

Wastewater Containing Synthetic Organic Dyes: Decontamination by Chicken Feather Films

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Abstract- Chicken feathers were hydrolyzed by using diluted alkaline solution (NaOH) and free amino groups were treated by the process dithiocarbamylation by carbon disulphide. The resultant product was moulded into eco friendly films in the presence of polyvinyl alcohol, glutaraldehyde and glycerol. These films were examined by atomic force and scanning electron microscopies, FTIR spectroscopy, powder X-ray diffractometry, and differential scanning calorimetry. The films were found to be an efficient adsorbent of remove methylene blue (MB) and crystal violet (CV) from their aqueous solutions. The adsorption mechanism was analyzed by applying various kinetic models, namely pseudo-first order, pseudo-second order, Elovich, and intraparticle diffusion. Freundlich, Langmuir, Temkin and Dubinin-Kaganer-Radhuskevich isotherm models were applied. The results show that films derived from chicken feathers are highly effective in water decontamination. Maximum adsorption capacity was found to be 234.011 mg/g at 305 K for methylene blue and 113.636 mg/g at 285 K for crystal violet.

Keyword: Chicken feather, dye adsorption, dithiocarbamylation, crystal violet, methylene blue.

I. INTRODUCTION

Many industries, such as textile, cosmetic, paper, leather, light-harvesting arrays, food, agricultural research, pharmaceutical and photoelectrochemical cells [1], produce large volumes of wastewater polluted with high concentration of dyes and other components. Public perception for water quality recognizes colour to be first contaminant in wastewater [2]. A range of classes of dyes is used in textile industry including disperse, reactive, acid, basic, direct, azoic, sulfur, and direct dyes of them, vat, azoic, sulfur, and disperse dyes are insoluble in water and therefore easy to separate from the effluent [3]. On the other hand, reactive, direct, basic and acid dyes are highly soluble in water and therefore, it is difficult to remove them from effluent by conventional separation processes [4]. Some of the synthetic dyes are accompanied with life threatening factors due to their eminent toxic, and even carcinogenic, dominance [5]. The contamination of natural waters with dyes produces modification in biological cycles affecting mainly photosynthesis process [6]. The regular wastewater treatment works cannot demean most of these organic pollutants [7]; hence, there have been significant efforts in researching alternate methods of wastewater decontamination. Besides adsorption [8], other techniques such as chemical oxidation, photocatalytic decolourization [9], biological treatment [10], coagulation, electrochemical coagulation [11], settling and filtration are on demand for removal of dyes from effluents. However, adsorption is the most commodious process; hence, efficient, economical and easily approachable adsorbents are being explored [12].

The by-products from the agriculture and food industries could be assumed to be low-cost adsorbents since they are abundant in nature, cheap, require little processing and are effective materials [13]. Attention of scientific community is currently focused on various natural solid supports, which are able to remove pollutants from contaminated water at low cost [14]. Cost constitutes an important parameter for comparing the adsorbent materials. Chicken feathers were accounted to be capable adsorbents [15]. The discarding of chicken feathers (CF) raised a serious environmental menace with the zoom of poultry industry. CF are mostly disposed in landfill and sometimes incinerated or composted. However, quick prior treatment is necessary due to a multitudinous of microflora, including pathogens; cling to feathers before disposal [16]. Disposal by incineration requires high energy and generates high carbon footprint. Composting is a prolonged process and requires periodic microbiological tests and veterinary inspection [17]. Though CF are a prolonged resource of keratin, their utilization is limited by poor digestibility and minimum biological value [18]. Feathers are difficult to hydrolyze/degrade because they composed of 90% of keratin proteins that are tightly packed either in α -helices or β -sheets into keratin super coiled polypeptides [19]. CF was chemically modified with aqueous sodium hydroxide (NaOH) and treated by carbon disulphide for the process dithiocarbamylation. Dithiocarbamylated CF was molded into films by mixing with different amounts of polyvinyl alcohol (PVA) and glycerol in the presence of HCl and glutaraldehyde. These films were characterized using

Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) and differential scanning calorimetry (DSC).

II. EXPERIMENTAL

A. Materials and methods

Methylene blue, crystal violet or gentian violet and other chemicals of standard grade received from Merck, India were used without further purification. Deionized water (DIW) inclined by Millipore water purification system model Elix-3 was used in all the experiments. Chicken feathers were brought from poultry farm. Raw CF were exhaustively perturbed with distilled water, washed several times with DIW, then dried in hot air oven at 60 °C for 24 h, after that cut with the help of scissors and hard middle rachis was removed and discarded. The barbs of about 1-2 cm of length were cut into modest pieces and loaded in aqueous NaOH for 24 h, washed exhaustively with water to remove adhered dirt and other materials, and then dried. 1g CF and 100 mL of 2% aq. NaOH were magnetically stirred in a flask for 48 h at 10°C. 2mL of CS₂ were added by dropping funnel. Solution turned orange in colour after 2 h and then it turned yellow. After 2 days again added 2ml CS₂ in the solution then it tuned to light green in colour. This dispersion was mixed with PVA, glycerol and HCl in different proportions to optimize film preparation. Contents were uniformly mixed by magnetic stirring and followed by ultrasonication for 30 min, then before pouring the whole content into the petridish, glutaraldehyde was mixed and kept in hot air oven at 60 °C for 1 h 30 minutes to prepare films, to be attributed as CFDTTC hereafter.

B. Characterization

FTIR spectra were recorded on a Shimadzu FTIR spectrophotometer model 8400 S as diffused reflectance spectra (DRS) using KBr as dispersive medium. Energies of hydrogen bonds (E_H) were calculated with the Eq. 1:

$$E_H = (1/k) \cdot (v_0 - v) / v_0 \quad \dots (1)$$

where, v_0 (3600 cm⁻¹) and v are frequencies corresponding to the free and bonded -OH groups, respectively and $k = 1.68 \times 10^{-2}$ kcal⁻¹ [20].

Wide-angle powder X-ray diffraction data were collected using a Rigaku X-ray diffractometer, model MiniFlex 600 equipped with Cu K α radiation at 30 kV and 15 mA in the range $2\theta = 3-80^\circ$, at a rate of 3° per min. Scherrer equation was used to calculate the average size of the crystallites [21]:

$$D_{hkl} = K\lambda / (\beta_{hkl} \cdot \cos\theta) \quad \dots (2)$$

Where D_{hkl} = crystal dimension perpendicular to the diffracting planes with Miller indices of hkl , K = the Scherrer constant, λ = wavelength of radiation (1.5406Å for K α), and β_{hkl} = full width at half maximum (FWHM) of the diffraction peaks. Segal's empirical method [22] was used to evaluate crystallinity index (I_c) Eq. 3:

$$I_c = (I_{200} - I_{am}) / I_{200} \quad \dots (3)$$

Where I_{200} = peak intensity at plane (2 0 0) ($2\theta = 22.6^\circ$), and I_{am} = minimum intensity at the valley between plane (2 0 0) and (1 1 0) ($2\theta = 18.7^\circ$). I_{200} represents both crystalline and amorphous material and I_{am} amorphous material [23]. Inter planar distance (d) was calculated using Bragg's equation:

$$2d \sin \theta = n\lambda \quad \dots (4)$$

Where n = positive integer and λ = wavelength of radiation.

Field emission gun scanning electron microscope (FEGSEM) of FEI Ltd. (Nova Nano SEM 450) with an accelerating voltage of 20 V-30 kV was used to analyze surface morphology of CFDTTC coated with gold using sputtering technique. Film surface topography was analyzed by AFM images obtained by using Veeco nanoscope IV multimode AFM. Differential scanning calorimetric analysis was carried out on an instrument 2910 MDSC V4.4E in the 25 - 150 °C range at a heating rate of 10 °C under nitrogen atmosphere. Antimicrobial activity of CFDTTC film was examined by the method of zone of inhibition [24]. Escherichia coli NCIM 2256 and Staphylococcus aureus NCIM 2079 were cultured on a nutrient agar plate supplemented with a circular piece of test film. The plate was examined for a possible zone of inhibition after incubation at 37 °C for a period of 24 h.

C. Adsorption studies

Adsorption studies with CFDTTC films were carried out by batch technique at 285, 295 and 305 K. A series of 50 mL boiling test tube containing 20 mL solutions of methylene blue or crystal violet in varying concentrations and required amount of the CFDTTC films were employed at desired pH and temperatures. Concentration of the dye in the solution after a definite interval of time was determined spectrophotometrically by measuring the absorbance of methylene blue and crystal violet at λ_{max} of 665 and 570 nm, respectively using a Shimadzu spectrophotometer Pharma Spec UV-1700. The % removal and the amount of adsorbed dyes were calculated by Eq. 5 and 6:

$$q_e = \frac{C_0 - C_e}{W} \times V \quad \dots (5)$$

$$\% \text{ Removal} = \frac{C_0 - C_e}{C_0} \times 100 \quad \dots (6)$$

Where, q_e is the amount of dyes adsorbed onto unit amount of the CFDTTC (mg g⁻¹), C_0 and C_e are initial and final dye concentrations in the solution, respectively. V (L) is volume of aqueous phase and W (g) is weight of CFDTTC.

III. RESULTS AND DISCUSSION

Alkali was chosen over enzymatic or acidic hydrolysis to achieve low cost and simple hydrolysis of CF. Dithiocarbamylation with CS₂ of partly hydrolyzed CF was taken place. DTCCF was mixed with glycerol, PVA, glutaraldehyde and HCl in various proportions. PVA was

used for film constitution as it has polymerisation tendency. Glycerol is a good plasticizer, glutaraldehyde act as a cross linker and HCl as a catalyst for cross linking. Out of the several experiments to find optimum proportion of various

constituents, films showing satisfactory adsorption behaviour are presented in Table 1. The film with low proportion of PVA (CFDTC2) has low density and high thickness.

Table 1: Composition and characteristics of CFDTC films

S. No.	Film Code	Amount					Density	Average thickness
		CFDTC	PVA	Glycerol	Glutaraldehyde	0.1 N HCL		
		g	g	mL	mL	mL	g/cm ³	Mm
1	CFDTC1	0.7	0.3	1	0.5	0.25	1.112	0.205
2	CFDTC2	0.9	0.2	1	0.5	0.25	1.032	0.563

FTIR spectroscopy of PVA, CF and various films are depicted in Figure 2. CFDTC1 and 2 showed characteristic absorptions from CF as well as PVA. As equate to PVA, absorption due to amide I from CF is well manifest around 1600 cm⁻¹ in the films. Strong and broad bands in 3200–3500 cm⁻¹ range are ascertained in all the films, which are associated with the stretching vibrations of N-H of amide bonds from CF and O-H from PVA. Further, absorption convention is similar to that of CF in both the films below 750 cm⁻¹.

XRD patterns of films CFDTC were compared with their components CF and PVA (Figure 3). PVA shows a wide maximum centered around 2θ 20°, whereas, CF exhibits two peaks near 2θ 10° and 20°. For CFDTC films crystallinity is somehow greater than their components. Both the films show maximum centered around 2θ 20°. Crystallinity increases with increasing amount of PVA. DSC study in the temperature range -50 to 150 °C showed melting endotherms at 99.5 °C for CFDTC2 (Figure 4). Morphology of CFDTC1 and CFDTC2 were examined by SEM images (Figure 5). CFDTC1 has a slightly rough morphology, whereas CFDTC2 shows phase separation.

AFM analysis (Figure 6) indicates that the surface roughness of the films increases with increasing ratio of CFDTC, which is further substantiated by RMS roughness data, which is 107.018 and 120.851 for CFDTC 1 and CFDTC 2 respectively.

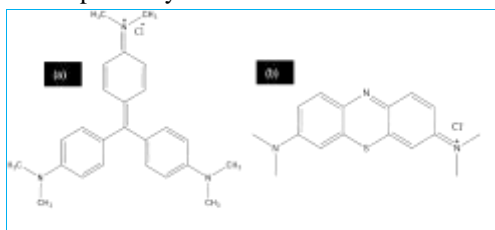


Figure 1: Structures of (a) Crystal violet and (b) Methylene blue

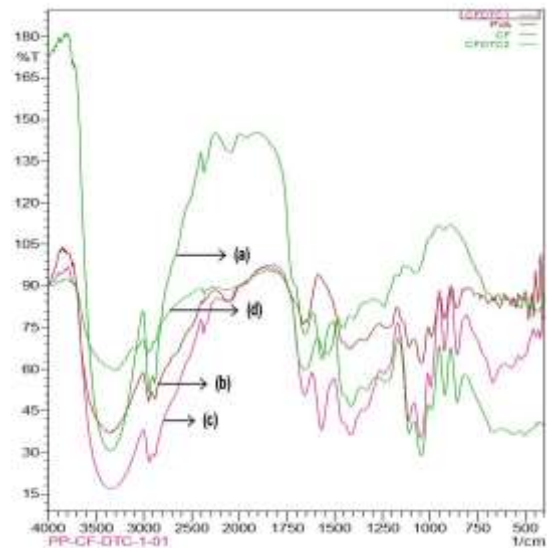


Figure 2: FTIR spectra of (a) CF, (b) PVA, (c) CFDTC1 and (d) CFDTC2

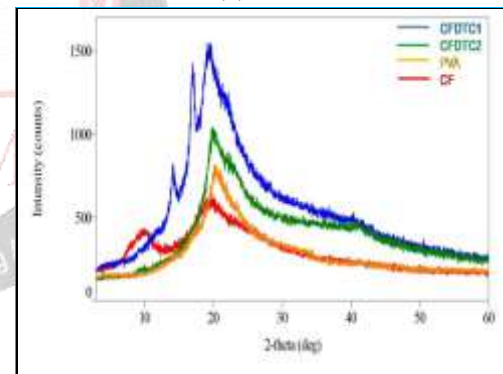


Figure 3: XRD graph of CF, PVA and CFDTC films

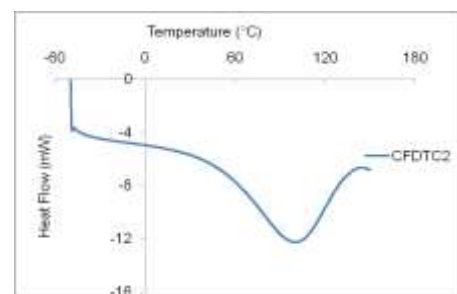


Figure 4: DSC thermogram of CFDTC2

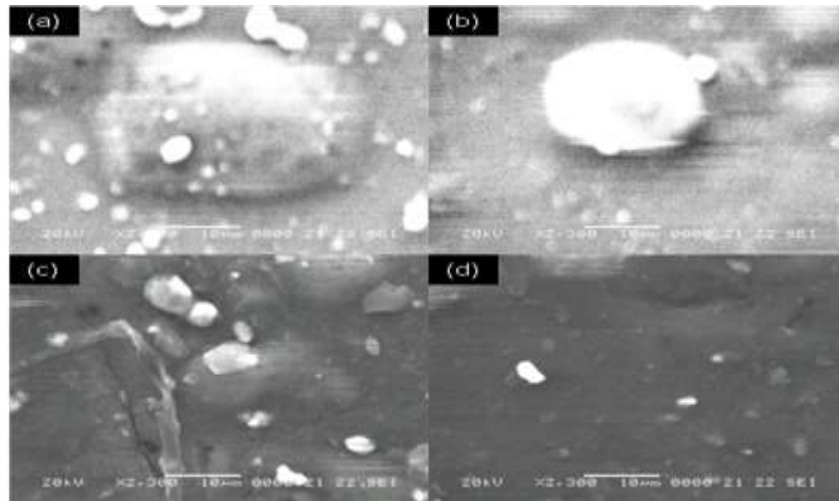


Figure 5: SEM images of CFDTC1 (a) & (b) and CFDTC2 (c) & (d)

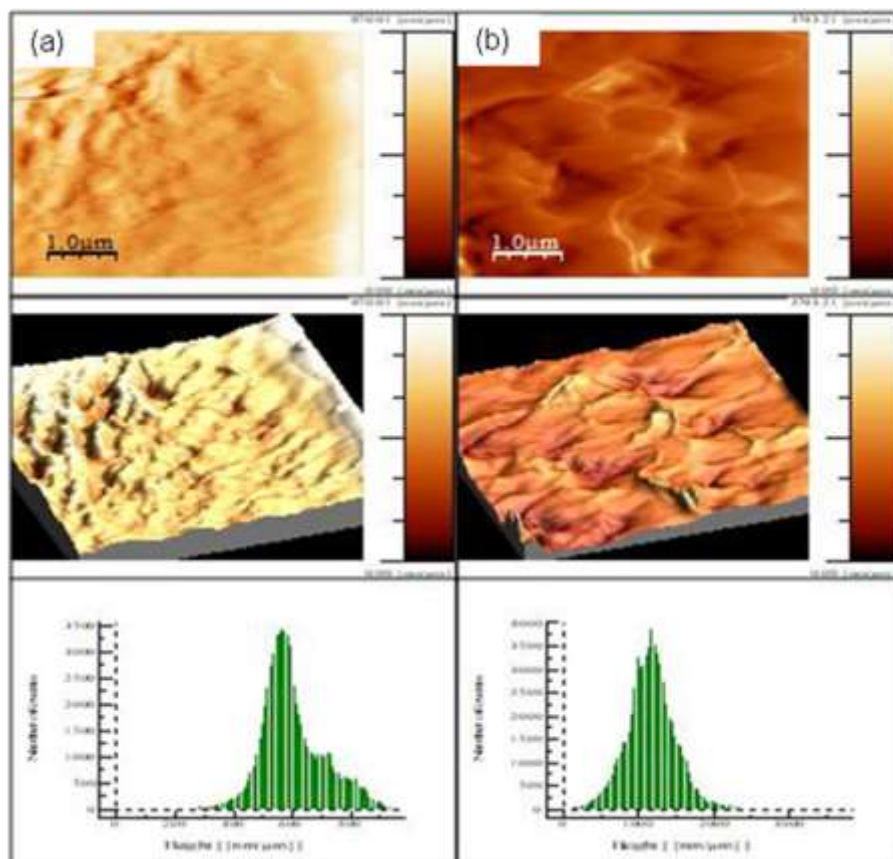


Figure 6: AFM images of (a) CFDTC1 and (b) CFDTC2- top:2D and middle:3D images, bottom:surface roughness.

A. Adsorption studies

Crystal violet or gentian violet (tris(4-(dimethylamino)phenyl)methyl) chloride), a triarylmethane dye and methylene blue, also known as methylthionium chloride, a thiazine dye were used as models to study adsorption function of CFDTC films. Crystal violet and Methylene blue removal by CFDTC film was carried out in the pH range 4, 7 and 9. It was found that the adsorption somehow decreasing with increasing pH of the solution.

Adsorption kinetics is determined to optimize operating

conditions for batch procedure. Mechanism of adsorption process was studied by different theoretical models and consequences are represented in Figure 7 and Tables 2 and 3.

Eq. 8 gives the pseudo- first order equation of Lagergren and pseudo-second order model is constituted by Eq. 9:

$$\log(q_e/q_t) = \log q_e - k_1/2.303 \times t \quad \dots(8)$$

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad \dots(9)$$

where q_t and q_e are adsorption capacities at time t and at equilibrium, respectively, k_1 is first order rate constant, k_2 is second order rate constant ($g \text{ mg}^{-1} \text{ min}^{-1}$). Following formula represents Elovich model [25],

$$q_t = (1/\beta)\ln\alpha\beta + (1/\beta)\ln t \quad \dots(10)$$

where, α and β are initial rate of adsorption and desorption constant protectorates with energy of activation and magnitude of surface coverage for chemisorptions, respectively, which were dectated from the slope and intercept of q_t versus $\ln t$ plot (Figure 7 c). R^2 values are low, except for low concentration of crystal violet (10 mg/L) and at pH 9, which shows that in all other examples dye removal using CFDTC film cannot be represented by Elovich kinetic model.

Weber-Morris model is used to further elucidate adsorption kinetics on the adsorbent and is stated by Eq.

11 [26]:

$$q_t = k_{id}t^{1/2} + C_i \quad \dots (11)$$

Where, k_{id} = intraparticle diffusion rate constant. A pattern between amount of dyes adsorbed and \sqrt{t} was plotted to find out probable adsorbate transport into pores of adsorbent and results to are presented in Figures 8 (d) for different initial concentrations. Slopes of the graphs conceded k_{id} . Best agreement was ascertained for intraparticle diffusion except for crystal violet and methylene blue at 10 and 30 mg/L initial concentrations, respectively. For former Elovich and for latter pseudo-second order models were applicable.

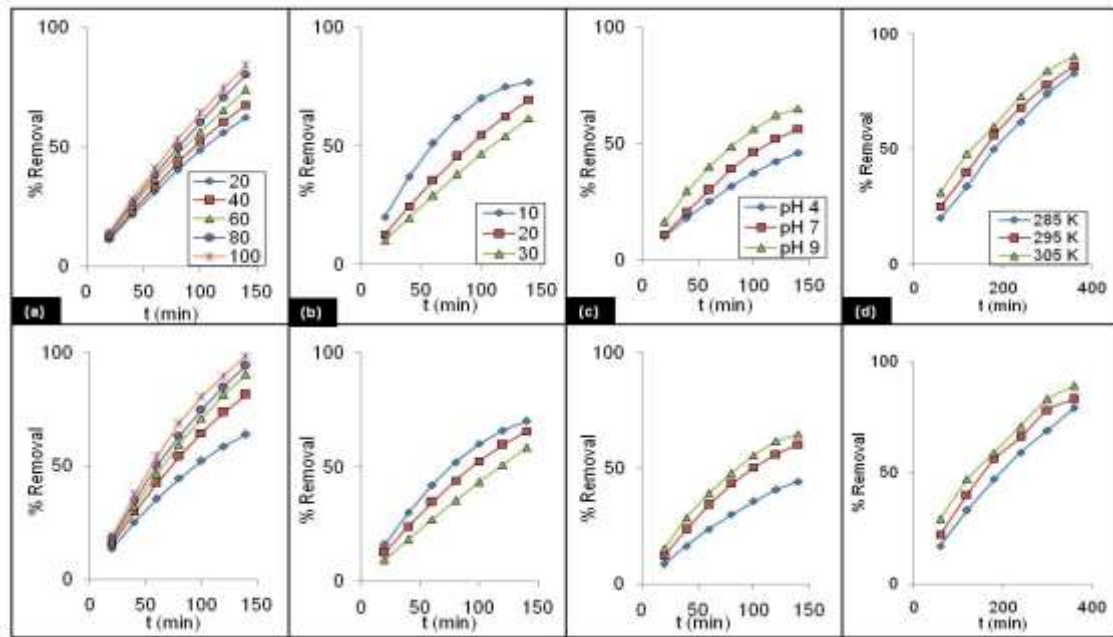


Figure 7: Effect of various parameters on adsorption of crystal violet (upper row) and methylene blue (lower row) on CFDTC (a) adsorbent dose (mg), (b) adsorbate dose (mg/L), (c) solution pH and (d) temperature (K)

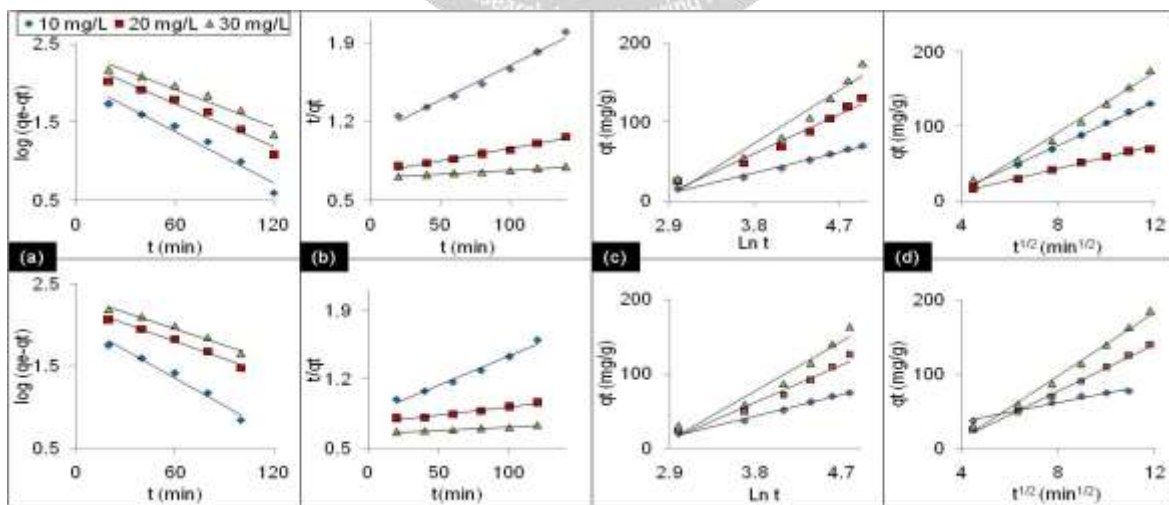


Figure 8: Curve fitting for adsorption of crystal violet (upper row) and methylene blue (lower row) into kinetic models (a) pseudo-first-order, (b) pseudo-second-order, (c) Elovich, and (d) Intraparticle diffusion

B. Adsorption isotherms

The adsorption Mechanism was ascertained by applying dye adsorption data into Langmuir, Freundlich, Temkin

and Dubinin-Kaganer-Radushkevich (D-K-R) isotherm models and results are represented in Figure 8 and Table 4. Linearized form of Langmuir equation is given as follows [27]:

$$1/q_e = (1/q_m) + (1/K_L q_m C_e) \quad \dots (12)$$

Where, q_e = adsorption capacity at equilibrium; C_e = concentration of dye at equilibrium and q_m = monolayer adsorption capacity. K_L = Langmuir constant. R_L , the separation factor is dimensionless equilibrium parameter, is expressed as

$$R_L = 1/[1 + q_m(C_0)] \quad \dots (13)$$

Freundlich isotherm is uttered by the Eq. 13 or as the logarithmic form of linearized Freundlich equation (Eq. 14) [28]:

$$q_e = K_F C_e^{1/n} \quad \dots (14)$$

$$\ln q_e = \ln K_F + (1/n) \ln C_e \quad \dots (15)$$

K_F indicates adsorption capacity and n adsorption intensity. Value of n in Freundlich equation is declarative of favourability of adsorption.

Temkin isotherm was used to examine adsorption using Eq. 15 [29].

$$q_e = B \ln A_T + B \ln C_e \quad \dots (16)$$

B = constant related to heat of adsorption ($B=RT/b_T$, where R = universal gas constant, T = temperature (K) and b_T = Temkin isotherm constant). A_T is Temkin isotherm equilibrium binding constant. Slope and intercept of the plot of q_e and $\ln C_e$ give values of B and A_T .

Quality of adsorption process, characteristic porosity of adsorbent and evident energy is predicted by Dubinin-Kaganer - Radhuskevich (DKR) adsorption isotherm [30], [31]. Linearized form of the DKR model is represented as Eq. 16:

$$\ln q_e = \ln q_s - \beta \epsilon^2 \quad \dots (17)$$

q_e =equilibrium amount of adsorbate; q_s = saturation capacity of theoretical isotherm; β = activity coefficient for mean adsorption energy and ϵ = Polayni potential calculated by equation:

$$\epsilon = RT \times \ln(1 + 1/C_e) \quad \dots (18)$$

Adsorption potential (E) of adsorbate can be computed by Eq. 18:

$$E = 1/\sqrt{-2\beta} \quad \dots (19)$$

E is dependent on the nature of the adsorbent and adsorbate but independent of the temperature. Values of E in 8 - 16 kJ/mol range indicate the chemical ion exchange and physical nature of adsorption is shown by E less than 8 kJ/mol. Values of E were calculated to be in range 0.500-1.118 kJ/mol showing the physical nature of adsorption.

The graphs demonstrate good agreements for Freundlich and Langmuir models, slight divergence for Temkin model and no agreement with D-K-R model. The Freundlich model indicates multilayer adsorption and surface heterogeneity of the adsorbent, whereas Langmuir model suggests surface homogeneity of the adsorbent. Correspondence with these models leads to the conclusion that the surface of CFDTC2 is made up of small heterogeneous adsorption patches, which are very much similar to each other in respect of adsorption phenomenon. R_L is between 0.0012 and 0.006 suggesting favourable adsorption process. Values of Freundlich constant (n) were computed to be in the range 1.1-1.28, i.e., moderately difficult [24].

IV. CONCLUSION

CFDTC2 the adsorbent films prepared from chicken feathers were used to investigate adsorption of methylene blue and crystal violet. Intraparticle diffusion was found to be rate restricting factor for the adsorption of crystal violet and methylene blue. Exothermic adsorption for both dyes was inferred. Adsorption isotherm studies indicated that though the surface of CFDTC2 is heterogeneous, it consists of small patches, which are very much similar to each other in respect of adsorption phenomenon. This inference was drawn on the basis of agreement with Freundlich and Langmuir models both. CFDTC2 was found to be a quit promising adsorbent for treatment of wastewater polluted by dye effluents under ambient conditions of temperature and pH.

TABLE 2: Constants and correlation coefficients for crystal violet for various adsorption kinetics models

Kinetic model	Parameters	Initial crystal violet concentrations (mg/L)			pH of the solution		
		10	15	20	4	7	9
Pseudo-first order	k_1 (min ⁻¹)	0.026	0.017	0.0152	0.015	0.016	0.018
	q_e (mg/g)	107.920	169.903	226.100	69.823	94.733	99.747
	R^2	0.976	0.981	0.974	0.991	0.983	0.994
Pseudo-second order	q_e (mg/g)	168.35	617.28	1492.53	166.67	416.67	196.08
	k_2 (g mg ⁻¹ min ⁻¹)	4.1×10^{-4}	3.5×10^{-5}	7.0×10^{-6}	2.7×10^{-4}	4.0×10^{-5}	3.2×10^{-4}
	R^2	0.981	0.981	0.983	0.987	0.972	0.999
Elovich Model	β (g/mg)	0.032	0.018	0.014	0.043	0.032	0.030
	α (mg/(g min))	2.781	3.800	4.710	1.923	2.251	3.009
	R^2	0.992	0.965	0.951	0.97	0.966	0.985
Intraparticle diffusion	k_{id} (mg g ⁻¹ min ^{-1/2})	6.382	15.740	21.360	6.9051	9.147	9.7988
	R^2	0.978	0.997	0.992	0.997	0.995	1.000

Table 3: Constant and correlation coefficients for methylene blue for various adsorption kinetics models

Kinetic model	Parameters	Initial methylene blue concentrations (mg/L)			pH of the solution		
		10	20	30	4	7	9
Pseudo-first order	k_1 (min ⁻¹)	0.025	0.021	0.018	0.022	0.023	0.026
	q_e (mg/g)	109.144	192.442	248.886	94.406	131.978	141.547
	R^2	0.955	0.952	0.942	0.942	0.957	0.937
Pseudo-second order	q_e (mg/g)	162.075	452.489	1400.992	208.33	256.41	193.05
	k_2 (g mg ⁻¹ min ⁻¹)	3.5×10^{-4}	6.6×10^{-5}	7.0×10^{-6}	1.4×10^{-4}	1.4×10^{-4}	3.1×10^{-4}
	R^2	0.981	0.987	0.998	0.984	0.946	0.983
Elovich Model	β (g/mg)	0.035	0.018	0.013	0.0381	0.0277	0.0284
	α (mg/(g min))	2.258	3.707	4.440	1.771	2.538	2.818
	R^2	0.987	0.969	0.943	0.97	0.981	0.988
Intraparticle diffusion	k_{id} (mg g ⁻¹ min ^{-1/2})	7.554	14.71	20.15	6.975	9.526	9.237
	R^2	0.995	0.998	0.990	0.998	0.997	0.994

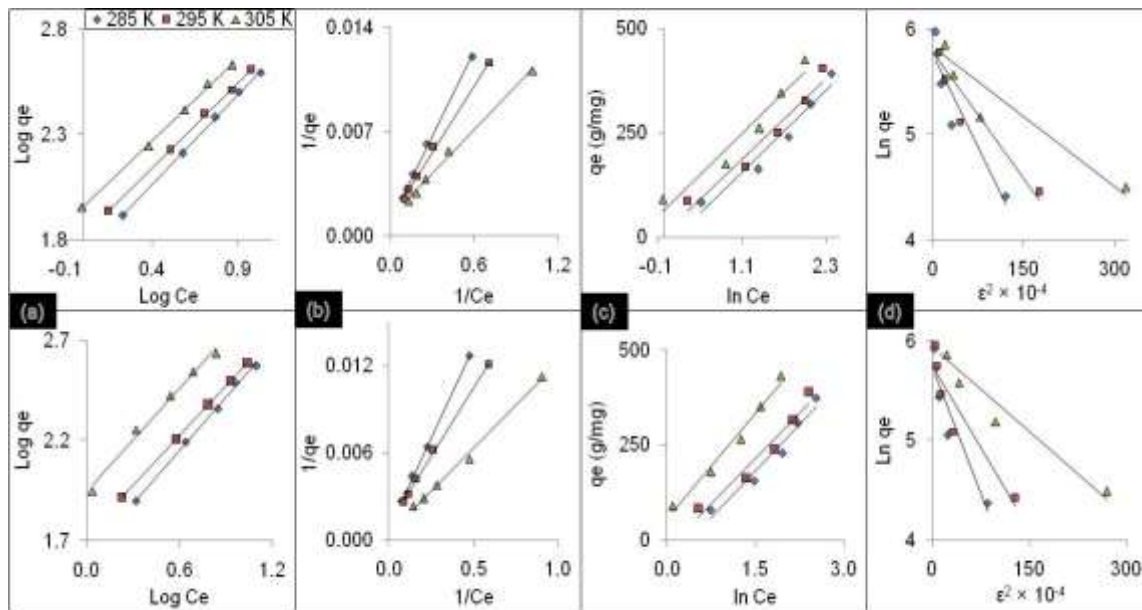


Figure 9: Adsorption isotherms for crystal violet (upper row) and methylene blue (lower row) adsorption (a) Freundlich (b) Langmuir (c) Temkin (d) Dubinin -Kaganer-Raduskevich

Table 4: Constant and correlation coefficients for various adsorption isotherm models

Isotherm Model	Parameters	Methylene blue			Crystal violet		
		285 K	295 K	305 K	285 K	295 K	305 K
Freundlich	K_F (mg g ⁻¹) (L mg ⁻¹) ^{1/n}	41.591	53.580	87.498	52.723	65.917	90.782
	n	1.138	1.215	1.166	1.176	1.245	1.282
	R^2	0.997	0.999	0.989	0.999	0.999	0.998
Langmuir	q_{max} (mg g ⁻¹)	151.0	97.1	234.011	113.636	93.458	82.645
	K_L (L mg ⁻¹)	38.08	18.31	27.37	21.75	13.91	8.09
	R_L	0.0012	0.0025	0.0017	0.002	0.003	0.006
Temkin	R^2	0.999	0.998	0.995	0.998	0.999	0.995
	B (J/mol)	164.000	159.100	185.600	165.500	161.300	164.700
	$\ln A$	-0.390	-0.156	0.294	-0.176	0.050	0.401
Dubinin-Kaganer-Raduskevich	A (L mg ⁻¹)	0.677	0.856	1.342	0.839	1.051	1.494
	Bt	14.245	15.207	13.483	14.116	14.999	15.194
	R^2	0.957	0.950	0.982	0.953	0.955	0.947
Dubinin-Kaganer-Raduskevich	B (mol ² /kJ ²)	2.0×10^{-5}	1.0×10^{-5}	6.0×10^{-6}	5.0×10^{-5}	3.0×10^{-5}	1.0×10^{-5}
	Q_m (mg g ⁻¹)	309.20	312.93	378.79	318.30	325.70	342.06
	E (kJ mol ⁻¹)	0.500	0.707	0.913	0.707	0.791	1.118
	R^2	0.884	0.871	0.934	0.872	0.875	0.866

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