Efficient Removal of Coralene Red 3g Dye in Ec Process with Zn/Tio_2 using Statistical Algorithm

Parameswari Kalivel, Jegathambal Palanichamy, M.Vijila

Abstract — This work describes the statistical approach for the efficiency of electrodes Zn/TiO_2, prepared by spray pyrolysis and thermal decomposition of $TiCl_3$, in treating Coralene Red 3G dye for its color removal in electrocoagulation process with the optimization of operational parameters. Using Design Experts software, ANOVA design model was carried out and the efficiency of color removal for the parameters such as pH, concentration of dye, electrolyte, applied current and time were derived. Replicates were maintained for all experiments, and the results were statistically analyzed by performing ANOVA at 5% level of significance using SPSS software. A better efficiency was observed for the electrodes Zn/TiO_2 obtained from spray pyrolysis.

Keywords: Electrocoagulation, Coralene Red 3G dye, Zn/TiO_2, Analysis of Variance

I. INTRODUCTION

Advances in technology have resulted in increased demand for water from the various types of industries. The amount of water generated and discharged from these industries as waste has been growing rapidly, containing a wide variety of pollutants [1]. The effluent from various units in such textile hub have high levels of COD, color and salt. In treating the wastewater from these industries, simple methods like chemical coagulation causes more amounts of sludge and biological methods take much time and cannot degrade complicated dyes. Advanced oxidation methods are costly and uneconomical [2-4].

The electrochemical method is a better treatment method with high efficiency for treating textile wastewaters containing a high concentration of dye since these processes are mostly quite expensive and not effective [5]. Research related to wastewater treatment has focused on electrocoagulation [EC] over the past few years since it is simple in operation with less production of sludge [7]. EC has been successfully used for decades in order to treat the wastewaters of textile [8] food and protein [9] phosphate [10] tannery wastewater [11] restaurant wastewater and defluoridation [12]. Although standard electrode materials (Al and Fe) in EC process may have been excellent in color removal of the dye but likely to get dissolved and consumed more energy. The EC method's accounts for novel electrode tools in the literature remain highly inadequate.

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The conventional electrode materials like Al and Fe are more active in reducing color and can be dissolved [13]. Therefore, a modern, economically viable EC electrode or electrode / hybrid system, with less current and time is highly desirable. On continuation of our research works in this area, modified electrodes Zn/Ti^{2+} prepared from two different methods were compared for the color removal efficiency of Coralene Red 3G dye in EC [14].

II. EXPERIMENTAL METHODS

A. Preparation of Electrode

The electrodes Zn/TiO_2 were prepared by thermal decomposition and spray pyrolysis methods at 250°C using the same precursor. [15]

B. Synthetic Dye Solution

From one of the small textile dyeing industry in Coimbatore, Tamil Nadu, the disperse dye used in this work has been acquired (Commercial name; Coralene Red 3G,). Synthetic wastewater was prepared at concentrations of 15 0mg/L in distilled water.

C. Electrocoagulation (EC) Process

The electrocoagulation unit is set up as an electrochemical reactor with glass beaker along with magnetic stirrer and DC energy. Two sets of EC process were run Zn/TiO_2 – Zn/TiO_2 from spray pyrolysis [SP] and Zn/TiO_2 – Zn/TiO_2 from thermal decomposition (TD) (Hereafter these electrodes will be represented 'A' Spray pyrolysis (SP) Zn/TiO_2 – Zn/TiO_2) and 'B' (Thermal Decomposition Zn/TiO_2 – Zn/TiO_2) The electrode region of 28.16 cm^2 and a distance of 10mm between the two electrodes was used.

A sample solution of 250 ml was drawn in a 500 ml capacity beaker and a magnetic stirrer was used to stir the solution. Before each test, electrodes were washed with water and placed in 15% hydrochloric acid to remove dirt from the electrode plate. After each test, the electrodes were washed with water, dried and weighed. The subsequent processed sample was filtered and the filtrate was used for analysis. Individual impacts of electrolysis moment and applied current on the percentage of efficiency of extraction of color (EEC %) have been quantified in this research. All experiments at 25° C were performed.

Two sets of EC processes were conducted with ' A' and' B' electrodes with operating parameters such as pH, EC time, NaCl electrolyte and applied current were adopted for optimization to obtain greater color elimination.

D. Efficiency of Extraction of Color (EEC %)

The spectrophotometer JascoV-670 was used to determine the dye solution's wavelength (542 nm). Upon electrocoagulation, the following formula was used to measure EEC percent.

$$(EEC\ \%) = 100*(Ab-At)/Ab \quad (1)$$

Where Ab and At respectively where the dye's absorption in solution before electrocoagulation and at the moment t and to measure the pH of the solution pH meter is used (Eutech).

E. Structural Investigation and Surface Morphology

Micro structure of the particles present in the electrode was studied by X-Ray diffractometer [SHIMADZU-6000]. Scanning Electron Microscope (SEM) [JEOL 6390], was used to analyze the surface morphology. Elemental analysis was done by Energy Dispersive X-Ray Spectroscopy (EDS).

F. Statistical Analysis

The 2 Level ANOVA studies were carried out using the Design Expert software. The following table illustrates the results obtained after carrying out each experiment. Operational parameters like varying pH, concentration of dye, NaCl, current and time were analyzed for finding its efficiency of the each parameter for optimization. Imitates were maintained for all experiments, and the results were statistically analyzed by performing regression analysis at 5% level of significance using SPSS software [6]

III. RESULTS AND DISCUSSION

A. XRD Analysis of the Zn/TiO_2

The X-ray diffractogram of Zn/TiO_2 obtained from both the methods and zinc are shown in Fig 1. The diffraction peaks of TiO_2, and zinc are present in the investigated electrode. The electrode from spray pyrolysis method has more numbers of TiO_2 peaks than compared to the one obtained by thermal decomposition method. The planes corresponding to (002), (100), (101), (102), (103) are the peaks for zinc substrate. From the XRD results, deposition of TiO_2 is confirmed with 2theta values (30,32,35,47,56,63,68) and deposition of Zn with 2 theta values (36,43,54,70,78). Peaks (104), (110), (024) and (018) are the peaks of TiO_2 present in the electrode from thermal decomposition method, besides these peaks, four more peaks of TiO_2 are present in the electrode from spray pyrolysis method, and since with more TiO_2 peaks in this electrode showed better results in the percentage color removal of the dye under electrocoagulation study

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B. SEM and EDX Analysis

Fig 2 represents the SEM micrograph of zinc, Zn/TiO_2 from TD and SP methods. From the SEM micrographs, the TiO_2 particles are irregular in shape and appeared in an agglomerated state. The electrode by TD has been found with cracks, but from SP method crystalline particles are irregular in shape and appeared in an agglomerated state. The electrode by TD has been found with cracks, but from SP method crystalline particles are seen on zinc.

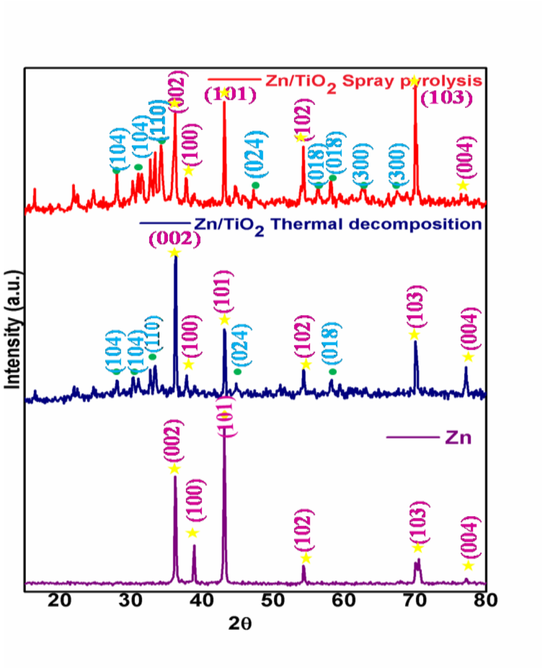

Fig. 1. XRD Pattern for Zn and TiO_2/Zn by TD, and SP

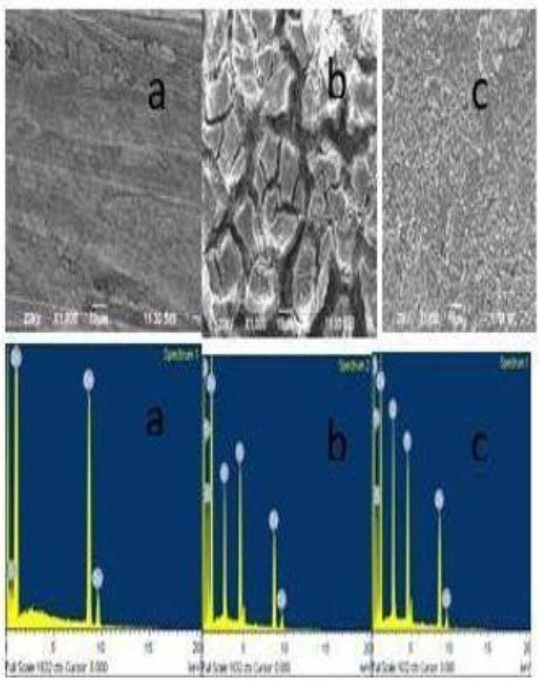

Fig 2 SEM Monograph (a) Zn, (b) Zn/TiO_2 (TD) (c) Zn/TiO_2 (SP)

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The coating of TiO_2 on zinc substrate has increased the surface roughness which in turn resulted in more contact area. And the increase in more contact area resulted in increasing the efficiency of dye removal in the case of Zn/TiO_2 by SP method in EC process. The percentage of elements in the uncoated and coated zinc substrate are done through EDS analysis and the percentage composition of elements in both the electrodes are given in Table 1 [16].

Table 1: Percentage composition of elements in the electrodes

Zn Substrate		*Zn/TiO₂ (TD)*		*Zn/TiO₂ (SP)*
Element	*Weight (%)*	*Element*	*Weight (%)*	*Weight (%)*
O	7.27	O	30.70	59.81
Zn	92.23	Cl	11.01	9.68
		Ti	10.14	15.58
		Zn	42.71	20.37

The results obtained were analyzed using ANOVA (Analysis of variance) and the following results were accomplished for adsorption efficiency using TiO_2/Zn (TD) and TiO_2/Zn electrodes (SP) [17].

The F-value of 3.625 obtained from the model implies that, it is significant at 5% level.

Mean 72.3875 Adj R^2 0.658

St. Dev. 25.7597 R^2 0.689

The r^2 value comparing pH to Dye was 0.689 (significant at $P < 0.05$). In the present study, this method was used to develop the effectiveness of absorption of the dye in solution for all the parameters like varying pH, concentration of dye, NaCl, current and time. The "Pred R-Squared" of 0.689 agrees with the "Adj RSquared" of 0.658.

This model can be used to find the efficiency of adsorption of the dye with the flocs the metal hydroxides formed from both the anode and cathode during EC process .The result shows slight deviation from the fitted non-linear equation of Langmuir model as indicated by the high value of R^2 ranging from 0.658 to 0.689. The highest R^2 value was obtained at 25°C [18]. The values of (1/n) obtained were in the range 0 and 1 thus indicating the favorable nature of the process and heterogeneous nature of the adsorbent surface. Therefore, the linear regression model is also well fitted using the data. Hence, it can be concluded that TiO_2/Zn is indeed releases more of $Ti(OH)_3$ which adsorb the dye molecules, using design of experiment in two factor model [19].

From the table 2, the electrodes Zn/TiO_2 (SP) has more consistency than Zn/TiO_2 (TD) in treating the Coralene Red 3G dye for its color removal in electrocoagulation process.

Table 2: Effect of varying parameter on EEC%

Zn/TiO₂ (SP) EEC % with varying					Zn/TiO₂ (TD) EEC % with varying				
pH	*Dye Concentration*	*NaCl Concentration*	*Applied Current*	*Time*	*pH*	*Dye Concentration*	*NaCl Concentration*	*Applied Current*	*Time*
90.56	94.47	96.83	78.40	90.27	10.7	88.2	35.9	12.9	87.3
94.82	82.54	97.44	87.39	96.23	12.9	88.4	97.1	22.1	92.8
92.95	79.27	82.93	97.32	96.01	91.7	98.6	98.0	99.5	97.2
95.60	64.13	87.28	87.39	95.14	93.9	92.3	98.2	98.6	93.1
95.32	56.40	82.24	85.46	93.89	98.1	92	98.2	98.0	92.9
87.95	48.77	79.12	81.06	94.12	73.6	84.2	97.6	93.8	88.7

The results obtained were analyzed using ANOVA (Analysis of Variance) and the following results were obtained for adsorption capacity:

Table 3: ANOVA Analysis Table for Electrodes.

Source	*Sum of Squares*	*df*	*Square of Mean*	*F Value*	*P Value Prob.>F*	*Significant @ 5%*
For All	6426.3	4	1606.59	3.652	0.01040	< 0.05
A-pH	5032.7	1	5032.755	22.20	0.00082	< 0.05
B-Dye	2007.2	1	2007.253	3.145	0.10652	
C-NaCl	2206.4	1	2206.483	5.506	0.04088	< 0.05
D-Current	15.962	1	15.96213	0.594	0.45855	
E-Time	674.70	1	674.7000	5.067	0.04809	< 0.05
AB	1242.57	1	1242.57	1.742	0.20038	
AC	17.18	1	17.18	0.0279	0.868679	
AD	956.97	1	956.97	2.7761	0.109855	
AE	1242.57	1	1242.57	2.9377	0.100589	

The F-value of 22.20 obtained from the model implies it's significant [20]. There is a 0.05% chance that a "Model F-Value" this large could happen due to the presence of more of T_i^{3+}.

IV. CONCLUSION

In conclusion, the percentage of dye removal has a linear relationship with pH, amount of adsorbent and concentration of dye with current and time and there is also a significant interaction between amount of adsorbent and concentration of dye. The electrodes Zn/TiO_2 from spray pyrolysis has more efficiency than the one from thermal decomposition in treating the Coralene Red 3G dye for its color removal in electrocoagulation process because of more number of TiO_2 peaks which in turn formed more $Ti(OH)_3$ and adsorption of dye with these flocs resulted in increasing the coagulation of dye molecules, so better removal of disperse dye.

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