effluent and prolonged discharge of this into water bodies may lead to eutrophication in due course. The average value of **sulphate** in mg/L is 845. The average value of Na⁺, Ca²⁺, Mg²⁺ **and K**⁺ ions in the dyeing effluent are higher than the allowed value of BIS²⁰⁻²³.

Bio-chemical Oxygen Demand is a measure of biologically degradable organic matter and COD is a measure of chemically oxidisable organic as well as inorganic matter. High load of organic compounds in the effluent may cause an increase in **BOD** and **COD** load and so inflict simultaneous depletion of dissolved oxygen (DO) concentration. The low DO corroborated with high BOD or COD may be due to high organic load, which may lead to the depletion of dissolved oxygen. Higher BOD indicates higher microbial activity. Absence of DO and high BOD and COD in the effluent is due to high organic load. Hence, these parameters are more significant than other WQPs in the characterisation of quality of the industrial effluents. The average values of BOD and COD of dyeing effluent are 1459 and 2637mg/L respectively.

Water Quality Index (WQI)

The WQI values of dyeing effluent are computed and it is approximately eleven times higher than the prescribed limit by BIS³⁴.

 Table 4 Linear Regression Study of dyeing industrial effluent

Sulphate vs TSS y = 3.47x + 874.98; r = 0.9297

1	5		,
S. No.	TSS	SO_4^{2-}	TSScal
1	1640	215	1621
2	3810	235	1690
3	17648	4760	17392
4	6892	452	2443
5	1210	272	1819
6	12555	3431	12781
7	1682	932	4109
8	112	132	1333
9	5440	618	3019
10	1242	232	1680
11	754	914	4047
12	5851	1081	4626
13	2013	312	1958
14	3342	794	3630
15	1453	265	1795
16	451	135	1343
17	1362	268	1805
18	1672	232	1680
19	1016	210	1603
20	1672	192	1541
21	5665	1842	7267
22	2368	292	1888
23	10112	2210	8544
24	1447	265	1795

Assessing Printing Effluent Quality for Irrigation Needs

The sodium absorption ratio (SAR_{min}, SAR_{max} and SAR_{av}), percent sodium (PS_{min}, PS_{max} and PS_{av}), Kelly's ratio (KR_{min}, KR_{max} and KR_{av}) and magnesium ratio (MR_{min}, MR_{max} and MR_{av}) of dyeing effluent study show that effluent is suitable

for irrigation purpose only after proper treatment has been $done^{35.40}$.

Electrocoagulation Treatment Studies of Dyeing Industrial Effluent

The treatment by EC processes has been carryout on dyeing industrial effluent to evaluate the percentage removal of various WQPs with and without adsorbents namely CAC, MWCNTs and GR. The optimization of dose of adsorbents (CAC, MWCNTs and GR) for EC studies of dyeing effluent was determined by measuring TDS (in mgL⁻¹), CAC was found to be 1g, for both MWCNTs and GR were 200mg²¹⁻²⁵.

Removal of WQPs of Dyeing effluents before and after EC processes

The physico-chemical WQPs of dyeing industrial effluent before and after EC with and without adsorbents in presence of iron cathode (MS) and aluminium anode electrodes is given in Table 5.

Table 5	Characteristics of dyeing industrial effluent
before	and after EC with and without adsorbent

	WQPs Values									
WOD-	D.f	After EC								
wQrs	EC	Without adsorbent	With CAC	With MWCNTs	With GR					
Colour	Black	Light brown	Almost colourless	Almost colourless	Almost colourless					
pН	8.1	7.5 (7)	7.4 (9)	7.5 (7)	7.4 (9)					
Κ	7996	2154 (73)	674 (92)	334 (96)	368 (95)					
TDS	12846	868 (93)	466 (96)	283 (98)	318 (98)					
TSS	10112	1006 (90)	578 (94)	250 (98)	296 (97)					
Na^+	912	416 (54)	204 (78)	140 (85)	162 (82)					
K^+	102	68 (33)	38 (63)	35 (66)	32 (69)					
Ca ²⁺	76	44 (42)	36 (53)	40 (47)	34 (55)					
Mg^{2+}	171	97 (43)	55 (68)	38 (78)	50 (71)					
Cl	2612	460 (83)	269 (90)	185 (93)	212 (92)					
SO_4^{2-}	2210	617 (72)	452 (80)	164 (93)	286 (87)					

The pH of the effluent is considerably reduced after EC with and without adsorbents. The result indicates that decrease in pH is due to decline in the concentration of H^+ ions present in the dyeing effluent.



Figure 1 Removal of TDS and TSS in dyeing effluent by EC with and without adsorbents

The H^+ ions present in the dyeing effluent may undergone electronation at cathode resulting in evolution of H_2 gas and

also adsorption of pollutants in the effluents on the adsorbent surface. The decrease in the values of TDS in dyeing industrial effluent after electrocoagulation with MWCNTs or GR are relatively higher than CAC and without adsorbent., and details are given in Fig. 1. The high percentage removal of TDS and TSS is due to the formation of coagulants and flocculants by electrolytically added Al^{3+} generated from aluminium anode. The dissolved and suspended particles undergo coagulation with Al^{3+} . The heavy coagulants get settled at bottom while light ones undergo electroflocculation and the hydrogen bobbles produce in the electrolytic cell uplift the later particles to the surface, from where in can be easily removed.

The reduction in the concentration of anions such as Cl^{-} and $SO_4^{2^-}$ during EC of dyeing effluent with and without absorbents, it may be due to (i) de-electronation of these anions at anode resulting in electrochemical oxidation, (ii) reaction of anions with Al^{3+} , $Al(OH)_3$ resulting in corresponding chloride and sulphate precipitates and (iii) the anions undergo adsorption on the surface of CAC, MWCNTs and GR during electrocoagulation.

$Cl^- \rightarrow e^- + \frac{1}{2}$	$Cl_2\uparrow$	(at anode)
$Al^{3+} + 3H_2O$	\leftrightarrow Al	$(OH)_{3(s)} + 3H^+$
$Al^{3+} + 3Cl^{-}$	\rightarrow	AlCl ₃ ↓

The decrease in the concentration of cations such as Na^+ , K^+ , Ca^{2+} and Mg^{2+} in dyeing effluent after EC with and without adsorbents, and details are given in Fig.2. It may be due to electronation of these cations at cathode and also adsorption of cations on the surface of CAC, MWCNTs and GR during electrocoagulation²⁵⁻²⁷.



The percentage removal of pollutants in dyeing effluent is higher in the case of EC with GR or MWCNTs than CAC. It can be attributed to good adsorption and due their conducting behavior. Since EC process is common in all the four treatment studies of dyeing effluent, the efficiency of treatment lies on the adsorbing capacity of the adsorbent material used during EC processes²⁷⁻²⁹. Due to the high conducting property of MWCNTs and GR, the surfaces of these materials are electron rich. These electrons act as a driving force for the attraction of cations to the surface of GR and MWCNTs. Hence they undergo adsorption more readily on the surface, but in the case of CAC the cations undergo adsorption rather slowly on its surface $^{28-31}$.

FT-IR Spectral Studies of Adsorbents Before and After EC Processes of Dyeing Industrial Effluent

FT-IR spectra were recorded for the adsorbent samples namely *viz.*, CAC, MWCNTs and GR before and after EC process of dyeing effluent. The characteristic functional group frequencies (wave number in cm⁻¹) were observed for various surface functional groups are shown Fig 3 and Table 6. The spectra of adsorbents before and after EC process are almost similar with slight differences in frequencies. The spectral values of adsorbents before and after EC process suggest that the adsorption of pollutants of dyeing effluent on the surface of adsorbent materials might be physisorption.



Figure 3 FT-IR spectra of adsorbents before and after EC of dyeing industrial effluent

 Table 6 FT-IR spectral values of adsorbents before and after EC of dyeing industrial effluent

Eunotional	CA	.C	MWC	CNTs	GR		
Groups	Before	After	Before	After	Before	After	
	EC	EC	EC	EC	EC	EC	
-OH	3315	3385	3315	3315	3315	3317	
-CH	2900	2900	2924	2923	2924	2924	
-CH ₂	1400	1387	1400	1387	1400	1400	
-C-O-C	1115	1115	1115	1117	1115	1117	
-C=O	1640	1650	1680	1700	1690	1695	



Figure 4 SEM images of A, C and E represent CAC, MWCNTs and GR respectively before EC processes: B, D and F represent CAC, MWCNTs and GR respectively after EC processes of dyeing effluent

Surface Morphological Study of Adsorbents Before and After EC

The surface morphology of adsorbents (CAC, MWCNTs and GR) were carried out using scanning electron microscope (SEM) and studies provide useful information regarding the textural morphological characteristics of the surface of the adsorbents. The typical SEM photographs of adsorbents, before and after EC of dyeing effluent are shown in Figure 4 A-F. SEM photographs clearly reveal the surface texture and porosity of the adsorbents. SEM photographs also show that the particles can be roughly approximate as spheres or globules, if the roughness factor is included to account for their regularities. SEM photographs also depict the porosity nature of the carbonaceous materials and the presence of grains in it. The effluent molecules are either engulfed or adsorbed on the surface porous adsorbent particles^{40, 41}.

The TGA studies were carried out to study the thermal stability of adsorbent materials before and after EC processes (Figure It is observed that all the three adsorbents viz., CAC, 5). MWCNTs and GR undergo two step decomposition patterns. In the first step of degradation (~0-200 C), a small fraction of weight loss occurred, which is due to the removal of water molecules/moisture that might be present in adsorbents^{32,33}. The second step of weight loss occurs in the temperature range of 200-700 C is due to the removal of the removal of labile oxygenated surface groups and carbon skeleton corresponding to the release of COx, H₂O and carbon combustion. It was seen that all adsorbent materials have a greater thermal stability after EC than before EC processes. This higher thermal stability of the all the adsorbent materials after EC correspond to the adsorption of suspended particles of dye effluents on the surface. It is also observed from the TGA curve that, the thermal stability of MWCNTs and GR are relatively higher than CAC in both before and after EC processes.



Figure 5 Thermogravimetric Studies (TGA) of Adsorbents Before and After EC Processes

CONCLUSION

The dyeing effluent samples were collected from industry located between Aruppukkottai and Kariyapatti near Madurai, Tamilnadu, India. Characterization, correlation analysis and treatment by EC process were carried out. The following conclusions are made from the results of present study:

- Most of the WQPs of dyeing effluents are found to be higher than the tolerance limit prescribed by BIS for industrial effluent.
- The WQI value of dyeing industrial effluent showed that it is contaminated eleven times higher than the prescribed limit.
- Correlation and linear regression were carried out in order to study the rapid monitoring of water pollution.
- The studies of quality of effluent for irrigation showed that it is not suitable for irrigation purposes as such.
- EC technique was employed for the treatment of dyeing effluent with and without adsorbents such as CAC, MWCNTs and GR. pH of the effluent is considerably reduced after EC with and without adsorbent. The decrease in values of TDS is higher in the case of EC with GR or MWCNTs than CAC and without adsorbent. The decrease in concentration of both anions and cations is relatively higher in EC with GR or MWCNTs than CAC without adsorbent and
- FTIR, TGA and SEM studies were made for adsorbents before and after EC processes and the SEM studies show that effluent molecules are either engulfed or adsorbed on the surface of porous adsorbent particles.

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