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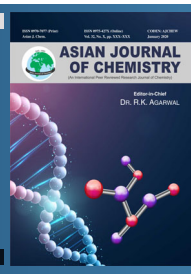

Isotherm, Kinetics and Thermodynamic Study of Adsorption of Phthalocyanine and Azo Dyes by $CoCl_2$ Doped Polyaniline

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In this work, $CoCl_2$ doped polyaniline was prepared using chemical oxidation method and used as an effective adsorbent for the removal of industrial dyes reactive blue 21 and reactive red 180. The adsorption experiments were performed with the variation of initial concentrations of dyes, pH of the solution, weight of adsorbent, temperature and agitation speed. Isotherm analysis, evaluating the correlation coefficients by ANOVA technique, kinetic study and calculation of thermodynamic parameters are carried out. Furthermore, the adsorption density of dyes onto PANI-$CoCl_2$ adsorbent is characterized by SEM and FTIR. This study is compared with other previous works to establish that PANI-$CoCl_2$ is an efficient adsorbent to remove dye from aqueous solution.

Keywords: Industrial dye, Polyaniline, SEM, Adsorption, Degradation.

INTRODUCTION

Water covers most of the world and makes up more of the world than land. Pure water is the world's first and foremost medicine which polluted through many sources such as urban runoff, agricultural, industrial, sedimentary, leaching from landfills, animal wastes and other human activities. Water pollution has become a continuous increasing problem on the earth and the textile industry is one among the biggest polluters of clean water in India. As a result, the dye has become common industrial environmental pollutant during their synthesis and later on its usage like fiber dyeing. The turbidity and toxic elements of effluent water from the textile industry have detrimental effects to humans, animals and make the potable water sources unfit for consumption in the nearby areas, whereas an increase in the pH causes incrustation in the sewers and may also damage crops by hampering their growth rate [1,2].

To maintain the utilizability of water quality various techniques based on physical, chemical, electrical and biological processes were developed for the elimination of dye from effluents and to decrease their impact on the environment, but these processes are generally expensive and require special treatments to carry out [3,4]. Among the above-mentioned methods, adsorption being a simple, non-toxic and a low-cost method for dye removal from wastewaters [5,6]. Many scientific workers have investigated the feasibility of low-cost adsorbent such as coconut husk [7], orange peel [8], wheat bran [9], sawdust [10], spent tea leaves [11], *etc*. for the removal of pollutants from aqueous solutions. Apart from these adsorbents conducting polymers gains attention due to its excellent environmental stability and high capacity in the removal of dyes from water. The common feature of most electrically conducting polymers is the presence of an extended *p*-conjugation system with single- and double-bond alteration along the polymer backbone [12,13]. In the past works, $CoCl_2$ doped polyaniline is mainly used as supercapacitors, but in the current study, we report that PANI-$CoCl_2$ is an effective adsorbent for the removal of colour from aqueous solution. The synthesis of PANI-$CoCl_2$ is based on the chemical oxidative polymerization of aniline in a strongly acidic environment, with ammonium peroxodisulfate as oxidant and $CoCl_2$ as a dopant to enhance the photocatalytic activity of the adsorbent [14-17]. The mechanism causing the structural changes during adsorption of dye is mainly recognized due to the presence of –NH group in the polymer backbone, whose protonation and deprotonation will bring about a change in the backbone as well as in the colour of the polymer.

Reactive blue 21 and reactive red 180 are used to dye cotton, linen, silk, polyamide fiber and can be used in viscose fabric printing. These dyes may cause sensitization by inhalation and skin contact (28-33 %). Removal of reactive blue 21 and reactive red 180 dyes from aqueous solution is necessary to control water pollution. Past works accounted on reactive blue 21 and reactive red 180 are very few in numbers and the reported natural adsorbents have minimum adsorption capacity [18-21]. Review of literature revealed that there is no adsorption process reported up to this time on PANI-$CoCl_2$, hence we report the viability of PANI-$CoCl_2$ for adsorption of textile dyes (reactive blue 21 and reactive red 180). The adsorption process is carried out under various conditions like agitation with UV exposure, UV exposure alone; agitation alone and by exposure to sunlight at two seasons. The highest percentage of removal is confirmed by doing the adsorption process in different conditions men-tioned above.

EXPERIMENTAL

To synthesize $CoCl_2$ doped PANI, the chemicals used were of analytical grade and used without any further purification and all solutions were prepared using deionized water. Aniline and HCl were procured from Merck Specialties (P) Ltd., Mumbai. Ammonium persulphate was obtained from Loba Chemie Pvt.Ltd., Mumbai. $CoCl_2$ was obtained from E. Merck (India) Ltd., Mumbai. Reactive blue 21 and reactive red 180 dyes used in the adsorption studies were obtained from Colourtex Industries Private Limited.

Preparation of adsorbent: By chemical oxidation coupled with polymerization, cobalt chloride doped polyaniline was synthesized using ammonium persulphate as an oxidant. Ammonium persulphate was dissolved in water and mixed with the solution of aniline in acidic medium (1.5 N HCl) and diluted $CoCl_2$ of required percentage dissolved in water was added to the solution mixture and stirred in a magnetic stirrer at 400 rpm for 4 h. The dark green $CoCl_2$ doped PANI sample was precipitated. On completion of polymerization, PANI-$CoCl_2$ composite from solution was filtered, washed, dried and stored.

Batch experiment: The stock solution of the dye was prepared by dissolving 0.5 g of dye in distilled water and made it up to 100 mL in a standard flask. The dyes were standardized with Beer-Lambert's law by measuring the optical density at various concentrations of dye solutions at λ_{max} 624 nm (reactive blue 21) and 540 nm (reactive red 180), using MAPADA-V-1100D spectrophotometer. The required amount of PANI-$CoCl_2$ composite was taken in a 250 mL beaker containing 100 mL dye solution with the required concentration. This mixture is agitated at 250 rpm under exposure to UV lamp in RIS 24-BL orbital shaker. Simultaneously the mixture with the same concentration and the amount of adsorbent is kept under solar radiation without agitation. At appropriate time interval, 10 mL of aliquot was withdrawn, centrifuged and filtered. The filtrate was collected separately and then the optical density of the clear dye solution was measured until the equilibrium was attained. The concentration of dye solution was measured using UV-visible spectrophotometer.

RESULTS AND DISCUSSION

Effect of contact time: The experiments were carried out by taking 0.8 g/L of PANI-$CoCl_2$ for reactive blue 21 (105 mg/L) and 1.2 g/L for reactive red 180 (55 mg/L) at fixed temperature by varying only the contact time between dye molecules and absorbent sites. The amount of dye adsorbed by the PANI-$CoCl_2$ increases with time and reaches equilibrium when there is no space for the dye molecules to get accumulated on the surface of the adsorbent [22]. The equilibrium time attained at 120 min for reactive blue 21 and 90 % of dye has been adsorbed whereas 93 % of reactive red 180 is adsorbed at an equilibrium time of 150 min. The same experiment was carried out under different conditions like exposure to sunlight alone at summer (78.19 % for reactive blue 21 and 71.58 % for reactive red 180) as well as winter (32.05 % for reactive blue 21 and 27.09 % for reactive red 180), agitation alone (68.14 % for reactive blue 21 and 68.07 % for reactive red 180) and UV lamp exposure alone (41.63 % for reactive blue 21 and 38.13 % for reactive red 180). The results revealed that the maximum adsorption capacity was observed under agitation with UV lamp exposure of dye solution with adsorbent as shown in Fig. 1a-b.

Variation of initial dye concentration, adsorbent dosage, temperature, agitation speed and pH: Effect of the initial concentration of dye shows a decrease in the percentage removal of adsorption of dyes onto PANI-$CoCl_2$ adsorbent with an increase in the initial concentration for reactive blue 21 and reactive red 180. The number of active sites available for the adsorption process to take place is more when concentration of the dye solution is low, on the other hand, increase in the initial concentration of dyes demand more active sites for adsorption but the availability of sites to meet the requirement is constant and so the percentage of removal of the dye is less at higher concentration [23].

It is observed that the increase in adsorbent dosage (Table-1) from 0.4 to 1.2 g/L for reactive blue 21 and from 0.8 to 1.6 g/L for reactive red 180 led to a gradual increase in the percentage of removal which can be attributed to the increase in active sites for the adsorption of dyes with increasing adsorbent dosage of PANI-$CoCl_2$ [24].

The of variation of temperature on the percentage removal of both dyes was carried out at three different temperatures (30, 40 and 50 °C). It is observed that for both dyes the percentage of adsorption increases marginally with an increase in temperature, which shows the endothermic nature of adsorption [25]. Agitation is an important parameter in adsorption phenomena, which influence the distribution of the solute in the bulk of adsorbent [26] and the tabulated result revealed that the dye uptake increases when there is an increase in the agitation speed.

Reactive blue 21 and reactive red 180 molecules have negatively charged sulfonic ($-SO_3-$) functional groups and the surface of the adsorbent is positively charged which result in electrostatic interaction between the dye molecule and the adsorbent. At acidic pH, the sodium salts of dye is ionized and adsorbed naturally, thus resulting in higher percentage of dye removal, contrary to it, increase in pH of the solution

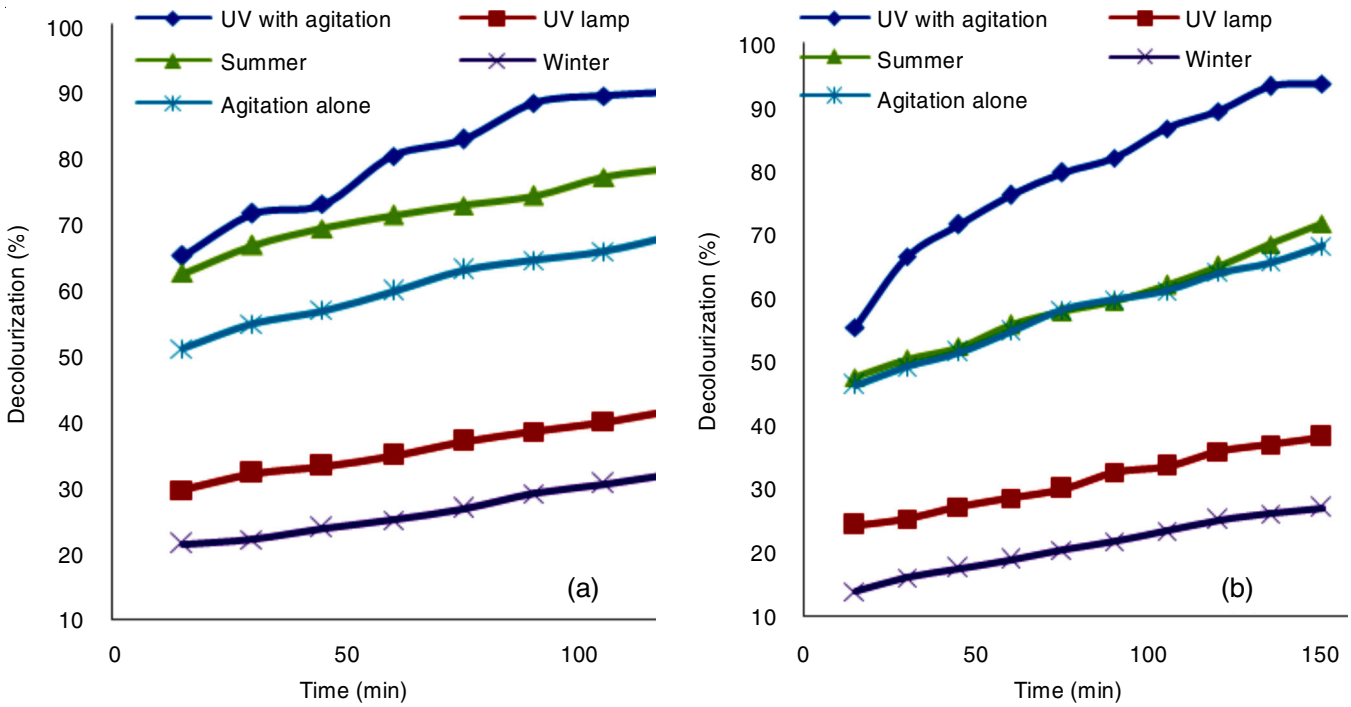

Fig. 1. Contact time of reactive blue 21 (a) and reactive red 180 (b) at different conditions

TABLE-1
VARIATION OF DIFFERENT PARAMETERS AT CONTACT TIME FOR REACTIVE BLUE 21 AND REACTIVE RED 180

Reactive blue 21						Reactive red 180					
Conc. (mg/L)	Dosage of adsorbent (g/L)	Temp. (°C)	pH	Agitation speed (rpm)	Removal (%)	Conc. (mg/L)	Dosage of adsorbent (g/L)	Temp. (°C)	pH	Agitation speed (rpm)	Removal (%)
85	0.8	30	5.32	250	100	35	1.2	30	5.93	250	100
95	0.8	30	5.32	250	99	45	1.2	30	5.93	250	95
105	0.8	30	5.32	250	90	55	1.2	30	5.93	250	94
115	0.8	30	5.32	250	84	65	1.2	30	5.93	250	86
125	0.8	30	5.32	250	76	75	1.2	30	5.93	250	63
105	0.4	30	5.32	250	62	55	0.8	30	5.93	250	72
105	0.6	30	5.32	250	76	55	1.0	30	5.93	250	82
105	0.8	30	5.32	250	90	55	1.2	30	5.93	250	94
105	1.0	30	5.32	250	92	55	1.4	30	5.93	250	98
105	1.2	30	5.32	250	94	55	1.6	30	5.93	250	100
105	0.8	30	5.32	250	89	55	1.2	30	5.93	250	93
105	0.8	40	5.32	250	91	55	1.2	40	5.93	250	96
105	0.8	50	5.32	250	92	55	1.2	50	5.93	250	99
105	0.8	30	5.32	50	53	55	1.2	30	5.93	50	70
105	0.8	30	5.32	100	67	55	1.2	30	5.93	100	80
105	0.8	30	5.32	150	71	55	1.2	30	5.93	150	82
105	0.8	30	5.32	200	80	55	1.2	30	5.93	200	89
105	0.8	30	5.32	250	90	55	1.2	30	5.93	250	94
105	0.8	30	2.00	250	96	55	1.2	30	2.00	250	99
105	0.8	30	4.00	250	91	55	1.2	30	4.00	250	96
105	0.8	30	6.00	250	88	55	1.2	30	6.00	250	93
105	0.8	30	8.00	250	41	55	1.2	30	8.00	250	26

decreases percentage of decolourization and this may be due to the competition of OH^- and dye molecule to the adsorbent site thus resulting in electrostatic repulsion between adsorbent and the negatively charged dye molecules [27].

Electrical conductivity: The electrical conductivity was measured for PANI-$CoCl_2$ before and after adsorption of dye molecule and found that the electrical conductivity of PANI-$CoCl_2$ composite after exposure to the dye solution is decreased which suggest that the dye adsorbed on the surface of the adsorbent.

Desorption: After adsorption of dye molecules onto PANI-$CoCl_2$, the adsorbent with adsorbed dye molecules were collected. The dyes loaded PANI-$CoCl_2$ was put in contact with different concentrations of NaOH and HCl solutions (0.025, 0.1 and 0.5 N of NaOH and HCl) and desorption experiment was performed. It is found that desorption has a positive effect on

the alkaline medium whereas in acidic medium desorption does not occur. The percentage of desorption increased with an increase in the strength of NaOH to some extent as shown in Fig. 2, after a particular strength there is no much difference in the percentage of desorption [28]. The observed value of desorption under investigations are low which shows that physical bonding occurs to a small extent only between the dyes and adsorbent materials and that chemical bonding is largely responsible for the sorption process.

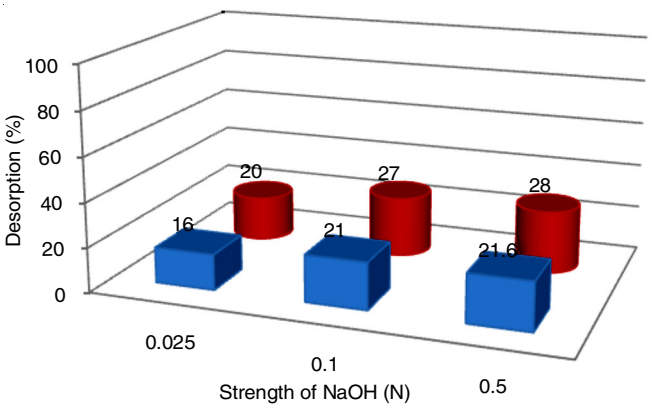

Fig. 2. Desorption of reactive blue 21 and reactive red 180 with respect to strength of NaOH

Adsorption isotherm: The adsorption isotherm study was done to find the most suitable correlation for an equilibrium curve of the adsorption system. There were four adsorption isotherm models used in this study, *e.g.*, Langmuir, Freundlich, Jovanovic and Temkin isotherms. Generally, the Langmuir isotherm model demonstrates monolayer adsorption coverage on a homogenous surface with the limited number of the identical site without any interaction between adsorbed molecules. Meanwhile, Freundlich isotherm deals with reversible adsorption on a heterogeneous surface and it is not restricted to the formation of the monolayer [29-32]. The applicability of these models was compared in terms of correlation coefficients value, R^2 possessed by each sample. Table-2 shows the important parameters calculated from all the models.

Langmuir isotherm: The Langmuir adsorption model was found to fit the experimental data well for both dyes (Fig. 3) sufficiently in accordance with the correlation coefficients (R^2). The larger values of R^2 (0.995 and 0.999) indicted the applicability of the Langmuir isotherm for dye adsorption. The linear equation of Langmuir adsorption model is given in eqn. 1.

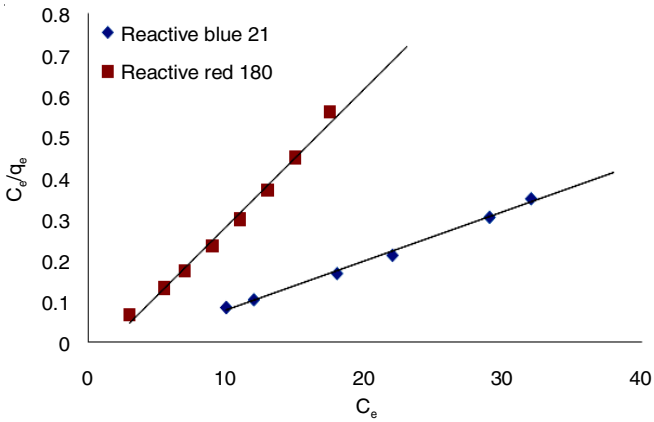

Fig. 3. Langmuir adsorption isotherm model for reactive blue 21 and reactive red 180

$$\frac{C_e}{q_e} = \frac{(1+bC_e)}{Q_o b} \quad (1)$$

The adsorption nature is found to be favourable for both the dyes ($0 < R_L < 1$) from the calculated R_L value [29]. The calculated R_L value for reactive blue 21 is 0.029 and for reactive red 180 R_L value is 0.020. Both the values are greater than zero and lesser than one, so it is found to be a favourable process.

Adsorption kinetics models: The kinetic models were tested to fit experimental data of reactive blue 21 as well as for reactive red 180 dye adsorption on PANI-$CoCl_2$ sample and kinetic parameters were calculated from corresponding linear plots (Fig. 4). As shown in Table-3, higher values of the correlation coefficients (R^2) were obtained which confirms the

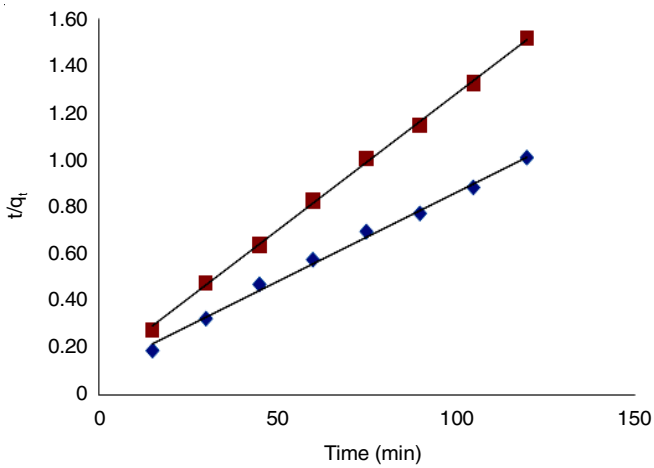

Fig. 4. Pseudo second order model for reactive blue 21 and reactive red 180

TABLE-2
ISOTHERM MODELS AND THEIR CALCULATED CONSTANTS

Isotherms	Linear form of equation	R^2 value	Calculated values
Freundlich	$\ln q_e = \ln K_f + \frac{1}{n}\ln C_e$	Reactive blue 21: 0.975 Reactive red 180: 0.917	Reactive blue 21: n = 4.629; K_F = 197 (L/g) Reactive red 180: n = 4.878; K_F = 81.77 (L/g)
Langmuir	$\frac{C_e}{q_e} = \frac{(1+bC_e)}{Q_o b}$	Reactive blue 21: 0.995 Reactive red 180: 0.991	Reactive blue 21: Q_0 = 90.9 mg/g; b = 0.28 (L/mg); R_L = 0.02 Reactive red 180: Q_0 = 30.3 mg/g; b = 0.73 L/mg; R_L = 0.02
Temkin	$q_e = B_T \ln K_T + B_T \ln C_e$	Reactive blue 21 -0.987 Reactive red 180: 0.912	Reactive blue 21: B_T = 0.045 KJ/mol; K_T = 1.350 L/mg Reactive red 180: B_T = 0.123 KJ/mol; K_T = 6.237 L/mg
Jovanoic	$\ln q_e = \ln q_{max} - K_J C_e$	Reactive blue 21: 0.969 Reactive red 180: 0.995	Reactive blue 21: K_J = 0.013; q_{max} = 137.82 mg/g Reactive red 180: K_J = 0.024; q_{max} = 47.79 mg/g

TABLE-3
PSEUDO SECOND ORDER KINETIC PARAMETERS FOR THE REMOVAL OF REACTIVE BLUE 21 AND REACTIVE RED 180

Dye	q_e (mg/g)		K_2	R^2
	Exp.	Calc.		
Reactive blue 21	111	118	5.1×10^{-4}	0.998
Reactive red 180	44	47	4.5×10^{-4}	0.999

adsorption kinetics is following the pseudo-second-order model. Apart from this, there is a close match in the quantity of dye adsorbed at equilibrium condition between the calculated (q_e, calc) and experimental (q_e, exp) values. Therefore, the rate-limiting step may be the chemisorption process involving electrostatic interactions between the adsorbent and dye molecules [29].

Adsorption thermodynamics: According to van't Hoff equation plots of ln K_d *versus* 1/T which helps to evaluate the thermodynamic parameters such as changes in Gibbs free energy ($\Delta G°$ KJ mol^{-1}), the heat of adsorption enthalpy ($\Delta H°$ KJ mol^{-1}) and entropy ($\Delta S°$ KJ mol^{-1}). The van't Hoff equation is given by:

$$\ln K_d = \frac{\Delta H°}{RT} + \frac{\Delta S°}{R} \quad (2)$$

where K_d is the equilibrium constant, the values of $\Delta H°$ and $\Delta S°$ were calculated from the slopes and intercepts. The $\Delta H°$ value for reactive blue 21 and reactive red 180 calculated from van't Hoff equation is 66.51 and 47.73 KJ mol^{-1}. The positive value of $\Delta H°$ indicates that the overall adsorption process was endothermic in nature and the positive value of $\Delta S°$ (5.1 and 21 KJ mol^{-1}) shows that there is a disorder in the system [33]. The value of Gibb's free energy was calculated at different temperatures according to the equation

$$\Delta G° = -RT \ln K_L \quad (3)$$

The negative value of $\Delta G°$ indicates the spontaneous nature of the adsorption process and the calculated values are listed in the Table-4.

TABLE-4
CALCULATED THERMODYNAMIC PARAMETERS AND THEIR OUTCOME RESULTS

Thermodynamic parameters	Reactive blue 21	Reactive red 180	Outcome results
$\Delta G°$ (KJ mol^{-1})	-62.32 -67.39 -72.98	-67.26 -88.24 -120.8	Spontaneous and feasible in nature
$\Delta S°$ (KJ mol^{-1})	5.1	21.5	Entropy driven
$\Delta H°$ (KJ mol^{-1})	66.51	47.73	Endothermic in nature

The activation energy (E_a) is calculated from the slope of plots of ln k *versus* 1/T using the Arrhenius equation:

$$\ln k = \ln A - \frac{E_a}{RT} \quad (4)$$

Determination of activation energy also gives information about the type of adsorption. The activation energies for physisorption processes are usually in the range 5-40 kJ mol^{-1}, while the chemisorption processes are characterized by higher activation energies (40-800 kJ mol^{-1}). The activation energy for reactive blue 21 and reactive red 180 is found to be 58.19 and 44.35 kJ mol^{-1}, respectively, indicating that the adsorption process is governed by chemical adsorption in both cases [34].

ANOVA for correlation coefficients: To analyze the suitability of the four isotherm models their fitness to the experimental data was assessed. The fitness of the data was established using coefficients of each isotherm. The isotherms with their R^2 values and ANOVA factor and the summary of the analysis are given in the Table-5. Consideration of the comparative average of the R^2 values suggests that the Langmuir adsorption isotherms model provides a better model for the adsorption of dye molecules onto PANI-$CoCl_2$ [35].

Morphological study: Scanning electron microscopy studies are widely used to study the morphological and surface characteristics of the adsorbent material before and after adsorption. It also reveals the surface texture and porosity of the adsorbent material. Evidence from the Fig. 5a-c, the structure of PANI-$CoCl_2$ before adsorption is changed after the adsorption of reactive blue 21 and reactive red 180 dyes on comparison to before adsorption.

FTIR characterization: The bands observed at 3436 cm^{-1} were due to secondary N-H stretching vibration after and adsorption it is shifted to 3433.85 and 3434.67 cm^{-1} for reactive blue 21 and reactive red 180, respectively. The peak present at 1649.49 cm^{-1} is attributed to C=N stretching vibration of PANI-$CoCl_2$ is changed to 1632.90 cm^{-1}. The C-N stretching vibration of aromatic secondary amine is observed at 1242 cm^{-1} was shifted to 1299.15 and 1298.61 cm^{-1}. Strong peaks of the sulphonic group (O=S=O and S-O stretching) at 1342.33 and 1385.04 cm^{-1} shows the adsorption of sulphonic groups present in dyes onto the adsorbent which is absent in PANI-$CoCl_2$. Dye adsorbed PANI-$CoCl_2$ gives different FT-IR spectrum compared with unadsorbed PANI-$CoCl_2$ composite (Fig. 6a-c).

Comparison of effectiveness of PANI-$CoCl_2$ as adsorbent: Various works in the literature discussed the removal of dye from aqueous solutions using PANI-based material as adsorbent. A selective summary of PANI based materials used for the adsorption of dyes to compare with maximum adsorption capacities of PANI-$CoCl_2$ as well as the adsorption of reactive blue 21 and reactive red 180 by other adsorbents are shown in Table-6.

Conclusion

The results of different experiments showed that PANI-$CoCl_2$ can adsorb and remove commonly used textile dyes

TABLE-5
FOUR ISOTHERMS WITH THEIR R^2 VALUES AND SUMMARY OF ANOVA FACTORS

Isotherms	Reactive blue 21	Reactive red 180	Count	Sum	Average	Variance
Freundlich	0.975	0.917	2	1.892	0.946	0.001682
Langmuir	0.995	0.991	2	1.986	0.993	0.000008
Temkin	0.987	0.912	2	1.899	0.9495	0.002812
Jovanovic	0.969	0.995	2	1.964	0.982	0.000338

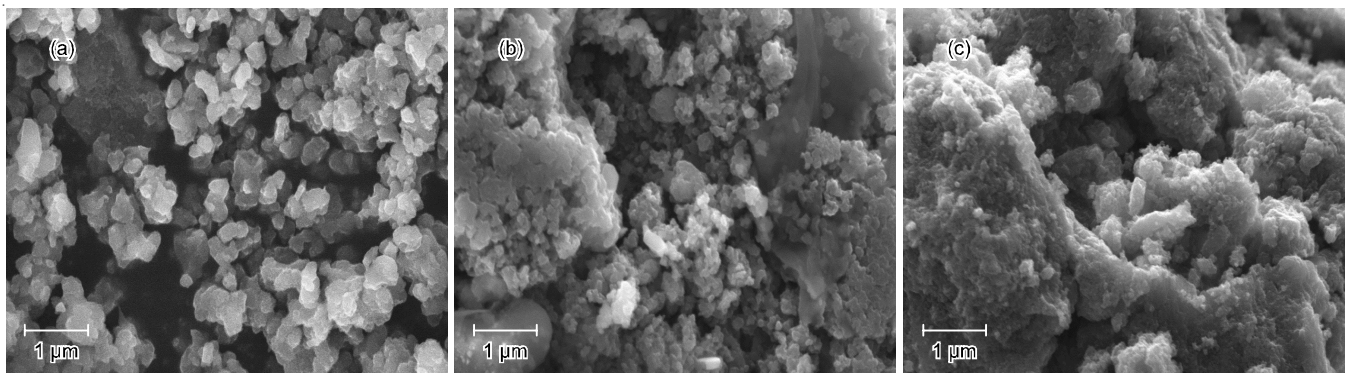

Fig. 5. PANI-$CoCl_2$ before adsorption (a) after adsorption of reactive blue 21(b) and reactive red 180 (c)

TABLE-6
COMPARISON OF THE MAXIMUM ADSORPTION CAPACITIES OF PANI BASED ADSORBENTS AND SOME DYES

Materials used as adsorbents	Dye	Maximum adsorption capacity (mg/g)	Ref.
PANI-EB	Methylene blue	6.1	[35]
PANI nanocomposite	Methylene blue	9.2	[36]
PANI nanotube	Methylene blue	10.3	[37]
PANI silica composite	Methylene blue	13.8	[38]
PANI ZSP nanocomposite	Malachite green	12	[39]
PANI-HCl	Procion red	18.4	[40]
PANI-H_2SO_4	Methyl orange	75.9	[41]
Natural zeolite	Reactive blue 21	9.65	[18]
Clay, activated clay, modified clay	Reactive blue 21	44.43, 37.34, 48.26	[19]
Bagasse fly ash	Reactive red 180	18.28	[20]
Wheat bran	Reactive red 180	34.42	[21]
PANI-$CoCl_2$	Reactive blue 21	90.9	This study
PANI-$CoCl_2$	Reactive red 180	30.3	This study

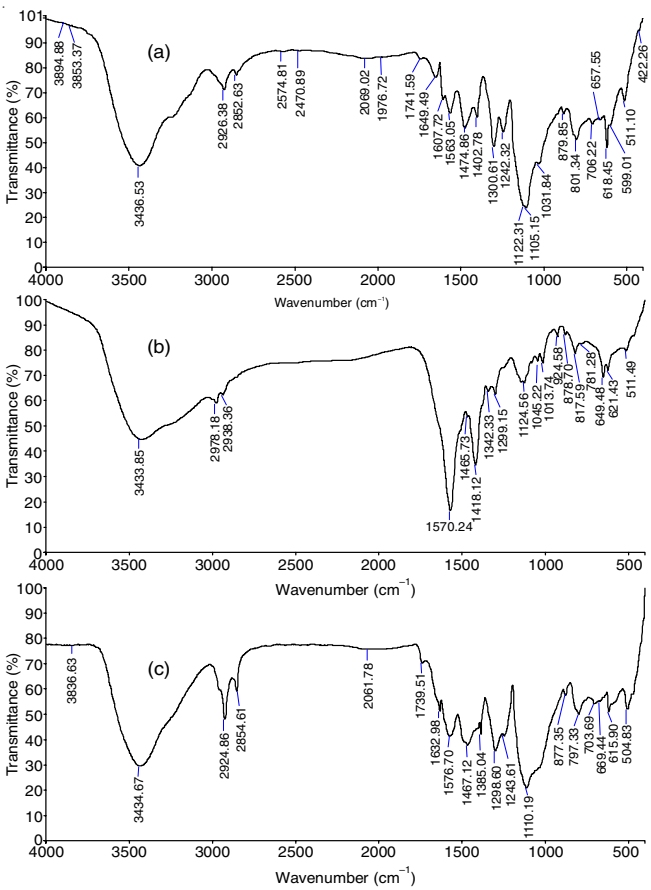

Fig. 6. PANI-$CoCl_2$ before adsorption (a) after adsorption of reactive blue 21(b) and reactive red 180 (c)

reactive blue 21 and reactive red 180 dye from the aqueous solution. Different variables, such as contact time, adsorbent dose, initial concentration, pH and temperature influenced the adsorptive quantity. The best correlation was obtained using the pseudo-second-order kinetic model. The adsorptive process is endothermic and spontaneous.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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